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83. Effect of Cavity Desing on Gap Formation Observed by OCT. **H. Takahashi*[1]**, N. Iwasaki[1], Y. Sumi[2] ([1]Tokyo Medical and Dental University; Japan, [2]National Center for Geriatrics and Gerontology, Japan).

84. Study of the Oxygen-Inhibited Layer of three Universal Dental Adhesives. N. Rosenthal, C.T. Fiuza, **R. França*** (University of Manitoba, Canada).

85. Engineered Peptide for Adhesive/Dentin Interfacial Integrity. **P. Spencer,*[1,2]**, Q. Ye,[1] C. Tamerler[1,2] ([1]Bioengineering Research Center, University of Kansas, Lawrence, USA; [2]Department of Mechanical Engineering, University of Kansas, Lawrence, USA).

86. Simulated Gap Wear of Resin Luting Cements. **A. Tsujimoto*[1,2]**, W.W. Barkmeier[1], T. Takamizawa[2], M. Miyazaki[2], M.A. Latta[1] ([1]Nihon University, Japan; [2]Creighton University School of Dentistry, USA).

87. Selective Etching in NCCL for Self-Etch Adhesives: SR And Meta-Analysis. **M. Schroeder***, A. Szesz, S. Parreiras, A. Reis, A. Loguércio (Universidade Federal do Rio de Janeiro, Brazil).

88. Bond Strength and Sealing Ability between Fiber Posts to Dentin. **P.H. Dos Santos*[1]**, T.Y.U. Suzuki[1], M.A. Pereira[1], J.E. Gomes-Filho[1], L Wang[2], A.L.F. Briso[1], W.G. Assunção[1] ([1]Araçatuba School of Dentistry, UNESP, Brazil; [2]Bauru School of Dentistry, USP, Brazil).

89. Monomer Conversion and Mechanical Properties of Contemporary Bulk-Fill Composites. E.C. Rodrigues Júnior[1], C.E. Francci[1], P.P.A.C. Albuquerque[1], M.C. Dos Reis[1], C.M.C. Tapety[2], P.F. Cesar[1], **L.E. Rodrigues Filho*[1]** ([1]University of São Paulo, Brazil; [2]Federal University of Ceará, Brazil).

90. Effect of Cranberry and Proanthocyanidin in Dentine Erosion Prevention. **H.M. Honorio*[1]**, A.P. Boteon[1], M.T. Kato[2], M.A.R. Buzalaf[1], A. Prakki[3], L. Wang[1], D. Rios[1] ([1]Bauru School of Dentistry- University of São Paulo, Bauru, Brazil; [2]Sacred Heart University, Bauru, Brazil; [3] University of Toronto, Toronto, Canada).

91. Influence of Biosilicate Incorporation on the Mechanical Properties of GIC. **F. Pires-de-Souza*[1]**, M.M.M.G. Contente[1], S.A.F. Vicente[1], R. Tonani[1], E.J. Nassar[2], S. Geraldeli[3] ([1]Ribeirao Preto School of Dentistry, University of Sao Paulo, Brasil; [2]University of Franca, Brasil; [3]University of Florida, USA).

92. Flexural Strength and Contrast Ratio Comparison for Translucent Zirconia. **M. Carrabba***, A. Vichi, R. Sorrentino, R. Fabian Fonzar, M. Ferrari (University of Siena, Department of Medical Biotechnologies, Siena, Italy).

93. Bond Strength of Composite Resin to Nickel-Chrome Alloy Using Primers. **G. Nima***, P.V.C. Ferreira, M. Giannini (Piracicaba Dental School, Brazil).

94. Are Novel Universal Adhesives more Resilient to Salivary Contamination? **P. Nair***, N. Ilie (Ludwig Maximilians University, Department of Conservative Dentistry and Periodontology, Munich, Germany).

95. Fabrication of Gradient Scaffolds for Bone and Dental Tissue Engineering. **M. Rasoulianboroujeni*[1]** S. Pitcher[1] L. Tayebi[1,2] ([1]Department of Developmental Sciences, Marquette University School of Dentistry, Milwaukee, USA; [2]Department of Engineering Science, University of Oxford, Oxford, UK).

96. Polymerization Properties Assessment of Bulk Fill Resin Composites. **F.A.P. Rizzante*[1]**, J.A. Duque[1], R.M. Maenosono[1], R.F.L. Mondelli[1], A.F.S. Borges[1], A.Y. Furuse[1], G. Mendonça[2], S.K. Ishikiriama[1] ([1]Bauru School of Dentistry, University of São Paulo, Brazil; [2]University of Michigan School of Dentistry, USA).

97. Improving Mechanical Properties of GICs by adding Hydroxyapatite and Fluorapatite. F. Barandehfard[1], M.A Kianpour Rad[2], A. Hosseinnia[2], **M. Tahriri[3,4,5*]**, L. Tayebi[3] ([1]Nanomaterials Department, Materials and Energy Research Center (Merc), Tehran, Iran; [2]Environmental and Energy Research Group of Materials and Energy Research Center (Merc), Tehran, Iran; [3]Marquette University School of Dentistry, Milwaukee, USA; [4]Dental Biomaterials Group, School of Dentistry, Tehran University of Medical Sciences (Tums), Tehran, Iran; [5]Biomaterials Group, Faculty of Biomedical Engineering, Amirkabir University of Technology, Tehran, Iran).

98. Preparation of Strontium-Containing Calcium Phosphate Cements for Maxillofacial Bone Regeneration. R. Masaeli[1], T.S. Jafarzadeh Kashi[1], **W. Yao[2*]**, K. Khoshroo[2], M. Tahriri[2], L. Tayebi[2] ([1]Dental Biomaterials Department, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran; [2]Marquette University School of Dentistry, Milwaukee, USA).

99. 3 Dimensional Scaffolds based on PCL/Titania Nanotube Fabricated via Microsphere Sintering. K. Khoshroo[1], T.S. Jafarzadeh Kashi[2], **P. Shah[1*]**, M. Tahriri[1,2], L. Tayebi[1] ([1]Marquette University School of Dentistry, Milwaukee, USA; [2]Dental Biomaterials Department, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran).

100. Biodegradation Properties of PLGA/Nano-Fluorhydroxyapatite Composite Microsphere-Sintered Scaffolds. M. Tahriri[1,2,3], F. Moztarzadeh[2], K. Hresko[1*], **K. Khoshroo[1,3]**, L. Tayebi[1] ([1]Marquette University School of Dentistry, Milwaukee, USA; [2]Biomaterials Group, Faculty of Biomedical Engineering, Amirkabir University of Technology, Tehran, Iran; [3]Biomaterials Department, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran).

101. Device for Automatic Evaluation of Biomaterials Micro-Leakage by Gas Permeability. **D. Chavarria-Bolaños*[1]**, A. Sánchez[1], E. Conejo[2], A. Pozos-Guillén[3] ([1]Universidad De Costa Rica, Facultad De Odontología, San José, Costa Rica; [2]Universidad De Costa Rica, Centro De Investigación En Ciencias Atómicas, San José, Costa Rica; [3]Universidad Autónoma De San Luis Potosí, San Luis Potosí, México).

102. Synthesis of Fluorine-Substituted Hydroxyapatite Nanopowders for Dental Applications. M. Tahriri[1], **J. White[1*]**, B. Shah[1], H. Eslami[2], L. Tayebi[1] ([1]Marquette University School of Dentistry, Milwaukee, USA; [2]Biomaterials Group, Faculty of Biomedical Engineering, Amirkabir University of Technology, Tehran, Iran).

103. Novel Glassflake Resin Composites. A. Abyad[1], Y. Zhao[2], **X.Chen[2*]** and D.C. Watts[2] ([1]School of Materials, the University of Manchester, Manchester, UK; [2]School of Dentistry, the University of Manchester, Manchester, UK).

104. Interaction of Chlorhexidine with MDP-Based Dentin Bonding Systems. **L. Wang*[1]**, M.A.S. Agulhari[1], L.F.F. Brianezzi[1], M.C. Giacomini[1], M.M.A.C. Velo[1], P.M.C. Scaffa[1], H.M. Honório[1], M.R.O. Carrilho[2], J. Hebling[3], A.Y. Furuse[1] ([1]Bauru School of Dentistry, University of São Paulo, Bauru, Brazil; [2]Biomaterials Research Group, School of Dentistry, Anhanguera University, São Paulo, Brazil; [3]Araraquara School of Dentistry, University Estadual Paulista, Araraquara, Brazil).

105. Thermography Analysis during Laser Irradiation on Dentin-Bonding Agents. **L.F.F. Brianezzi*[1]**, G.S. Zabeu[1], R.M. Maenosono[1], P.A. Ana[2], R.G. Palma-Dibb[3], L. Wang[1], S.K. Ishikiriama[1] ([1]Bauru School of Dentistry, University of São Paulo, Brazil; [2]Biomedical Engineering Program, Federal University of ABC, Brazil; [3]Ribeirão Preto School of Dentistry, University of São Paulo, Brazil).

106. Proteolytic-Inhibitors use for Bonding Carious/ Eroded Dentin: Does it Work? **M.C. Giacomini*[1]**, P.M.C. Scaffa[1], C.M.P. Vidal[2], L. Tjäderhane[3], H.M. Honório[1], L. Wang[1] ([1]Bauru School of Dentistry, University of São Paulo, Bauru, São Paulo, Brazil; [2]School of Health Sciences, Department of Dentistry, University of Brasilia, Brasilia, Brazil; [3] Institute of Dentistry, University of Oulu, Finland).

107. Abfraction *versus* Erosion: Impact of Type-Lesion on 1-Year Restorative Performance. **M.A.S. Agulhari***, A.F. Soares, E.C. Consolmagno, O. Bim-Júnior, R.F. Rodrigues, M.S.A. Falcão, H.M. Honório, S.K. Ishikiriama, L. Wang (Bauru School of Dentistry, University of São Paulo, Bauru, São Paulo, Brazil).

108. Thermally Oxidized Ti_6Al_4V Alloys Enhance the Corrosion Behavior. **A. Bouchecham***, A. Karaali, Y. Keriti (University of Freres Mentouri-Constantin, Algeria).

109. Endocrine Disruptors in Paediatric Patients & *In-Vitro* Sealant Weight Variability. **S.J. Wileman*[1]**, S. Hanks[1], M. Priston[1,2], T. Deperalta[3], A.Rigby-Jones[1], Jr. Sneyd[1] ([1]Plymouth University Peninsula Schools of Medicine and Dentistry, Plymouth, UK; [2]Plymouth Hospitals NHS Trust, Plymouth, UK; [3] University of Michigan School of Dentistry, Ann Arbor, USA).

110. Mechanical, Microbiological and Copper Release from Copper Nanoparticles-Containing Adhesives. M.F. Gutiérrez[1,2], P. Malaquias[1], T.P. Matos[1], A. Szesz[1], S. Souza[1], J. Bermudez[1], A. Reis[1], **A.D. Loguercio*[1]**, P.V. Farago[3] ([1]Restorative Dentistry, State University of Ponta Grossa, Brazil; [2]Institute for Research In Dental Sciences, University of Chile, Santiago, Chile; [3]Department of Pharmaceutical Sciences, State University of Ponta Grossa, Brazi).

111. Preventive Effect of Potassium Nitrate-Glutaraldehyde Gel in the Bleaching-Induced Sensitivity. **A. Reis***, S. Parreiras, F.M. Coppla, E.C. Martini, A. Szesz, A.D. Loguercio (Restorative Dentistry Departament, State University of Ponta Grossa, Brazil).

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112. Proteolytic Activity of Dentin Caries-Like Lesions provided by Smutans Biofilm. **J.P. De Sousa*[1]**, L.F. Barbosa-Martins[1], F.D. Nascimento[2], R.M. Puppin-Rontani[1] ([1]FOP/University of Campinas, BR; [2] Universidade Anhanguera, BR).

113. Hydroxyapatite/Dicalcium Phosphate Dihydrate Composite for Dental Bone Defect Repair. D. Bizari[1], F. Moztazradeh[1], **M. Fahmy[2*]**, M. Tahriri[2], L. Tayebi[1] ([1]Biomaterials Group, Faculty of Biomedical Engineering, Amirkabir University of Technology, Tehran, Iran; [2]Marquette University School of Dentistry, Milwaukee, USA).

114. Biocompatibility of Modified Glass-Ionomer Cements on Periodontal Ligament (PDL) Cells. F. Barandehfard[1], **Z. Metalwala[2*]**, M. Tahriri[2], K. Khoshroo[2], F. Fahimipour[2], L. Tayebi[2] ([1]Nanomaterials Department, Materials and Energy Research Center (Merc), Tehran, Iran; [2]Marquette University School of Dentistry, Milwaukee, USA).

115. 3D-Printing of Porous Calcium Phosphate Cements for Bone Tissue Engineering. **K. Khoshroo[1*]**, T. Almela[2], M. Tahriri[1], F. Fahimipour[1], Z. Metalwala[1], K. Moharamzadeh[2], L. Tayebi[1] ([1]Marquette University School of Dentistry, Milwaukee, USA; [2]School of Clinical Dentistry, University of Sheffield Claremont Crescent, Sheffield, UK).

116. 3D-Printed ß-TCP/Collagen Scaffolds for Bone Tissue Engineering. **F. Fahimipour[1*]**, T.S. Jafarzadeh Kashi[2], K. Khoshroo[1], E. Dashtimoghadam[1], M. Rasoulianborujeji[1], L. Tayebi[1] ([1]Marquette University School of Dentistry, Milwaukee, USA; [2]Dental Biomaterials Department, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran).

117. 3-D Printed PCL/Halloysite Scaffolds for Craniomaxillofacial Bone Regeneration. H.E. Jazayeri, E. Dashtimoghadam, M. Rasoulianbroujeni, M. Bures, **L. Tayebi*** (Marquette University School of Dentistry, Milwaukee, USA).

118. Biomimetic Analogs/Bioactive Phosphates based Adhesives Promote Dentin Collagen Mineralization. M.A.C. Sinhoreti[1], E.F. Soares[1], G.F. Abuna[1], J.F. Roulet[2], **S. Geraldeli[2*]** ([1]State University of Campinas, Piracicaba Dental School, Piracicaba, Brazil; [2]University of Florida, College of Dentistry, Gainesville, USA).

119. Photo-Fenton: An Alternative for Dental Bleaching. **B.A. Lopes*[1]**, P.E.C. Cardoso[1], A.C.S.C. Teixeira[2] ([1]University of Sao Paulo, Brazil; [2]Polytechnic School of Engineering, University of São Paulo, Chemical Engineering Department, Brazil).

120. Resistance of Resin-Based Materials against Prolonged Erosive and Abrasive Challenges. **D. Rios***, G.C. De Oliveira, M.C. Jordão, M.A.G. Bassoto, E.J. Dionísio, L. Wang, H.M. Honório (Bauru School of Dentistry, University of São Paulo, Bauru, Brazil).

121. Transmittance of Ceramics and Dual-Cure Resin Cements Polymerization. **I.S. Ramalho*[1]**, L.M. Mendonça[1], L.A.S.N. Lima[1], L.A. Pires[1], M.D.S. Lanza[1], T.A. Pegoraro[2], L. Wang[1], L.F. Pegoraro[1] ([1]University of São Paulo, Bauru School of Dentistry, Bauru, Brazil; [2]Universidade Do Sagrado Coração, Bauru, Brazil).

122. Microfluidic-Directed Synthesis of Polymeric Nanoparticles for Bone Cancer Therapy. **E. Dashtimoghadam***, F. Fahimipour, B. Davaji, M.M. Hasani-Sadrabadi, L. Tayebi (Marquette University School of Dentistry, Milwaukee, USA).

123. Color Stability and Flexural Strength of 4-Meta/MMA-TBB Resin. S Jang, C Lee, B Lee, Y. Choi, **S. Park*** (Wongkwang University Dental Hospital, Department of Conservative Dentistry, Iksan, Korea).

124. Flexural Strength of Dual-Cured Provisional Resin with and without Visible-Light. **Y. Nagasawa***, Y. Hibino, H. Shigeta, H. Nakajima (Meikai University School of Dentistry, Japan).

125. Thermographic Analysis of Photo-Cured Composite in Tooth Cavity. **M.J. Kim***, N. Cho, I.B. Lee (Seoul National University, Republic of Korea).

126. Monomer Leaching and Degree of Conversion of Bioactive Dental Composites. H. Khalid[1], M.R. Syed[1,2], H. Iqbal[1], I.U. Rehman[4], **A.S. Khan*[1,3]** ([1]Interdisciplinary Research Centre In Biomedical Materials, Comsats Institute of Information Technology, Lahore, Pakistan; [2]University of Health Sciences, Lahore, Pakistan; [3]College of Dentistry, University of Dammam, Dammam, Kingdom of Saudi Arabia; [4]Kroto Research Institute, Department of Materials Science and Engineering, University of Sheffield, Sheffield, UK).

127. Discontinuous Lamellar Mesostructured Hydroxyapatite Formation in Sodium Dodecyl Benzene Sulfonate. **B. González[1]**, A.J. Salinas[1], O. Mersinlioglu[1], M. Vallet-Regí[1], **M. Özcan*[2]** ([1]Facultad De Farmacia, Universidad Complutense De Madrid, Spain; Dental Materials Unit, Center of Dental Medicines, Faculty of Medicine, University of Zurich, Switzerland).

128. 3D Roughness of Cranioplasty Titanium Implants Following Different Surface Treatments. **M.M. Hatamleh*[1]**, A. Alnazzawi[2], D.C. Watts[3] ([1]Department of Oral and Maxillofacial Surgery, King's College Hospital, UK; [2]College of Dentistry, Taibah University, Kingdom of Saudi Arabia; [3]School of Dentistry, University of Manchester, UK).

129. Effects of Beverages on Color Stability of PICN Material. **M.M. Özarslan***, U.S. Büyükkaplan, C. Barutcigil, K. Barutcigil (Akdeniz University, Turkey).

130. Tooth Substance Removal for Crown versus Filling Therapy. **M. Øilo***, H. Nesse (University of Bergen, Norway).

131. pH-Activatable Nano-Amorphous Calcium Phosphate to Reduce Dental Enamel Demineralization. **M.A.S. Melo*[1]**, M.D. Weir[1], V.F. Passos[2], M. Powers[1], H.H.K. Xu[1,3,4] ([1]Biomaterials & Tissue Engineering Division, University of Maryland Dental School, Baltimore, USA; [2]School of Dentistry, University of Fortaleza, Fortaleza, Brazil; [3]Center for Stem Cell Biology & Regenerative Medicine, University of Maryland School of Medicine, Baltimore, USA; [4]Department of Mechanical Engineering, University of Maryland, Baltimore, USA).

132. Evaluation of Universal Bonding Agent Containing MDP on Zirconia Bonding. T.W. Kim, S.I. Kim, S.J. Shin, H.K. Yoo, G.Y. Lee, **J.W. Park*** (Yonsei University College of Dentistry, Seoul, South Korea).

133. Service Simulation and Reliability of Multilayer Ceramic Structures. **A. Della Bona***, R. Alessandretti, M. Borba, P. Benetti, P.H. Corazza (School of Dentistry, University of Passo Fundo, Brazil).

134. Strength of 3 Y-TZP Depending on Grain Size and LTD. **R. Belli***, C. Loher, A. Petschelt, U. Lohbauer (University of Erlangen-Nuremberg, Erlangen, Germany).

135. Hydrothermal Degradation of Monolithic Zirconia: Guidelines for Finishing Treatments. R. Sorrentino[2], R. Leone[1], E. Camposilvan[3], J. Chevalier[4], **M. Ferrari*[2]**, F. Zarone[1] ([1]University" Federico II" of Naples, Italy; [2]University of Siena, Italy; [3]University Politècnica De Catalunya of Barcelona, Spain; [4]University of Lyon, France).

136. Biomimetic Remineralizing Agents Influence Artificial Caries-Affected Dentin Surface Properties. **J. Puppin-Rontani***, J.P. Sousa, L.F. Barbosa-Martins, R.M. Puppin-Rontani (State University of Campinas, BR).

137. In Vitro Analysis of Rhus Coriaria Extract Interaction with Demineralized Dentin Matrix. **R. Seseogullari-Dirihan[1]***, C.O. Navarra[2], L. Fontanive[2], M. Cadenaro[2], A. Tezvergil-Mutluay[1] ([1]University of Turku, Finland; [2]University of Trieste, Italy).

138. Fractographic Analysis of Fractured Lava Ultimate Crowns over Zirconia Abutments. **U. Lohbauer*[1]**, U. Schepke[2], M. Wendler[1,3], R. Belli[1] ([1]University of Erlangen, Germany; [2]University of Groningen, Netherlands; [3]University of Concepcion, Chile).

139. WITHDRAWN

140. Unbound Monomers do Diffuse Through the Dentin Barrier. **K. Mahdhaoui***, M.A. Derbanne (Université Paris Descartes - Sorbonne Paris Cité, Montrouge, France).

141. Oral Environment Simulation affects Ceramic Failure Behavior. E. Lodi, **M. Borba***, K.R.W. Rhoden, A. Della Bona, P. Benetti, P.H. Corazza (University of Passo Fundo, Passo Fundo, Brazil).

142. Effect of Pine Bark Proanthocyanidins on Dentin and Resin-Dentin Interfaces. **P.V.F. Braz*[1,2]**, A.A. Leme-Kraus[1], B. Aydin[1], J.W. Nam[1], R. Phansalkar[1], S.N. Chen[1], G. Pauli[1], A.K. Bedran-Russo[1] ([1]University of Illinois at Chicago, USA; [2]University of Brasilia, Brazil).

143. Experimental ZnO Cement Containing Bioactive Niobium Biophosphate Fillers. **P. Ferreira***, G. Abuna, A. Menezes, M. Sinhoreti, C. Carvalho, S. Consani, J. Bauer (University of Campinas, Brazil).

144. Influence of Acid Saliva on Wear of Human Dentin. **P.V. Soares*[1]**, M.F. Alfaro[2], I.V. Marques[2], A.C. Machado[1], M.T. Mathew[2], A.K. Bedran-Russo[2] ([1]Federal University of Uberlandia, Operative Dentistry and Dental Materials, Brazil; [2]University of Illinois at Chicago, Dentistry, USA).

145. Glass Ionomer Cements: Liquid Dynamics in Confinement. M.C. Berg[1,2], **A.R. Benetti*[3]**, H.N. Bordallo[1,2] ([1]Niels Bohr Institute, University of Copenhagen, Denmark; [2]European Spallation Source, Sweden; [3]Department of Odontology, University of Copenhagen, Denmark).

146. Fatigue Limit of Monolithic Zirconia FDP: Damage and Glass Infiltration. **M. Amaral*[1]**, R. Rocha[2], R.M. Melo[2], G.K. Pereira[3], T. Campos[4], Y. Zhang[5], L.F. Valandro[3], M.A. Bottino[2] ([1]University of Taubaté, BR; [2]Univ. Estadual Paulista, BR; [3]University of Santa Maria, BR; [4]Aeronautic Institute, BR; [5]New York University, USA).

147. Does Zirconia Translucency Affect Bond Strength of Resin-Based Cements? **N. Scotti***, E. Manzon, A. Comba, M. Alovisi, D. Pasqualini, E. Berutti (Dental School Lingotto, University of Turin).

148. Surface Finishing does not Affect Catastrophic Failure of Glass-Ceramic Crowns. **P. Benetti***, R.T. Mores, M. Borba, P.H. Corazza, A. Della Bona (University of Passo Fundo, Brazil).

149. Bond Strength Evaluation of a Novel Hydrophilic Adhesive System. **F. Petrolo*[1]**, A.Comba[1], L. Breschi[2], E. Berutti[1], N. Scotti[1] ([1]Dental School Lingotto, University of Turin; [2]Dibinem, University of Bologna).

150. Curing Effectiveness of Multiple Resin Composites with LED Curing Units. **A.P. Manso*[1]**, B.U. Peres[1], I. Feng[1], L.D. Carvalho[1], R.V. Rodrigues[1,2], R.M. Carvalho[1] ([1]University of British Columbia, Canada; [2]State University of Campinas, Brazil).

151. Interaction between a Bisphosphonate, Zoledronate, and Biomimetic Hydroxyapatite. P.V. Rocha[1], A.H.M. González[1,2], A.E.A. Castro[2]; **M.R. Carrilho*[1,2]** ([1]Biomaterials Research Program, Anhanguera University of São Paulo, Brazil; [2]Biotechnology and Innovation In Health Program, Anhanguera University of São Paulo, Brazil).

152. Fatigue Resistance of Cubic/Tetragonal Translucent Zirconia Crowns. P. Baldissara[1], **C. Parisi*[1]**, E. Evangelisti[1], V. Wandscher[2], D. Lodi[1] ([1]University of Bologna – Dibinem, Bologna, Italy, [2]Federal University of Santa Maria, Rio Grande Do Sul, Brazil).

153. Mini-Interfacial Fracture Toughness of Adhesive-Dentin Interfaces after Plasma Pre-Treatment. **A.P. Ayres[1,2*]**, P. Pongprueksa[3], J. De Munck[1], A. Vananroye[4], C. Clasen[4], F.D. Nascimento[5], M. Giannini[2], B. Van Meerbeek[1] ([1]University of Leuven, Department of Oral Health Sciences, Leuven, Belgium; [2]University of Campinas, Piracicaba, Brazil; [3]Mahidol University, Faculty of Dentistry, Bangkok, Thailand; [4]University of Leuven, Chemical Engineering Department, Leuven, Belgium; [5]Anhanguera University, Biomaterials Research Group, São Paulo, Brazil).

154. Marginal Adaptation of Composites on Dentin with Different Imaging Techniques. **A. Comba*[1]**, R. Michelotto Tempesta[2], G. Ventura[2], A. Luganini[4], C. Alovisi[3], E. Berutti[2], N. Scotti[2] ([1]University of Bologna, Dibinem, Italy; [2]University of Turin, Dental School, Italy; [3]University of Turin, Clinica Oculistica, Italy; [4]University of Turin, Department of Life Sciences and Systems Biology, Italy).

155. Marginal and Internal Space of Metallic Copings. **F.F. Piras*[1]**, J.R. Berro-Filho[1], J.H. Rubo[1], F. Ferruzzi[2], B.M. Ferrairo[1], V. Mosquim[1] ([1]University of Sao Paulo, Bauru School of Dentistry, Bauru, Brazil; [2]Faculty Inga, Dept. of Dentistry, Maringa, Brazil).

156. Fluoride Varnishes: *In Vitro* Assessment of Fluorine Diffusion on Enamel. S. Virdi, **A.C.T. Schettini***, R. França (Dental Biomaterials Research Lab, College of Dentistry, University of Manitoba, Canada).

157. Effects of Bioactive Suspensions on Resin-Dentin Interface. **J. Bauer*[1]**, E.M Carvalho[1], A.S. Silva[1], A.S. Menezes[1], C.N. Carvalho[2], A.P. Manso[3], R.M. Carvalho[3] ([1]Universidade Federal Do Maranhão (UFMA), Brasil; [2]Uniceuma, Brasil; [3]University of British Columbia, Canada).

158. Effect of Resin Cement and Aging on Bonding to Ceramic. **L. Correr-Sobrinho***, R.F.S. Lacerda, A.R. Costa, A.B. Correr, R.L.X. Consani, M.A.C. Sinhoreti, S. Consani, L.R.M. Martins (State University of Campinas, BR).

159. Fluoride Uptake Measurements from Sealants Formulated with Microencapsulated Remineralizing Agents. **S.M. Gross*[1]**, W.A. McHale[2], M.A. Latta[1], B. Zalsman[3] ([1]Creighton University, Chemistry and Oral Biology, Omaha, USA; [2]Premier Dental Products Company, Plymouth Meeting, USA; [3]BJM Laboratories, Yehuda, Israel).

160. Immediate effect of Different Hydrophobic-Resin-Coating Approaches on Dentin with Universal-Adhesive. **M.A. Munoz*[1]**, I.V. Luque-Martinez[2], C. Cademartori-Borquez[1], X. Gonzalez-Alarcon[1], O. Garrido-Bravo[1], A.D. Loguercio[3], A. Reis[3] ([1]Universidad De Valparaiso, Chile; [2]Pontificia Universidad Catolica De Chile, Chile; [3]Universidade Estadual De Ponta Grossa, Brasil).

161. Survival of Lithium-Disilicate Table-Tops as a Function of Thickness. C. Parisi, E. Onofri, **P. Baldissara***, D. Melilli, R. Fonseca, R. Scotti (University of Bologna, Italy).

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203. Effect of Radiation and Antioxidant on Dentin-Composite Bonding. **A. Costa*1**, L. Correr-Sobrinho[1], C.J. Soares[2], R.M. Puppin-Rontani[1], E.F. Soares[1], A.B. Correr[1], M.A.C. Sinhoreti[1] ([1]State University of Campinas, BR; [2]Federal University of Uberlândia, BR).

204. In vitro aging and mechanical properties of translucent monolithic zirconia. **R. Leone*1**, R. Sorrentino[2], E. Camposilvan[3], J. Chevalier[4], F. Zarone[1], M. Ferrari[2] ([1]University "Federico II" of Naples, Italy; [2]University of Siena, Italy; [3]University Politècnica De Catalunya of Barcelona, Spain; [4]University of Lyon, France).

Paffenbarger Award Finalists

P1. The Complexity of Proanthocyanidins on the Resin-Dentin Bond Strength. **A.A. Leme-Kraus*1**, A.B.S. Souza[1], R.S. Phansalkar[2], C.M.P. Vidal[1], J. Mcalpine[2], G.F. Pauli[2], S.N. Chen[2], A.K. Bedran-Russo[1] ([1]Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, USA; [2]Department of Medicinal Chemistry and Pharmacognosy; College of Pharmacy, University of Illinois at Chicago, Chicago, USA).

P2. Compounds Incorporation that Modify Streptococcus Mutans Virulence in Restorative Materials. **C.B. Andre***[1], J.L. Ferracane[2], P.L. Rosalen[1], C. Pfeifer[2], M. Giannini[1] ([1]State University of Campinas, Br; [2]Oregon Health and Science University, USA).

P3. Y-TZP Low Temperature Degradation: A Sigmoidal or a Linear Behavior? **A. Arata*1**, L.R. De Pretto[1], V. Ussui[1], N.B. Lima[1], A.Z. Freitas[1], J.P.B. Machado[2], R.N. Tango[3], G.M.D. De Souza[4], D.R.R. Lazar[1] ([1]Nuclear and Energy Research Institute, Br; [2]National Institute for Space Research, Br; [3]Institute of Science and Technology, Br; [4]University of Toronto, Ca)

P4. Quality of Cure on Depth of Bulk-Fill Composites: Cytotoxicity. **M. Gilli***, G. Leloup, J.G. Leprince (Université catholique de Louvain, Belgium).

P5. Modern Resin-Based Composite Filler Characteristics and Related Physico-Mechanical Properties. L.D. Randolph[1], **J.Y. Beauquis*1**, W.M. Palin[2], G. Leloup[1], J.G. Leprince[1] ([1]Université Catholique De Louvain, Louvain Drug Research Institute, Brussels, Belgium; [2]University of Birmingham, College of Medical and Dental Science, Birmingham, UK).

P6. Mica Glass Ceramics with Yttria Stabilized Zirconia for Dental Restorations. **S. Gali*1**, K. Ravikumar[2], B.V. Sreenivasamurthy[1], B. Basu[2] ([1]MS Ramaiah University of Applied Sciences, Bangalore, India; [2]Indian Institute of Science, Laboratory for Biomaterials, Bangalore, India).

P7. Microscale Spatial Variability of Monomer Conversion in Filled Dental Resin-Based-Composites. **S. Sirovica*1**, R.A. Martin[1], M.W.A. Skoda[2], O. Addison[3] ([1]School of Engineering and Applied Science and Aston Research Centre for Healthy Ageing, University of Aston, UK; [2]Isis Pulsed Neutron and Muon Source, Science and Technology Facilities Council, Rutherford Appleton Laboratory, Harwell Science and Innovation Campus, Didcot, UK; [3]Biomaterials Unit, School of Dentistry, University of Birmingham, Birmingham, UK).

P8. Comparison of Different Dentifrices in Organic Matrix After Erosion/Abrasion. **T.L. Bueno*1**, A.P. Botteon[1], F. Cardoso[1], M.R. Costa[1], B.K. De Souza[1], G.C. Oliveira[2]; A. Prakki[3], M.A.R. Buzalaf[4], H.M. Honório[2] ([1]Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil; [2]Bauru School of Dentistry, University of São Paulo, Department of Pediatric Dentistry, Orthodontics and Public Health; [3]University of Toronto, Department of Biological and Diagnostic Sciences-Preventive Dentistry, Toronto, Canada; [4]Bauru School of Dentistry, University of São Paulo, Department of Biological Sciences).

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1

Cytocompatibility and bacterial adhesion behavior of three pure titanium surfaces

Y. Wang*, C. Zhang, K. Wang

Guanghua School of Somatology, Sun Yat-Sen University, China

Purpose/Aim: To fabricate a titanium surface with nanostructure through a novel anodization technique and compare its cytocompatibility, bacterial adhesion and antibiotic treatment efficacy with mechanical polishing (MP) and sandblast – large grit and acid-etching (SLA) surfaces.

Materials and methods: Pure titanium foils were treated with a novel anodization technique to get an anatase titania nanotube (TNT-A) surface. The surface topography, roughness and wettability of the surface were detected respectively. The adhesion, proliferation and differentiation of MC3T3-E1 preosteoblasts on the surface were assessed through SEM observation, cell counting kit-8 (CCK-8) and alkaline phosphatase kit. The bacterial adhesion of the multi-species microcosm was measured by colony forming units (CFUs) counting and crystal violet staining. The treatment efficacy of 3 min rinsing of 0.12% chlorhexidine (CHX) for the adhered bacteria was evaluated by the ratio of the remaining microbial viability. All the results were compared with those of mechanical polishing (MP) and sandblast-large grit and acid-etching (SLA) surfaces.

Results: MP and SLA surfaces demonstrated a micro-scale structure, while TNT surfaces showed a nano-scale structure. TNT-A surface demonstrated the lowest surface roughness (Ra $0.792 \pm 0.080\,\mu m$) while SLA specimen showed the highest (Ra $1.618 \pm 0.131\,\mu m$). The water static contact angles of 3 surfaces were smaller than 90° and that of TNT-A surface was the lowest ($15.89 \pm 1.05°$). After 6 h, both SLA and TNT-A surfaces revealed higher cell adhesion than MP surface. After 1 d culture, the cell adhesion values of the 3 surfaces were similar. After 3 d, the values of TNT-A surface was the greatest, followed by SLA surface and MP. At day 5, TNT-A surface demonstrated the highest cell proliferation. At day 7, the ALP activity of the cells on SLA and TNT-A surfaces was significantly higher than those on MP surface after 7 d culture. The ALP activity was the highest on TNT-A surface, followed by SLA surfaces, and MP surface revealed the lowest after 14 days. The lowest amount of initial 2 h microcosm adhesion was presented on the MP and TNT-A surfaces, but at 5 d, no significant difference was revealed on all the 3 surfaces. The remaining viability of microcosm biofilm on TNT-A and MP surfaces after CHX rinsing was significantly lower than that on SLA surface.

Conclusions: TNT-A surface had favorable osteogenic properties, lower bacterial adhesion, and superior antibiotic treatment efficacy.

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2

Effect of curing modes on polymerization properties of dual-cured resin cements

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Purpose/Aim: To investigate the effect of different curing modes on degree of conversion, micro-hardness, shrinkage strain and shrinkage stress of dual-cured resin cements.

Materials and methods: Four curing modes, immediate light curing (IML), intermittent light curing (ITL), delayed light cuing (DL) and chemical curing (NL), were used to cure two dual-cured resin cements RelyX Unicem (RX) and PermaCem 2.0 (PC). Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR) was used to measure the degree of conversion (DC). Indentation method was used to get the micro-hardness (MH). Digital image correlation (DIC) was

Table 1 – Curing modes used in the study.

Curing modes	Illustrations
Immediate light curing mode	Irradiate for 20 s
Intermittent light curing mode	Irradiate for 2 s, wait in dark for 1 min, irradiate for 20 s
Delayed light curing mode	Wait in dark for 2 min, irradiate for 20 s
Chemical curing mode	No irradiance, keep in dark

used to determine shrinkage strain (SN). And uniaxial tensile configuration was used to gain shrinkage stress (SS). SPSS 13.0 was applied to analyze the interactive effect of material types and curing modes with two-way ANOVA with Bonferroni method to adjust the effect of curing modes and paired student t-test to analyze the effect of different material types ($\alpha = 0.05$). Correlation was established between SN and DC, MH and SS ($R^2 = 0.9$).

Results: The DC was 36.80–52.88% for RX and 62.64–85.50% for PC. Under different curing modes, there was no significant difference for RX and IML, INL > NL for PC. Between two materials, PC > RX under every curing mode. The MH was 5.51–31.27 for RX and 18.08–28.40 for PC. Under different curing modes, IML, INL, DL > NL for RX and DL > IML, INL, NL for PC. Between two materials, RX > PC under IML and DL and PC > RX under NL. The SN was 1.28–3.41% for RX and 2.43–4.20% for PC. Under different curing modes, IML, INL, DL > NL for RX and PC and INL > DL for RX. Between two materials, PC > RX under IML, INL and NL. The SS was 1.52–3.11 MPa for RX and 2.16–3.08 MPa for PC. Under different curing modes, IML, INL > NL and IML > DL for RX and IML > DL, NL for PC. Between two materials, PC > RX under NL. There was positive correlation between SN and DC, MH and SS.

Conclusions: Curing modes have influence on polymerization properties of dual-cured resin cements. Light irradiation could increase DC, MH and SN. Intermittent and delayed light curing could decrease the SS. Resin cements with different components react differently to different curing modes on polymerization properties. Positive correlation could be established between SN and DC, MH and SS.

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3

Evaluation of etched enamel using QLF

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Purpose/Aim: The purpose of this study is to evaluate the effect of etching on ground and unground enamel using Quantitative light-induced fluorescence (QLF).

Materials and methods: Thirty extracted incisors were used. One-half of the labial surface of each tooth was ground with #400-grit SiC paper. The other half was left intact. Ground and intact surfaces were coated with nail varnish leaving rectangular windows of enamel uncoated. A 32% phosphoric acid gel was applied for 15, 30, 60 s. Mineral loss in terms of percentage of fluorescene (delta F) were assessed by QLF at baseline and after etching. Effect of etching time and mechanical pretreatment were analyzed with independent *t*-test, one-way ANOVA.

Results: Grinding the enamel before etching for 15 s, 30 s had significant effect on demineralization. But there was no significant difference between ground and unground enamel for 60 s etching. ΔF values decreased with increased etching time. But there was no significant difference between 15 s and 30 s, 30 s and 60 s in both ground and unground enamel.

Conclusions: Demineralization after acid-etching can be quantified using QLF. The result indicated that the degree of demineralization increases with etching time and mechanical pretreatment.

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4

Intrinsically antibacterial adhesive systems based on polymeric derivative of eugenol

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Purpose/Aim: Imparting intrinsic antibacterial activity to root canal dentin bonding may help disinfecting the cavity from the residual bacteria and lowering the risk of reinfection and secondary caries. This preliminary study examined the curing properties, wettability, bonding ability and the antibacterial activity of two different dual-polymerising adhesive systems incorporating EgMA antibacterial monomer for their application in endodontic restoration bonding.

Materials and methods: EgMA monomer derived from eugenol was added at 20 wt% into the formulation of the single-component self-etch, Clearfil Universal Bond™ (CUB) adhesive and into the catalyst and the adhesive components of the total-etch Adper Scotchbond™ multi-purpose plus (SBMP) adhesive systems. The degree of conversion (DC) was calculated from FTIR spectra, glass transition temperature (Tg) determined by (DSC), water sorption and solubility were measured gravimetrically, and surface free energy (SFE) via contact angle measurements. The bonding performance to coronal and middle root canal dentin was assessed through push-out bond strength test after filling the canals with a composite core material and the surface integrity was observed using a confocal laser scanning microscope (CLSM). The standard agar diffusion test (ADT) was used to identify the sensitivity of three endodontically pathogenic bacteria, *E. faecalis*, *S. mutans* and *P. acnes* to uncured EgMA modified adhesives. Dental adhesives without modification were used as controls.

Results: Compared with the controls, both EgMA containing adhesives had comparable Tg and DC measured 24 h post curing. The incorporation of EgMA lowered

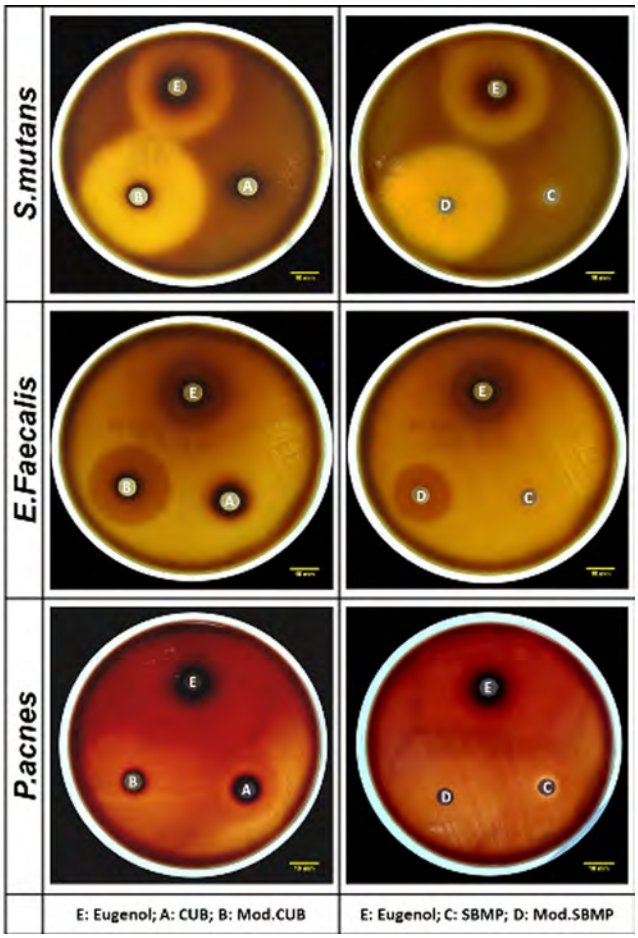

Fig. 1

polymerization exotherm, water sorption, solubility and enhanced the hydrophobic character of these adhesives. The total push-out bond strength of the CUB and SBMP employing EgMA-containing adhesive components were not significantly different from those of the controls ($p > 0.05$). The modification of self-etch adhesive system enhanced the bond strength in the middle region of the roots canal. CLSM examination showed the integrity of the resin-dentin interfaces. ADT showed that the CUB control exhibited some inhibition against *P. acnes* and *E. faecalis*, whilst the SBMP control produced inhibition against *P. acnes* only. The sizes of these inhibition zones were significantly smaller than those produced with EgMA modified adhesives, which demonstrated inhibition against all of the bacteria tested, exhibiting statistically significant differences among them following the order *P. acnes* > *S. mutans* > *E. faecalis* ($p < 0.05$).

Conclusions: The inclusion of EgMA could endow dental adhesives with obvious cavity disinfecting effects without influencing their curing and bonding ability to root canal dentin, indicating the usefulness of their application in endodontic restorations.

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5

Therapeutic bonding approaches to remineralize dentin-bonded interfaces

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Purpose/Aim: Dental adhesives (DAs) have improved substantially over the past few years. However, such materials may create hybrid layers characterized by enzymatic collagen degradation and polymer hydrolysis. It has been advocated that mineral-depleted dentin can be remineralized if alternative therapeutic bonding strategies are employed. However, effective hybrid layer remineralization seems to be achievable only when using "smart" analogues of phosphoproteins along with ion-releasing materials. Thus, the aim of this study was to show the ability of innovative bioactive/biomimetic bonding approaches in remineralizing and improve the durability of mineral-depleted bonded-dentin interfaces.

Materials and methods: Several experimental therapeutic approaches were tested in attempt to remineralize mineral-depleted (i.e. acid-etched) resin-dentin interfaces. For instance, dentin specimens were first air-abraded with bioactive glass 45S5 (BAG) or BAG/poly-acrylic acid (PAA), and then restored with fluoride-releasing glass ionomer cements (GIC or RMGIC). Moreover etch-and-rinse and self-etch adhesives containing different ion-releasing micro-fillers were formulated and then applied onto demineralized dentin, which was pretreated with or without different biomimetic primers doped with sodium trimetaphosphate, poly-aspartic acid (PASA) and/or PAA. All bonded-dentin specimens were aged in simulated body fluids (SBF: 24 h/6 months). Specimens were finally processed and assessed for microtensile bond strength, AFM nano-indentation, XRD, FTIR-ATR, FEG-SEM (fractographic analysis), TEM, dye-assisted confocal microscopy and Raman microscopy. All necessary statistical analysis were performed on quantitative data ($p < 0.05$).

Results: Air-abrasion performed with BAG and BAG-PAA created a therapeutic "bioactive smear-layer-covered surface" for bonding procedures, which reacted with SBF and remineralized the resin-dentin interfaces via apatite precipitation. These outcomes were especially evident when BAG and BAG-PAA air-abraded dentin specimens were bonded using GIC or RMGIC. Furthermore, the results of this study showed that the use of biomimetic primers in combination with the experimental ion-releasing adhesives produced "bottom-up" dentin remineralization that restored the modulus of elasticity of water-rich/resin-sparse dentin-bonded interfaces. Moreover, these latter specimens showed no bond strength reduction after 6 months of storage in SBF.

Conclusions: Alternative therapeutic bonding and/or pretreatments procedures, as well as the use of innovative resin-based materials such as ion-releasing flowable composites in combination with "smart" adhesive systems containing analogues of dentin phosphoproteins represent

a suitable strategy to remineralize and prevent degradation of resin-dentin bonds to enhance the longevity of aesthetic composite-restorations.

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6

TiF_2 incorporated into adhesive system: Physicomechanical characterization and bonding stability

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Purpose/Aim: The purpose of this study were to evaluate the long-term bond strength of TiF_4 incorporated into a two-step self-etching (primer and bond) adhesive system to dentin, the mode of fracture after microtensile bond strength tests, the micromorphological features of the hybrid layer formed after using TiF_4-incorporated adhesives, the degree of conversion of the TiF_4–incorporated adhesives, and their flexural strength.

Materials and methods: Thirty-two dentin surfaces received four different treatments ($n = 8$): CL (Clearfil SE Bond); TiF_4CL (TiF_4 aqueous solution + CL); TiF_4P + B (TiF_4 incorporated into the P); P + TiF_4B (TiF_4 incorporated into the B). After 24 h, 6 months and one year, respectively, sticks from the bonded area were submitted to microtensile bond strength tests and the failure mode was recorded. The slabs obtained from the dentin-resin interfaces of each group were examined under SEM. Degree of conversion and flexural strength were evaluated to determine whether the TiF_4 influenced the P and the B properties. Two-way ANOVA for repeated measures was applied to the microtensile bond strength data, and one-way ANOVA and Tukey tests to the degree of conversion and flexural strength results.

Results: There were no differences in microtensile bond strength among the groups ($p = 0.818$) regardless of time; the microtensile bond strength in each group was not significantly affected over time ($p = 0.061$). The failure mode showed mostly adhesive failures in all the groups and time periods, followed by cohesive in resin. Incorporation of TiF_4 into the P or B significantly affected flexural strength ($p = 0.004$). The degree of conversion for TiF_4P + B was not significantly different from that of CL, and was higher than that of TiF_4B.

Conclusions: TiF_4 incorporated into a two-step self-etching adhesive system presented similar microtensile bond strength and failure mode to dentin over time, compared with CL, although the number and length of the resin tags in the hybrid layer were lower than those of CL. TiF^{4-} incorporation affected the flexural strength of the adhesive. Degree of conversion was affected only when incorporating TiF_4 into the B.

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Dental radiometers – Comparison of accuracy

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Purpose/Aim: The power output of a light curing unit (LCU) is a critical factor in polymerization of light cured resin composites (RC). A negative correlation between compromised power output (PO) of a LCU and mechanical properties of RC has been cited in the literature. Therefore, it is necessary for a dental office to timely measure the power output of LCUs. Historically, dental radiometers available in the market have often measured PO with limited accuracy.

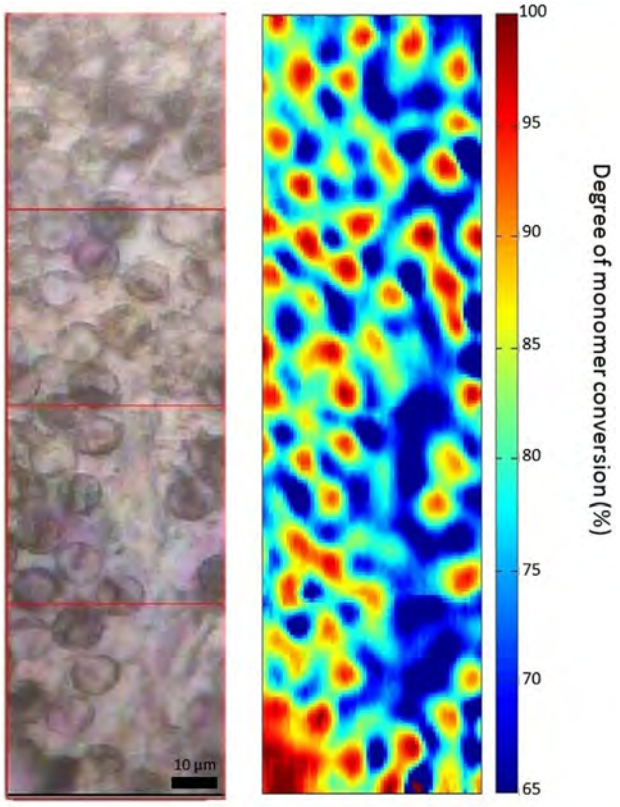
Objective: This study compared a contemporary dental radiometer with a laboratory standard for accuracy.

Materials and methods: Four LED curing lights (Table 1) were tested. Bluephase G2 and Bluephase 20i were tested at high and low curing modes. The power output of the listed LCUs was measured using Bluephase® Meter II (BM2) (Ivoclar Vivadent Inc.) and a laboratory standard radiometer- Integrating Sphere (IS). The LCUs were positioned in a metal stand to avoid an effect of LCU movement during measurements. Ten measurements were taken for each LCU per radiometer. The manufacturer of BM2 claims the measuring accuracy with a tolerance of ±10% compared to the measurement with IS. The percentage difference in the measured power output for tested LCU was calculated and compared with the manufacturer's claim.

Results: See Table 1.

Table 1 – List of tested LED light curing units and their power outputs (mW) using radiometers.

LCU	Manufacturer	Integrating Sphere (IS)	Bluephase® Meter 2 (BM2)	((IS-BM2)/IS)%
		Power Output (mW) (Mean ± SD)		
Bluephase® G2	High-Mode	789.9 ± 3.2	763.5 ± 2.7	3.3
	Low-Mode	430.2 ± 1.7	435.3 ± 2.5	−1.2
Bluephase® 20i	High-Mode	534.0 ± 1.7	515.1 ± 2.6	3.5
	Low-Mode	265.7 ± 0.9	272.0 ± 2.0	−6.3
Bluephase® Style	Ivoclar Vivadent Inc.	609.1 ± 5.4	557.0 ± 2.6	8.6
Elipar™ DeepCure-S	3M ESPE	785.4 ± 3.9	711.9 ± 3.2	9.4



(a) A microscope image of 8µm (diameter) monodisperse silica microspheres embedded into a 60/40wt% (Bis-GMA/TEGDMA – CQ initiator) resin matrix. **(b)** A synchrotron mid-FTIR map (IRENI-SRC, WI, USA) of the same region. The degree of monomer to polymer conversion is shown as a heat map, with red and blue areas representing high and low conversion respectively.

Fig. 1

Conclusions: Within the limitations of this study, Bluephase Meter II met the manufacturer's claim and accurately measured the power output of tested LCUs.

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The influence of FRCs positioning underneath CAD/CAM composite resins

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Purpose/Aim: Investigate the reinforcing and stress-breaking effects of long bidirectional E-glass fiber reinforced composites (FRCs) applied underneath CAD/CAM resin composites.

Materials and methods: A total of 120 specimens (9 mm × 5 mm, 4.2 mm-thick) were prepared. Specimens were divided in four Groups ($n = 30$): monolithic CAD/CAM resin composite (Cerasmart, GC) (Group A, control), composite resin base (Genial Posterior, GC) in a layer of 1 mm (Group B), 2 mm (C) and 3 mm (D) underneath two bonded layers of bidirectional e-glass FRC (Dentapreg, ADM A.S.). Over these FRCs sheets, CAD/CAM composite resin slices of 3 mm (Group B), 2 mm (C), 1 mm (D) were bonded. In each group, half of the specimens ($n = 15$) (Subgroups As, Bs, Cs, Ds) were loaded to fracture in a universal testing machine. Maximum fracture loads were recorded in Newtons and data were analyzed using Weibull analysis. The other half of specimens of each group ($n = 15$) (Subgroups Ad, Bd, Cd, Dd) were submitted to cyclic isometric stepwise loading (5 Hz) until completion of 105,000 cycles or failure after 5000 cycles at 500 N, followed by stages of 20,000 cycles at 750 N, 1000 N, 1250 N, 1500 N and 1750 N. Results were statistically analyzed by Kaplan–Meier life survival analysis and log rank test ($p = 0.05$). All fractured specimens were analyzed using stereomicroscope and SEM and modes of failure were determined (vertical fracture-split, partially deviated fracture or completely deviated fracture).

Results: The mean static loads (N) registered were: A – 2904.13 (159.33); B – 2716.55 (307.14); C – 2539.96 (153.17); D – 2263.17 (167.96). Statistically significant differences were found between all groups ($p < 0.05$). The differences in survival after fatigue between groups was statistically significant, except between Groups A and C ($p = 0.86213$). The mode of fracture was always catastrophic (split) in Group A (without fibers) while in Groups B and C the crack was mainly partially deviated, and in Group D it was mostly totally deviated, with few differences between fractures occurred under static or dynamic loads. In all deviated fractures, the fractographic analysis confirmed the stress-breaking effect of the FRCs layer.

Conclusions: The load bearing capacity of the specimens and their fatigue resistance seemed to be more related to the thickness of the CAD/CAM resin composite under the loading sphere more than to the subjacent FRCs layer. However, the fiber reinforcement protected the resin composite substructure leading to more favorable fracture patterns.

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Wear evaluation of dental Y-TZP opposing human enamel

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Purpose/Aim: The aim of this systematic review was to assess enamel wear on teeth opposing zirconia restorations by analyzing in vitro studies that quantify the wear of enamel opposing zirconia ceramics. Additionally to evaluate factors related to the wear of the natural tooth opposing zirconia ceramics.

Materials and methods: Five electronic databases were searched without limitations. Terms "antagonist*", "enamel", "wear" and "zirconi*" were used. All titles revealed by the electronic search were screened according to the following inclusion criteria: (1) In vitro studies; (2) Use of Y-TZP ceramic;

(3) Evaluation of the antagonist's enamel. In addition to the inclusion criteria the following exclusion criteria were applied: (1) Veneered zirconia specimens; (2) Absence of enamel wear evaluation. Study selection: Titles and abstracts were initially screened, and those that fulfilled the inclusion criteria were selected for a full-text assessment. Studies which evaluated only the material wear were not included. The studies were analyzed with regard to the wear mimicking device and wear method, its testing parameters, ceramic preparation, finishing technique, antagonist's enamel wear and zirconia wear. When information was unavailable or limited, authors were contacted in order to obtain missing information.

Results: The database search strategy retrieved 142 potentially eligible studies. After removing the duplicate studies, 62 studies were obtained. Titles and abstracts that fulfilled the inclusion criteria were selected for a full-text assessment (25). Seven laboratory studies met the inclusion criteria. Additionally, reference lists from the finally selected studies were also screened.

Conclusions: There was a large variation in relation to: wear test method quantification, applied force, lateral movement, number and frequency of cycles, number of specimens, and enamel specimen preparation. In all studies, enamel wear rates were lower against polished zirconia. Differences in the test methods did not allow for comparisons of the wear rates among the studies. Polishing the surface is recommended for a full contour zirconia restoration because polished zirconia presents favorable wear behavior opposing natural teeth.

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Anti-caries potential of high-fluoride dentifrices combined or not with tricalcium-phosphate

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Purpose/Aim: Fluoridated dentifrices have been advocated as an interesting strategy to prevent the development caries, but evidence regarding high-fluoride (F) dentifrices to prevent dentin root caries is still unclear. This in vitro study evaluated the effect of dentin demineralization inhibition of high-F dentifrices (5000 μg F/g; Colgate Prevident® – CP and 5000 μg F/g + tricalcium-phosphate; Clinpro® – CL, 3M ESPE) compared with a conventional dentifrice (1450 μg F/g; control group Colgate Total 12® – CT).

Materials and methods: This study involved root dentin (4 × 4 × 2 mm) as experimental units, which were prepared from bovine incisors. These specimens were selected by surface hardness (SH) presenting 35.28 ± 0.59 KHN. The specimens were randomly distributed ($n = 15$) and subjected to one of the following treatments: CT, CP and CL, during 5 min, twice a day. Daily challenges consisted in cycles of 8 h in the demineralizing and 16 h in the remineralizing solution, during 7 days at 37 °C. The treatments were conducted with dentifrices/water slurries (1:3, w/w), simulating the dilution that occurs in the oral cavity when tooth-brushing. The response variable was based on the percentage of surface hardness loss (%SHL) and cross-sectional hardness (CSH). Data of %SHL and ΔS were subjected to one-way ANOVA followed by Tukey's test and Kruskal–Wallis, respectively ($p \leq 0.05$).

Results: The data of %SHL/ΔS were, respectively: CT = 66.79 ± 3.51A/752.11 ± 297.14*; CP = 31.43 ± 4.43B/57.65 ± 22.92**; CL = 32.01 ± 10.32B/162.91 ± 81.10**. For both analyses, high-F dentifrices combined or not with tricalcium-phosphate were able to promote reduced demineralization of dentin compared to conventional fluoridated dentifrices.

Conclusions: High-F dentifrices are effective to protect dentin from demineralization.

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Influence of Er:Yag laser cavity preparation on micromorphology and adhesion

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Purpose/Aim: Er:YAG laser with varied pulse duration allows for cavity preparation by means of cold ablation. Analyzing the micromorphological features of irradiated dentin tissue is key to predict the bond strength to composites. This study evaluated the influence of different pulse durations on the adhesive interface morphology and microtensile bond strength (μTBS) of a resin composite, when cavity preparation was performed by Er:YAG laser.

Materials and methods: Forty two human freshly extracted molars were cut to expose middle dentin and then polished with #600 grit silicon carbide for 60 s to create a standardized smear layer. Afterwards, specimens were randomly assigned into 3 groups according to the cavity preparation method ($n = 11$): G1 (control – high speed conventional preparation); G2 (200 mJ/20 Hz/50 μs) and G3 (200 mJ/20 Hz/300 μs). The laser wavelength (2.94 μm) was set with cooling spray at 4A/6W. Thereafter, self-etch universal adhesive (3 M/ESPE) was applied to a flat occlusal dentin surface according to manufacturer's instructions and a resin composite (Filtek Z350 3M/ESPE) block (5 mm-high) was built in order to produce the specimens. After 24 h storage in distilled water at 37 °C, the composite/dentin sticks were prepared (1 mm^2) and μTBS test was performed. In addition, nine dentin discs per group were prepared to perform the analysis of the behavior of collagen fibrils by second harmonic generation (SGH) (at 380 nm excitation) and hybrid layer formation/resin tags extension using confocal laser (at 800 nm excitation). In both analyses, fluorochrome Rhodamine B was added to the adhesive.

Results: Statistical analysis was performed by ANOVA and post-hoc Tukey test ($\alpha = 0.05$). G1 displayed statistically higher microtensile bond strength values when compared to the values obtained for the other groups ($p = 0.00$). Group results for μSBS were: G1 (51.14 ± 7.16^a); G2 (29.17 ± 7.09^b); G3 (23.53 ± 4.5^b). When irradiated groups were compared, no statistical differences were observed, indicating lower μTBS values regardless of the pulse duration used. All groups

showed a thin hybrid layer and small resin tags. Such features are in accordance to those usually found for self-etch adhesives. However, irradiated groups showed deeper penetration of the adhesive into dentin, resulting in longer resin tags, when compared to the control group. For SGH, some laser groups revealed superficial changes immediately below the irradiated surface. This alteration was not detected in G1.

Conclusions: Cavity preparation with Er:YAG decreased bond strength values when compared to the control group. Although hybrid layer and resin tags were formed in all experimental groups, the organic matrix alteration was only observed for groups treated with laser with larger pulse widths.

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Effect of Er:Yag laser pulse width on dentin bond strength

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Purpose/Aim: Er:YAG lasers with controlled pulse duration can interact with dentin by means of cold ablation. Analyzing the micromorphological features analysis of irradiated dentin is key to predict adhesion. To investigate the effect of Er:YAG laser pulse width on collagen micromorphology and on bond strength of a composite resin to the irradiated tissue.

Materials and methods: Sixty caries-free human molars were cut to obtain middle flat dentin discs which were embedded in acrylic resin and polished with #600grit silicon carbide paper for 60 s to obtain a standardized smear layer. Samples were randomly divided into 4 groups according to Er:YAG laser surface pre-treatment ($n = 12$): G1 (control without laser); G2 (80 mJ/2 Hz/50 μs); G3 (80 mJ/2 Hz/300 μs) and G4 (80mJ/2 Hz/600 μs). The laser wavelength was 2.94 μm, with cooling protocol adjusted to 4A/6W. Next, a self-etch adhesive system (Scotchbond Universal-3M/ESPE) was applied and 8 cylinders of resin composite (Z350-3M/ESPE) were built to produce the specimens, which were stored at 37 °C for 24 h, and then subjected to a shear bond strength test (SBS) in an universal testing machine. Three additional dentin discs per group were prepared to investigate the behavior of collagen fibrils by means of second harmonic generation (SGH) (at 380 nm excitation) and hybrid layer formation/resin tags extension using confocal laser microscopy (at a 800 nm excitation). In both analyses, fluorochrome Rhodamine B was added to the adhesive in a concentration of 0.0016 g/ml.

Results: Statistical analysis was performed by ANOVA and post-hoc Tukey test ($\alpha = 0.05$). Group results for SBS were ($p = 0.00$): G1 (26.17 ± 3.78^{a}); G2 (22.14 ± 2.86^{b}); G3 (21.24 ± 3.03^{b}); G4 (20.61 ± 1.85^{b}). When mean SBS values of irradiated groups were compared, no statistical difference was observed, indicating that, regardless of the pulse width, SBS values were lower for all irradiated groups in comparison to the control, which showed the highest SBS mean value. Results from confocal microscopy showed that all groups have a thin hybrid layer and almost no resin tags. However, irradiated groups showed a deeper penetration of the adhesive into dentin, resulting in longer resin tags, especially in comparison to the control group. For SGH, G1 and G2 showed no alterations of collagen fibrils and organic matrix while G3 and G4 showed minimal alteration of the organic matrix below the irradiated surface.

Conclusions: Er:YAG laser pretreatment on dentin decreased shear bond strength to resin composite, although hybrid layer and resin tags were formed in all experimental groups. Collagen fibers disruption and organic matrix alteration were only observed in groups treated with higher pulse width.

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Ammonium based methacrylate into dental adhesive for bonding metal brackets

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Purpose/Aim: This study aimed to evaluate the effect of addition of ammonium based methacrylate to experimental adhesives used in the cementation of orthodontic brackets on the bond strength of these brackets before and after cariogenic challenge, and the development of carious lesions around the bonding interface.

Materials and methods: Adhesive blends were formulated using the monomers HEMA and BisGMA (60/40 by weight), and solubilized into 30 wt% of ethanol. The antibacterial monomer 2-(methacryloyloxy)ethyl]trimethylammonium chloride (MADQUAT) was added at 5 or 10 wt%, while adhesive without MADQUAT was used as control. Degree of conversion (DC) was measured using FT-IR spectroscopy ($n = 3$). The adhesives were used to lute brackets to enamel of human premolars associated to composite Transbond XT. Biofilm from microcosm model was cultivated in half of specimens under cariogenic challenge for 5 days, while the remaining specimens were stored in distillated water for the same time. Brackets were submitted to shear bond strength test followed by failure mode analysis ($n = 10$). The fractured specimens were transversally sectioned and the internal hardness of enamel was measured around the brackets to calculate the integrated mineral loss (ΔS). Data were submitted to 2-way ANOVA (1-way for DC) and Tukey's post hoc test ($\alpha = 0.05$). The effect of cariogenic challenge and adhesive on failure mode was analyzed by Chi-square test.

Results: The addition of MADQUAT affected the DC ($p = 0.008$), while the control adhesive showed the lowest values of DC. Using 10% of MADQUAT resulted in lowest values of bond strength, while no difference was observed between the concentration of 0% and 5%. The cariogenic challenge also did not alter significantly the distribution of failure mode (chi-square, $p = 0.090$). In opposite, the concentration of ammonium based methacrylate altered the distribution of

failure mode (chi-square, $p = 0.008$), while using higher concentration resulted in highest amount of type I failure. In absence of cariogenic challenge (control), no difference was observed between the different concentrations of MADQUAT. In opposite, adhesives containing 10% of MADQUAT resulted in lowest demineralization around the bracket, while no difference was observed between the other concentrations when the specimens were submitted to cariogenic challenge. Except for the concentration of 10%, the cariogenic challenge significantly increased the demineralization around the brackets.

Conclusions: MADQUAT was effective to reduce the ΔS only when added to adhesive at concentration of 10% despite the reduction on bond strength.

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Shear bond strength and antibacterial properties of different luting cements

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Purpose/Aim: The purpose of this *in vitro* study was to compare four different kinds of dental luting cement's [Conventional glass ionomer cement (GIC), Resin modified GIC, Glass Carbomer and Dual Cure Resin Cement] shear peel bond strength, assess the remnant characteristic and antibacterial characteristics.

Materials and methods: In the shear peel bond strength test part of the study, stainless steel bands were cemented to 80 extracted permanent molar teeth randomly by using one of four tested cements (20 per group). The force needed for debanding was evaluated by using a universal testing machine. The amount of cement remaining on the teeth after band removal was scored. The antibacterial effect of cements on selected bacteria (*S. mutans* and *C. albicans*) was tested with agar diffusion test.

Results: Molar bands cemented with Dual cure resin cement showed the highest and bands cemented with Glass carbomer cement showed the lowest shear peel bond strength among all luting cements. Conventional GIC specimens failed mostly at the enamel/cement interface. As for the antibacterial effects, resin modified GIC cement group was the only cement which showed antibacterial effect on *C. albicans*. All cements showed some antibacterial effect on *S. mutans*, dual cured resin cement being the least effective.

Conclusions: The findings show that different types of luting cements may be preferred according to the characteristics of the individual.

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BisGMA/TEGDMA based material with antibacterial activity

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Purpose/Aim: The aim of this study was to develop a composite material with antibacterial activity using MMT loaded with chlorhexidine (CHX). For that it was used a BisGMA/TEGDMA matrix, and added low concentration of MMT/CHX. The aim was to evaluate the drug release capacity of MMT, and not to provide reinforcement.

Materials and methods: Six experimental composites were made with organic matrix of BisGMA/TEGDMA in equal proportions by weight. The composites received organophlizated montmorillonite with or without CHX. The concentrations were 2.5%, 5% or 10% by volume. Degree of conversion (DC) was evaluated using FTIR (peak 6165 cm^{-1}; $n = 5$). Specimens for flexural properties ($10 \times 2 \times 1$ mm) were half immediate tested (24 h) and half storage (2 months). Elastic modulus (E) and flexural strength (FS) was measured using the three point bending test ($n = 10$). Inhibition halo was used to test the antibacterial activity against *Staphylococcus aureus*, *Streptococcus mutans*, and *Porphyromonas gingivalis* ($n = 5$ for each bacteria). The inhibition of biofilm formation (BF) was evaluated by inserting polymerized disc of composite in to a culture media colonized with *Streptococcus mutans* ($n = 10$). For this test a control group of a commercial composite was also tested. The release of CHX was measured using ultraviolet (255 nm) during 10 days ($n = 5$). The data of degree of conversion was analysed using Kruskal–Wallis/Mann–Whitney, and the other variables using

Table 1 – Shear Peel Bond Strength values for tested materials.

Cement	N	Mean (MPa)	Std. Dev.	Median	Int. range	Min	Max
Ketac-Cem	20	1.20[a,*]	0.36	1.19	0.52	0.45	1.75
Unitek Multicure Glass Ionomer	20	1.19[a]	0.33	1.17	0.41	0.68	1.97
Glass Carbomer	20	0.97[b]	0.42	0.94	0.52	0.25	2.10
Rely X	20	1.84[c]	0.67	1.65	1.15	0.83	3.25

* Different letters indicate significant difference ($P < 0.05$). Lower-case letters indicate differences in vertical directions.

two-way ANOVA/Tukey, always considering a global level of significance of 5%.

Results: Data for DC ranged from 46% to 60%, the group with 10% MMT/CHX presented a statistically lower value than the others. Data for E and FS at 24 h the 10% concentration presented the higher values, but after 2 months of storage this concentration showed the lower values of all when the CHX was present. For the three bacteria tested the composites with CHX loaded presented inhibition of growth for all concentration, except for 2.5% that did not inhibited the growth of *P. gingivalis*. BF was lower for the groups with CHX, but when compared to the commercial composites, all groups presented BF, even those without CHX loaded. All concentrations presented release off CHX during all the 10 days analyzed.

Conclusions: Within the limitation of this study it can be concluded that: all concentrations tested presented release of CHX and reduced BF. All concentration presented antibacterial activity for the three bacteria tested, except for 2.5% that did not inhibited the growth of *P. gingivalis*. The concentration of 10% resulted in a reduction of DC and the flexural properties after 2 months of storage.

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Light transmittance through esthetic monolithic CAD/CAM materials

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Purpose/Aim: To determine the amount of light (360–540 nm) passing various monolithic CAD/CAM-materials, in dependency of material thickness, initial curing unit irradiance, and exposure distance (distance between curing unit and specimen's surface).

Materials and methods: Nine different CAD/CAM monolithic materials were selected: TC: TelioCAD (PMMA-based), VCT: VITA CAD-Temp (PMMA-based and 10% filled with prepolymers), TEC: exp. nanocomposite (filled composite), LU: LAVA Ultimate (filled composite), VE: VITA ENAMIC (interpenetrating network ceramic), VM: VITA Mark II (feldspar ceramic), IEC: IPS EmpressCAD (leucite glass-ceramic), IEM: IPS e.max CAD (lithium disilicate glass-ceramic), and CD: CeltraDuo (zirconia-reinforced lithium silicate ZLS). CAD/CAM blocks were cut using a low-speed diamond saw in 1 and 2 mm thick slices ($n = 10$) resulting in 180 specimens.

The transmitted irradiance was assessed in real time by means of a Spectrometer and a blue-violet LED unit (VALO; Ultradent Products Inc), which was used in three curing programs (standard power, high power, and plasma). The curing unit was placed directly on specimen's surface as well as at 2 and 4-mm distances from it. Data were analyzed using a multivariate analysis and 1-way ANOVA with post-hoc Scheffé test ($p < 0.05$).

Results: The highest influence on the transmitted irradiance was exerted by the curing mode ($nP^2 = 0.991$), closely followed by specimen thickness ($nP^2 = 0.989$), CAD/CAM material ($nP^2 = 0.966$), and exposure distance ($nP^2 = 0.904$). All binary combinations of the above-mentioned parameters were also significant ($p < 0.05$). The highest transmitted irradiance was measured for VM and LU, followed by VCT and IEC, while the lowest values showed VE, followed by IEM and CD. The highest transmitted irradiance was recorded by exposing the material to the plasma mode, followed by the high and standard power modes. The transmitted irradiance related to the incident irradiance amounted only 16% to 39.2% by passing 1-mm thick slices, while only 4.5% to 19.4% for 2-mm thick slices. Fewer difference were measured when the curing unit was placed at 0 or 2-mm from the specimen's surface, while the transmitted irradiance was lower at an exposure distance of 4-mm.

Conclusions: Transmitted irradiance through VITA ENAMIC restorations might not allow for sufficient light passing through the material. Less light-sensitive dual-curing cements must therefore be used for cementation.

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WITHDRAWN

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Effect of shade and ageing on strength of translucent Y-TZP

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Purpose/Aim: To evaluate the effect of shade and ageing on the strength of a translucent yttria-stabilized tetragonal zirconia (Y-TZP) for monolithic restorations.

Materials and methods: A granulated Y-TZP powder (Zpex, Tosoh) was used to produce translucent zirconia specimens. Pigmentation of specimens was achieved by combining seven commercial dyeing solutions (Lava Frame Shade, 3M-ESPE), which were mixed according to manufacturer's instructions to achieve eight distinct shades (Table 1) of the Vita Classical guide (Vita Zahnfabrik). A control group without pigmentation was also tested. The ceramic powder was pressed to form discs (2.0 mm in thickness/12 mm in diameter) by uniaxial pressing (112 MPa/30 s) with subsequent cold isostatic pressing (200 MPa/30 s). These discs were pre-sintered (furnace N1100, Jung) at 900 °C for 2 h (heating rate: 5 °C/min). Presintered discs were immersed in a staining solution for 2 min. After pigmentation, final sintering occurred at 1500 °C for 1 hour (heating rate: 8 °C/min, Furnace Hot Spot 110, Zircar). The biaxial flexural strength was determined using the piston-on-three-balls design, in distilled water (37 °C at 0.5 mm/min). Flexural strength was calculated according to ASTM-F-39478. Half of the specimens of each shade ($n = 10$) had their strength measured after being aged in an autoclave (AHCM-10, Sercon) for 5 h at 134 °C/2 bars. The data were analyzed by means of two-way analysis of variances and Tukey's test with global significance level of 5%.

Table 1

Ageing	Shade								
	Control	A2	A3	B2	B3	C2	C3	D2	D3
No	637.6 ± 55.1A,B	548.4 ± 130.3A,B,C	504.2 ± 120.4A,B,C	685.3 ± 57.3A	605.8 ± 70.7A,B	612.3 ± 119.2A,B	505.3 ± 115.3A,B,C	414.1 ± 242.0B,C	619.9 ± 27.2A,B
Yes	440.3 ± 69.3A,B,C	572.0 ± 168.6A,B,C	460.8 ± 109.4A,B,C	579.8 ± 97.3A,B,C	490.4 ± 114.1A,B,C	582.6 ± 74.9A,B,C	338.4 ± 149.2C	559.9 ± 51.6A,B,C	416.3 ± 74.8B,C

Results: Mean flexural strength values and standard deviations as a function of the shade and ageing are shown in the Table 1. Ageing did not significantly affect the strength results for any of the colors tested, although a numeric decrease in the strength values was noted for several experimental groups. The color of the specimens affected the strength values, since for non-aged specimens, the flexural strength obtained for shade B2 (685.3 ± 57.3 MPa) was significantly higher than that obtained for shade D2 (414.1 ± 242.0 MPa). However, the effect of color on the strength of the specimens was not noticed for aged specimens.

Conclusions: The in vitro ageing protocol did not affect the flexural strength of the translucent Y-TZP for any of the shades tested. The coloring solutions used to produce specimens with different shades interacted with the material microstructure and affected the flexural strength of the translucent Y-TZP, since for two different shades of non-aged specimens (B2 and D2), the flexural strength was significantly different.

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Effects of pigments on the translucency of dental composite resins

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Purpose/Aim: The objectives of this study were to examine the effects of different colored pigments on the translucency of experimental dental composite resins.

Materials and methods: 12 samples of composite resins with different pigment concentrations, ranging from 0% to 0.08% (red, yellow and red + yellow mixed) were fabricated ($N = 3$) and cured. All the samples were polished to a 1.00 mm (±0.1 mm) thickness. Total (TT) and diffuse (DT) translucency were measured using a spectrophotometer with an integrating sphere across the visible spectrum (380–700 nm). CIE L*a*b* values were measured using the CIE L*a*b* Pecol software from the spectrophotometer data and colour difference was also measured using this equation $\Delta E^*ab = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2] \times 0.5$.

Results: Statistical analysis by One-way ANOVA followed by Tukey's test showed that there was a statistically significant difference in the translucency between composites with no pigments and the composites with increasing concentrations of the pigments. The translucency decreased with an increase in the concentration of the pigments. However after a certain concentration, the translucency of the pigment ceased to get affected and attained a plateau. The yellow and red pigments as well as the mixtures of the two showed the same pattern.

Conclusions: Addition of pigments affects the translucency of the resins composites to some extent and the translucency decreases initially, but with an increase in the concentration of the pigments, the translucency becomes unaffected. The addition of yellow pigment causes a significant change in b^* value whilst L and a^* only show, small changes. Thus the predominant shift in color is towards the yellow. The addition of red pigment causes both, a change in a^* and b^*, thus producing a shift towards red and also yellow.

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Restorative interface and resin infiltration analysis of nanogel-modified dental adhesives

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Purpose/Aim: The integrity of a dental restoration relies on the extent of resin infiltration into demineralized dentin and sealing of tubules from resin-hydrolyzing pulpal fluid. Combining resins with internally cross-linked, surface-functionalized nanogels offers control of their hydrophobic character, improvements in mechanical properties and reduced shrinkage stress. The study (i) examined the infiltration and sealing capacity of a nanogel-modified adhesive and (ii) monitored the movement of such adhesives through a demineralized dentin matrix in real time using two-photon microscopy.

Materials and methods: A model BisGMA/HEMA 40:60 wt% adhesive was prepared. Nanogels comprised of HEMA and BisGMA were labeled with a Polyfluor 570 fluorescent probe and added to the resin at 40wt%. Human molars were restored using nanogel-modified and nanogel-free resins, and 2-photon microscopy revealed detailed morphology of the interfaces. (ii) Dentin discs were demineralised in EDTA for 2 weeks. 100 μl nanogel-modified resin was deposited onto the disc surface and 2-photon microscopy was used to monitor fluorescent signals at the disc undersurface.

Results: The modification of the resin with nanogels had a dramatic positive impact on its tubule-sealing properties. The modified resins fully permeated demineralized dentin, the visualization and quantification of which was achieved using 2-photon microscopy.

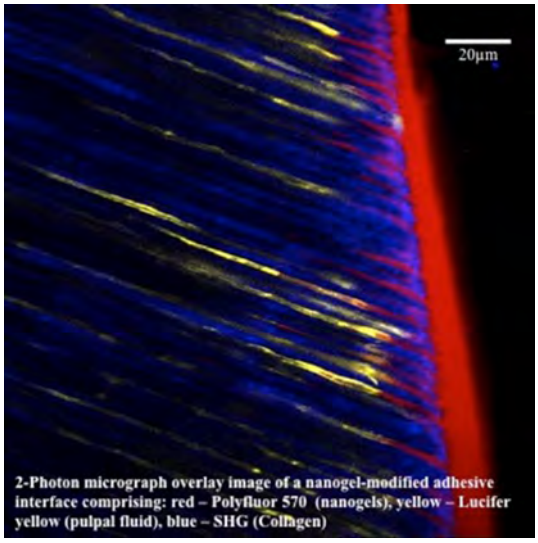

Fig. 1

Conclusions: This study substantiates a previous study that shows the use of the amphiphilic nanogel additives based on HEMA and BisGMA improves dentin bond strength, even after storage (unpublished data). The infiltration of fluorescently-labeled adhesives through dentin is easily monitored using 2-photon microscopy.

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Accuracy of four dental radiometers

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Purpose/Aim: To evaluate the accuracy of four dental radiometers when measuring light output from nine light curing units (LCUs).

Materials and methods: The light output from nine light-emitting diode LCUs (Bluephase G2, Bluephase Style, Valo, Elipar Deep Cure-S, Elipar S10, Paradigm, Smartlite Focus, Optilight Prime and Slim Blast) was measured with a laboratory-grade power meter (PowerMax-Pro 150 HD) and four dental radiometers (Bluephase Meter II, SDI LED Radiometer, Kerr LED Radiometer and LEDEX CM4000). Ten measurements were made from each LCU with each radiometer. Two-way analysis of variance (ANOVA) followed by Tukey tests ($\alpha = 0.05$) were used to determine if there was a significant difference between the irradiance values calculated from the power meter and those from the radiometers. Where applicable, the LCUs were ranked according to their power and irradiance values. The emission spectra from the LCUs were measured using an integrating sphere attached to a fiber-optic spectrometer ($N = 10$). The beam profile of the LCUs was also measured. Pearson correlation tests ($\alpha = 0.05$) were applied to verify if there was correlation between irradiance values obtained using the power meter and the dental radiometers.

Results: ANOVA showed no significant difference between power values measured with the power meter and the Bluephase Meter II ($p = 0.527$). The difference between the mean irradiance reported by the radiometers for the same LCU was up to 479 mW/cm^2 (Table 1). The ranking of the power and irradiance values was the same for the power meter and the Bluephase Meter II, but not with the other radiometers. There was a positive correlation between irradiance values obtained with power meter and values obtained with the Bluephase Meter II ($p < 0.001$) and with the Kerr LED Radiometer ($p = 0.0254$), but not between the values obtained with the power meter and values obtained with the SDI LED Radiometer ($p = 0.0605$) and with the LEDEX CM4000 ($p = 0.0511$). The emission spectra from the Bluephase G2, Bluephase Style, Valo and Smartlite Focus LCUs were wider than the wavelength range accepted by the SDI LED Radiometer, Kerr LED Radiometer and LEDEX CM4000 dental radiometers.

Conclusions: When compared to the power meter, the Bluephase Meter II provided the most accurate data, with no significant difference in the radiant power values. Considering the great variation between the irradiance values provided by some radiometers and their overall inaccuracy when compared to a laboratory-grade power meter, dentists should not

Table 1 – Means and standard deviations of radiant power and irradiance emitted by the LCUs, and the maximum difference in the mean irradiance values.

	Power (mW)		Irradiance (mW/cm^2)					
	PMP Power	Bluephase Meter II – Power	PowerMax Pro	Bluephase Meter II	SDI LED Radiometer	Kerr LED Radiometer	LEDEX CM 4000	Max. Difference
Bluephase G2	812 ± 17.9	783 ± 3.5	1305 ± 28.7	1281 ± 5.7	1236 ± 13.1	1066 ± 13.5	1012 ± 24.2	293
Bluephase Style	640 ± 3.9	602 ± 2.6	1005 ± 6.1	980 ± 14.9	950 ± 19.1	786 ± 13.5	780 ± 7.6	225
Valo	623 ± 9.0	600 ± 3.9	861 ± 12.5	797 ± 4.8	979 ± 22.0	848 ± 16.9	801 ± 15.0	182
Elipar Deep Cure–S	783 ± 11.9	730 ± 2.9	1231 ± 18.7	1196 ± 5.2	1555 ± 8.8	1198 ± 6.3	1184 ± 19.5	371
Elipar S10	679 ± 6.5	613 ± 1.5	1068 ± 10.2	1001 ± 5.7	1474 ± 30.7	1110 ± 21.1	1043 ± 18.6	473
Paradigm	619 ± 4.4	584 ± 1.4	952 ± 6.7	945 ± 8.5	1424 ± 42.7	1030 ± 25.8	1013 ± 8.2	479
SmartLite FOCUS	426 ± 3.3	504 ± 7.4	848 ± 6.5	550 ± 4.7	665 ± 35.0	912 ± 19.3	766 ± 14.5	362
OptiLight Prime	334 ± 5.8	333 ± 2.8	868 ± 15.0	780 ± 14.1	997 ± 25.2	750 ± 0.0	963 ± 6.8	247
Slim Blast	395 ± 10.2	419 ± 9.1	970 ± 25.0	972 ± 17.5	1127 ± 21.5	915 ± 24.1	982 ± 19.7	212

place too much faith in the accuracy of irradiance values from dental radiometers.

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HIF1A overexpression using protein transduction domain induces angiogenesis in HUVEC

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Purpose/Aim: Hypoxia inducible factor-1 alpha (HIF1A) is an important transcription factor for angiogenesis. And protein transduction domain (PTD) has been used to transfer genes in recent studies. But PTD is not used to induce the expression of HIF1A. This study is aimed to use novel PTD (Hph-1-GAL4; ARVRRRGPRR) to overexpress HIF1A gene and identify the effect of angiogenesis in vitro and in vivo.

Materials and methods: HIF1A was transfected using Hph-1-GAL4 in HUVEC. And the expression of HIF1A and HIF1A target genes were analyzed by quantitative real-time PCR (qPCR) 2 and 4 days later respectively. In vitro tube formation assay was identified using Diff-Quik staining. HIF1A and Hph-1-GAL4 were injected subcutaneously into the ventral area of each 5-week-old mouse. All plugs were retrieved after 1 week and the relative expression of HIF1A and HIF1A target genes were evaluated by qPCR. And each matrigel plug was evaluated by hemoglobin assay and HE staining.

Results: HIF1A and HIF1A target gene expressions were significantly higher in HIF1A transfected HUVEC than control HUVEC in vitro. And total tube length was higher in HIF1A transfected HUVEC than control. In vivo matrigel plug assay, the amount of hemoglobin per weight of matrigel was significantly higher in HIF1A treatment group than PBS treatment group. Blood vessels were identified in HIF1A treatment group through HE staining. And HIF1A, VEGF, and CD31 expressions were significantly higher in HIF1A treatment group than PBS treatment group.

Conclusions: It might be helpful to transfer the gene and regenerate tissues effectively when using Hph-1-G4D to overexpress HIF1A.

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Immediate and long-term dentin-composite bond strength: "In-vivo" and "in-vitro" conditions

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Purpose/Aim: To evaluate dentin microtensile bond strength (MTBs) of class I resin composite restorations performed in vivo (time) and under in vitro conditions (simulated pulpal pressure – SPP, aging protocols and storage time).

Materials and methods: Twenty class I restorations were performed in vivo and remained in service for 24 hours or 6 months prior to MTB analysis. In vitro specimens were prepared from 80 extracted human posterior teeth, divided into two groups ($n = 40$) according to whether SPP was used. Groups were subdivided according to aging protocol ($n = 20$) into: Water (storage in distilled water at 37 °C); or Cycling (thermomechanical cycling). Subgroups were further subdivided into two subdivisions ($n = 10$), according to storage time: 24 hours vs 6 months. Restorative procedures were performed with an etch-and-rinse adhesive (Single Bond 2–3 M ESPE) and resin composite (Grandio–VOCO). MTB evaluation was performed at 0.5 mm/min crosshead speed. Fractured beams and tooth slices were analyzed by scanning electron microscopy (SEM). Data were analyzed with ANOVA and post-hoc Tukey test at $\alpha = 0.05$.

Results: Bond strength data (means ± standard deviations; in MPa) are presented in Table 1. MTB decreased after 6 months under clinical conditions. In vitro tests indicated that MTB

Table 1 – Mean and standard deviation of tensile bond strength (in MPa).

Evaluated period	Conditions			
	In vivo	*In vitro*		
		Simulated pulpal pressure	Aging	MTB
24 hours	23.21 ± 3.91	Without SPP	Water	24.96 ± 3.00
			Cycling	23.96 ± 1.77
		With SPP	Water	17.65 ± 1.42
			Cycling	19.49 ± 1.90
6 months	14.17 ± 4.78	Without SPP	Water	16.68 ± 2.53
			Cycling	18.17 ± 2.53
		With SPP	Water	14.33 ± 4.69
			Cycling	17.89 ± 3.98

was significantly affected by all variables examined; MTB decreased with the use of SPP, in presence of water and after 6-month aging. For in vitro and in vivo comparison, the 24 hours results revealed lower MTB for SSP. No differences were detected at 6 months.

Conclusions: The use of SPP adversely affected in vitro bonding at 24 h, while results from all in vitro testing protocols were similar to in vivo conditions at 6 months.

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Influence of 5% sodium trimetaphosphate on MMP-2/9 and dentin bond-strength

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Purpose/Aim: Carious process compromises dentin substrate affecting both inorganic and organic portions, leading to deleterious consequences to adhesive restorative procedures. Sodium trimetaphosphate (STMP) has been introduced as a novel strategy that combines remineralizing potential to the strengthening of the dentin and their stability, increasing mineral nucleation. The aim of this study was to evaluate the anti-protolytic potential of STMP and its effect on dentin bond strength in artificial caries-like dentin.

Materials and methods: The evaluation of anti-proteolitic potential of 5% STMP over MMP-2 and 9 was assessed by zymography. To evaluate the effect of STMP on dentin bond strength (BS), 36 sound human molars were sectioned in occlusal third and 30 molars were challenged in demineralizing solutions (7 d/37 °C). The teeth were divided into 6 groups ($n = 6$): GI- sound (positive control group), GII-deionized water (negative control group), GIII– NaF solution, GIV– 5% STMP solution + saturated solution of $Ca(OH)_2$, GV– 5% STMP + sodium fluoride solution (NaF) and GVI– 5% STMP solution. After the substrates treatment (10 min), according to each group, all specimens were restored using Adper Scotchbond Universal (etch-and-rinse mode) and Filtek Z250. After 24 h, dentin/resin sticks of 0.8 mm^2 were obtained and subjected to the microtensile test (0.5 mm/min) in universal testing machine. DBS data were statistically analyzed by one-way ANOVA and Tukey tests ($p < 0.05$).

Results: The zymography analysis showed that the 5% STMP solution was able to promote 100% inhibition of gelatinolytic activity of MMP-2 and 9. According to the DBS data (GI: 38.49 (6.89)A, GII: 8.67 (1.97)C, GIII: 18.53 (6.78)B, GIV: 15.48 (4.80)C, GV: 8.90 (2.03)C and GVI: 10.09 (3.05)C) it can be observed that the STPM solutions at 5% were not able to enhance the bond strength in dentin submitted to an acidic challenge.

Conclusions: STMP at 5% displays anti-proteolytic activity against MMP-2 and 9. Nevertheless, the solutions containing STMP at 5% are not able to enhance the dentin bond strength in a demineralized dentin.

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Light effects over the color of ormocer X methacrylate composites

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Purpose/Aim: The aim of this study was to analyze the effects of light artificial aging over the color of different ormocer x methacrylate composite resins.

Materials and methods: Sixty samples with a disk shape (1 mm thick and 6 mm of diameter) were prepared with three methacrylate based composites (Filtek Z350 XT – 3M/Espe – nanoparticles, TPH3 – Dentsply – nanohybrid, GrandioSO – Voco – nanohybrid) and one ormocer based composite (Admira Fusion – Voco – nanohybrid) ($n = 15$). The samples were immersed in water for 24 h for post-curing and the surface was polished with 1200, 2400 and 4000-Grit silicon-carbide sandpapers. Fifteen enamel samples with the same dimensions were obtained from bovine incisors, polished in the same way as the composites, and used as a control group. The baseline color was evaluated using a reflectance spectrophotometer (CM-2600d, Konica Minolta), with the samples positioned over a standard white background. The samples were immersed in artificial saliva and subjected to artificial aging using a UV emitter device (SUNTEST CPS+, Atlas), according to ISO 7491. The accelerated aging was performed during 300 hours in order to simulate one-year of clinical use. The temperature was set in 37 °C, with an irradiance of 765 W/m^2. After that the color was measured again, and the total color change (ΔE) was calculated. The data were analyzed using one-way ANOVA and Tukey's post-hoc test.

Results: ANOVA showed significant differences among the groups ($p = 0.001$). The means (±SD) of ΔE and the results of Tukey's test were: Enamel – 3.70 (±1.18)^a, Admira Fusion – 5.26(±0.65)^b, TPH3 – 5.89 (±1.30)^b, GrandioSO – 6.11 (±0.82)^b, Z350 – 14.18 (±1.15)^c. Same superscript letters indicate non significance.

Conclusions: All groups showed color change after light artificial aging. All the composites tested showed higher color change than enamel. The ormocer based composite Admira Fusion showed similar results to TPH3 and GrandioSO. The nanoparticles methacrylate based composite Z350 showed the higher color change of all materials tested.

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Mechanical properties and ion release of composites containing calcium phosphate

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Purpose/Aim: The purpose of this study was to evaluate the effect of replacing glass filler particles with dicalcium phosphate dihydrate nanoparticles (DCPD) either non-functionalized or functionalized with different TEDGMA contents on the biaxial flexure strength (BFS), elastic modulus (E) and ion release of experimental composites.

Materials and methods: Four composites were manipulated, all containing 1:1 BisGMA:TEGDMA (molar ratio), with EDMAB and camphorquinone (0.5 wt% each) as photoinitiators. The total filler content was 60 vol%, including 50% of barium glass (2 μm) and 10% of DCPD nano-structured agglomerates ($CaHPO_4{\cdot}2H_2O$, 15–17 μm). TEGDMA contents (by weight) on the particles were 0% (non-functionalized, NF), 4% (F4) or 30% (F30). As controls, an experimental composite with 60% silanated glass, as well as Charisma and Durafill (Heraeus Kulzer) were tested. BFS and E were evaluated using disc-shaped specimens (12 × 1.2, $n = 10$) kept in distilled water (24 h or 28 d 37 °C) prior to fracture in a "piston-on-three-balls" device (0.5 mm/min). For ion release, specimens (5 × 1 mm, $n = 5$) were individually immersed in 5 ml of NaCl solution (133 mmol/L) buffered to pH 7.0 with 50 mmol/L of HEPES solution, replaced weekly. Ca^{2+} and HPO_4^{2-} release were measured after 7, 14, 21 and 28 d using spectrophotometric methods. BFS and E data were analyzed by Kruskal–Wallis test. Ion release data were analyzed by two-way ANOVA/Tukey test (alpha: 5%).

Results: The results for BFS (MPa) and E (GPa) are shown in the Table 1. After 24 h all materials containing DCPD were statistically similar to the commercial composite (Charisma), but only the material containing F30 was statistically similar to the experimental control without DCPD. Prolonged immersion significantly reduced the BFS only for the materials containing DCPD. Nevertheless, the F30 composite remained similar to Charisma. For E (GPa), after 24 h, all experimental materials were statistically similar ($p > 0.05$). After 28 days, the materials containing DCPD remained similar but presented a significant reduction for E. Functionalization did not affect ion release. For Ca^{2+} release, a significant decrease was observed between 7 and 28 d only for the material containing F4 ($p < 0.05$). All materials released lower concentrations of HPO_4^{2-} after 28 d compared to 7 d ($p < 0.001$).

Conclusions: DCPD functionalization with 30% TEGDMA had a positive effect on the mechanical strength of the composite in comparison to the use of non-functionalized particles, without reducing ion release. However, the presence of DCPD increased the degradation of the composite after 28 d in water, leading to significant reductions in the BFS and modulus (FAPESP 2012/25253-6).

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Influence of try-in moment on bonding between cement and ceramics

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Purpose/Aim: The aim of this study was to evaluate the influence of try-in moment on bond strength of a light-cured resin cement to lithium disilicate (emax-CAD) and reinforced leucite (Empress-CAD) glass ceramics.

Materials and methods: Twelve slices of each ceramic were embedded in epoxy resin and distributed in groups according to different cementation protocols: G1-control (try-in + HF + silane), G2 (HF + silane + try-in) and G3 (HF + try-in + silane). In all groups the try-in gel was followed by air/water spray; the HF, by ultrasonic bath; and the silane, by hot air. After the treatment of each group, a monolayer of adhesive was applied into 4 perforations of an adhesive tape placed on the treated ceramic surface and light-cured. The cement was inserted and light-cured into starch tubes (internal diameter of 1.35 mm) positioned to coincide with tape perforations, and light-cured. Specimens were stored in deionized water at 37 °C for 24 h and tested for microshear bond strength at crosshead speed of 0.5 mm/min. Data was analyzed with one-way ANOVA ($\alpha = 0.05$). Fracture analysis was performed by photographs under high magnification and representative specimens were analyzed by scanning electron microscopy.

Table 1 – Means and standard deviations for biaxial flexural strength (MPa) and flexural modulus (GPa). Uppercase letters in the same column indicate lack of statistically significant difference. Asterisk in the same row indicate statistically significant reduction between 24 h and 28 days of immersion (BG: barium glass, F4: DCPD particles functionalized with 4% of TEGDMA, F30: DCPD particles functionalized with 30% of TEGDMA, NF: non-functionalized DCPD particles).

	Flexural strength (MPa)		Flexural modulus (GPa)	
	24 hours	28 days	24 hours	28 days
BG only	152.2 (12.2) A	132.8 (17.8) A	13.3 (1.0) A	12.7 (1.3) A
F30 + BG	128.2 (11.6) AB*	100.6 (9.6) BC*	13.7 (1.2) A*	11.3 (1.5) AB*
F4 + BG	113.1 (10.3) B*	92.2 (5.8) C*	13.9 (1.1) A*	11.8 (0.9) AB*
NF + BG	113.9 (13.0) B*	88.8 (6.7) CD*	13.2 (1.1) A*	12.0 (2.1) AB*
Charisma	125.0 (19.0) AB	111.4 (24.4) B	11.5 (1.1) B	11.1 (0.8) B
Durafill	77.3 (13.0) C	72.9 (7.8) D	5.3 (0.3) C	5.2 (0.5) C

Results: There were no statistical significant differences between cementation protocols for both ceramics: emax-CAD ($p = 0.245$) and Empress-CAD ($p = 0.102$). More adhesive fractures were observed for emax-CAD, and more cohesive fractures in ceramic for Empress-CAD.

Conclusions: The moment of try-in paste application did not influence in the bond strength between cement and ceramics.

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Roughness and bond strength of glass-ceramic using acid ceramic primers

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Purpose/Aim: The aim of this study was to evaluate the surface topography, roughness and bond strength produced by hydrofluoric acid or acid built-in ceramic primer on CAD-CAM lithium disilicate-based glass-ceramic.

Materials and methods: Thirty disc-shaped specimens, 2.0 ± 0.1 mm diameter and 0.5 ± 0.05 mm in thickness, were milled from lithium disilicate CAD/CAM blocks (IPS e.max CAD, Ivoclar/Vivadent) on an E4D Dentist System (D4D Technologies, LLC, Richardson, TX) using a custom-mill file and fired unglazed according to the manufacturer's instructions. All disks were polished with 1000 grit sandpaper. Fifteen specimens were sandblasted (50 μm aluminum oxide, 2-Bar/5 mm/5 s) and 15 were not. Each group was divided in three sub-groups ($n = 5$) to be treated as follows: 1.no etching (C); 2.Hydrofluoric acid 5%/20 s (HF); 3.Monobond Etch & Prime (Ivoclar/Vivadent) for 60 s (MBEP). All specimens were ultrasonically cleaned in distilled water for 10 min. Surface roughness (Sa-μm) was measured with a confocal 3-D laser-scanning microscope/software (Lext OLS 4000, Olympus) at X216 magnification. Specimen surfaces were analyzed with scanning electron microscopy (SEM). Sixty ceramic sticks ($2 \times 2 \times 4 \pm 0.1$ mm) were cut from IPS e.max CAD ceramic blocks with a low-speed diamond saw, then divided into 2 groups (Sandblasted, Non-Sandblasted). Each group was distributed into three sub-groups ($n = 10$): 1.no etching(C); 2.Hydrofluoric acid 5% for 20 s + silane (Monobond Plus)(HFS); 3.Monobond Etch & Prime (Ivoclar/Vivadent) for 60 s (MBEP). After treatment, composite blocks ($2 \times 2 \times 4 \pm 0.1$ mm) were bonded to the treated ceramic blocks using resin cement (Variolink II, Ivoclar-Vivadent). The ceramic-composite assemblies were stored for 24 h in 100% relative humidity at 37 °C prior microtensile bond strength (μTBS) testing. Statistical analysis was performed using 2-way ANOVA and Tukey test ($\alpha = 0.05$) for roughness and μTBS. Failure pattern was analyzed on SEM.

Results: Two-way ANOVA indicated that the factor "sandblasting" was statistically significant for roughness ($p < 0.001$). Sandblasted group showed statistically higher roughness values than the non-sandblasted group. The sandblasting treatment also altered the ceramic surface, exhibiting shallow irregularities on the surface for C and MBEP groups. For the HF/sandblasted group, those irregularities appeared to be more pronounced and deeper. Two-way ANOVA showed that the factor "etching" was statistically significant for bond strength ($p < 0.001$). HFS and MBEP bond strength did not differ among them ($p > 0.05$). The adhesive failure pattern was predominant for non-sandblasted groups, except for MBEP. For sandblasted groups the failure patterns were mostly mixed, except the control group.

Conclusions: Sandblasting increased the ceramic roughness and morphological irregularities. No difference was detected on bond strength produced by HFS and the usage of MBEP as glass-ceramic surface treatment.

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Effect of silane and resin-cement on flexural strength of glass-ceramic

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Purpose/Aim: The purpose of this study was to evaluate the effect of different silane treatments and the application or not of resin cement on the biaxial flexural strength (BFS) and microstructure of a lithium-disilicate glass-ceramic.

Materials and methods: One hundred disc-shaped specimens (6.5 ± 0.1 mm diameter and 0.5 ± 0.05 mm in thickness) were milled from lithium disilicate CAD/CAM blocks (IPS e.max CAD, Ivoclar/Vivadent) on an E4D Dentist System using a custom-mill file and fired unglazed according to manufacturer's instructions. All disks were polished with 1000 grit sandpaper, sandblasted (50 μm aluminum oxide, 2-Bar/5 mm/5 s), etched with 5% hydrofluoric acid (HF) for 20 s (except for 20 specimens) and divided into five groups of twenty specimens to be treated as follows: (1) (RCP) RelyX Ceramic Primer (3M ESPE); (2) (RCP + SB) RelyX Ceramic Primer + Adper Singlebond 2 (3M ESPE); (3) (SBU) Scotchbond Universa (3M ESPE); (4) (S + HF) just sandblasting and HF; (5) (S) just sandblasting. Half specimens from each group ($n = 10$) were treated also with resin cement(C) and polymerized for 40 s (600 mW/cm^2), the other half were not (0). Specimens' thickness was kept between 0.45–0.55 mm. After 24 hours storage, all specimens were placed on a biaxial-flexure jig and a vertical load was applied (1.27 mm/min) on a universal testing machine until failure. Fractured fragments were gold coated and analyzed on scanning electron microscope (SEM) evaluating failure side and treated surface. Two-way ANOVA and Tukey test ($p < 0.05$) were applied. Additionally, Weibull analysis was performed to analyze reliability.

Results: ANOVA showed the factor "treatment" was statistically significant ($p < 0.0001$). The highest values were obtained by RCP + SB and did not differ from RCP. No statistical difference was recorded between RCP and SBU. SBU was not significantly different from the control groups (S + HF, S). Highest Weibull characteristic strength (σ_0) was obtained by RCP + SB groups and was not different from RCP groups and SBU/0. Lowest Weibull characteristic strength (σ_0) was obtained by S/0 which didn't differ from S/C and S + HF groups. No differences were found on Weibull modulus (m) (Table 1).

Table 1 – Biaxial flexural strength values (BFS) with standard deviation (SD), Weibull characteristic strength (σ_0) and Weibull modulus (m) with confidence intervals (CI) from all experimental groups.

Groups	Treatment	BFS (SD) (MPa)	σ_0 (CI) (MPa)	m (CI)
RCP	None	259.9 (24.6)	270.4 (256.7–285.0) ab	12.5 (7.8–20.2)
	Resin Cement	267.3 (30.7)	280.5 (262.1–300.1) ab	9.7 (6.1–15.4)
RCP + SB	None	269.6 (27.9)	281.3 (264.5–299.1) ab	11.9 (6.9–20.5)
	Resin Cement	275.4 (30.8)	288.8 (269.6–309.5) a	9.5 (6.1–15.0)
SBU	None	234.3 (38.8)	249.8 (229.1–272.3) abc	7.5 (4.6–12.4)
	Resin Cement	241.2 (27.5)	252.4 (239.1–266.4) b	12.0 (7.1–20.2)
S + HF	None	214.2 (42.9)	230.7 (207.4–256.5) bc	6.1 (3.8–10.0)
	Resin Cement	224.6 (38.9)	240.5 (217.7–265.7) bc	6.6 (4.1–10.6)
S	None	205.2 (30.8)	218.1 (200.2–237.7) c	7.6 (4.7–12.3)
	Resin Cement	220.7 (44.8)	238.2 (214.0–265.1) bc	6.1 (3.7–10.0)

Failure side analysis exhibited ceramic-cement separation (SBU/C, HF + S/C, S/C) and bubbles within resin-cement layer (all C groups). Treated surface analysis revealed a mostly uniform surface for SBU/0 and groups treated with resin-cement. Adhesive surface form RCP + SB/0 was more irregular than SBU/0. Abraded surfaces were observed for groups S + HF/0 and S/0.

Conclusions: RCP and RCP + SB improved the ceramic's BFS. The usage of resin cement did not improve the ceramic's BFS. Treatment reliability was similar for all treatments. The usage of a silane layer or accompanied by a separated adhesive layer was fundamental on maintaining the integrity of ceramic-cement interface.

Different capital letters represent statistical differences on BFS among the treatments (column) (Tukey, $p \leq 0.05$). The treatment factor "Resin Cement" was not significant. Different lowercase letters represent differences on Weibull Characteristic strength (σ_0), as the confidence interval from those groups did not overlap at any point (column). For Weibull modulus (m), no differences were found. Symbology: Biaxial flexural strength (BFS), standard deviation (SD), confidence interval (CI), RelyX Ceramic Primer (RCP), RelyX Ceramic Primer and Adper Singlebond 2 (RCP + SB), Scotchbond Universal (SBU), control-sandblasted/5%HF acid etching (S + HF), control-sandblasted (S).

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Longer-term measurement of shrinkage stress

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Purpose/aim: A simple, versatile in vitro methodology that enables indirect quantification of shrinkage and expansion stresses under clinically relevant conditions without the need for a dedicated instrument.

Materials and methods: Description of the experimental and commercial materials based on manufacturer's safety data sheets. The following materials were tested: RelyX Luting Plus Automix, 3M ESPE, Paste/paste resin-modified glass-ionomer (RMGI) cement, GC FujiCEM Automix, GC, Paste/paste RMGI cement, Ketac Cem Aplicap, 3M ESPE, Powder/liquid conventional glass-ionomer cement, Panavia F 2.0, Kuraray, Paste/paste dual-curing resin-based adhesive cement, Adper Easy Bond, 3M ESPE, Self-Etch Adhesive, RelyX Ceramic Primer, 3M ESPE, Ceramic primer, Experimental compositions, 3M ESPE, dual-curing composite filling. Shrinkage and expansion effects including resulting cusp deformation of aluminum blocks with MOD type cavity, filled with novel filling compositions, have been measured using a bench-top micrometer and a Linear Variable Differential Transformer based instrument.

Results: The technique was successfully used in longer-term measurements of shrinkage and expansion stress for several dental compositions.

Conclusions: The presented technique can be reliably used to quantify stress development of curing materials under clinically relevant (oral) conditions. This enables direct examination and comparison of structural properties corresponding to the final stage of formed networks. The methodology is

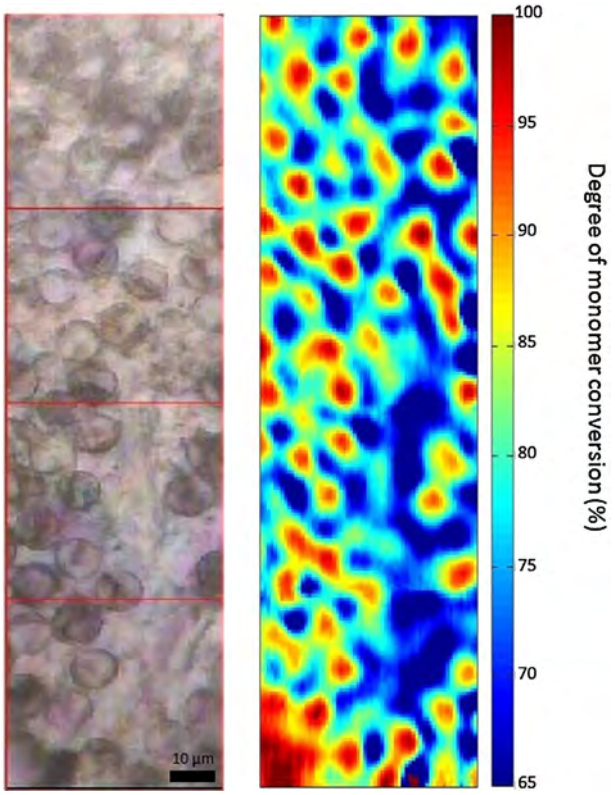

Fig. 1 – Cuspal deformation during curing and after days of immersion in 37 °C water: short-term shrinkage versus longer-term expansion of cements.

directly applicable to the study of self-curing systems as they require mouth-type conditions (temperature and humidity) to achieve their designed kinetics and reactions.

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Effect of loading-direction, crown-coverage, adjacent-teeth on stresses in post-restored premolars

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Purpose/Aim: A relationship between the prognosis of post-endodontic restorations and a variety of factors such as numbers of proximal contacts, occlusal contacts, tooth position in the dental arch, crown placement, post placement, has been reported. These factors may also have an effect on the stresses within the tooth structure thus the biomechanics of the tooth may change. The aims of this Finite Elemental Stress Analysis (FEA) study were to evaluate the effects of loading direction, crown coverage and adjacent teeth on stress distributions in premolar tooth models restored using by fiber-post.

Materials and methods: Three-dimensional (3D) premolar tooth FEA models were created as follows: Model 1: Sound tooth; Model 2: Tooth-model including root filling, fiber-post and composite resin restoration; 3: Tooth-model including root filling, fiber-post, composite resin core and ceramic crown. Materials and structures used were assumed to be homogenous and isotropic except the fiber post. The elastic properties of the structures were provided from the literature and manufacturers. Every model was loaded from the functional cusp either vertically or with a 135° angle using with a 300 N load. The stress distributions or concentrations within the models were evaluated either with or without the presence of adjacent teeth. The Cosmoworks structural-analysis program (SolidWorks Corp, Waltham, MA) was used for analysis and the results were presented considering the von Mises criteria.

Results: High stress concentrations were observed at the mesial side of the sound tooth models while the stresses were localized at both sides in the others. Loading with an angle caused high stress concentrations within the tooth structure when compared to the other models which were loaded vertically. The presence of adjacent teeth affected the stress distributions and concentrations in sound, composite-resin or crown restored tooth models. Less stresses were observed within the models in the presence of adjacent teeth. The presence of crown restoration did not change the stress patterns.

Conclusions: Oblique loading caused greater stress concentrations in the root structures while the presence of a crown did not have an effect on stress distributions when compared to the tooth models with composite resin restoration only. The presence of adjacent teeth has an effect on the stresses thus adjacent teeth should be modeled in further FEA studies.

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Fatigue resistance of 3-unit zirconia and lithium disilicate molar FPDs

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Purpose/Aim: To estimate the fatigue resistance of a new translucent zirconia material in comparison to lithium disilicate for 3-unit FPDs.

Materials and methods: Nine 3-unit FPDs (replacement of first upper molar) with a connector size of 4 × 4 mm were milled dry with a five-axis milling machine (Zenotec Select, Wieland, Germany) using discs of a new translucent zirconia material (Zenostar MT, Wieland). Another 9 FPDs were milled and coloured with B4 liquid (Wieland). The zirconia FPDs were sintered at 1500 °C. As a comparison, 9 FPDs with the same dimensions were made of e.max Press by milling the FPDs from a wax disc (Wieland), investing and pressing them at 920 °C. All 27 FPDs were double-glazed with Ivocolor glaze (Ivoclar Vivadent). The FPDs were adhesively luted to PMMA dies with Multilink Automix. Dynamic fatigue loading was carried out on the molar pontic using Dyna-Mess testing machines (Stolberg, Germany) with 2 million cycles at 2 Hz in water (37 °C). Two specimens per group and load were subjected to decreasing load levels (at least 4) until the two specimens no longer showed any failures. Another third specimen was subjected to this load to confirm the result. All the specimens were evaluated under a stereo microscope (20x magnification). The number of cycles reached before observing a failure, and their dependence on the load and on the material, were modeled, using a lognormal model. This allowed estimating the fatigue resistance as the maximum load for which one would observe less than 1% failure after 2 million cycles.

Results: The failure mode of the zirconia FPDs was mostly a fracture at the distal connector, whereas the failure mode of the lithium disilicate FPDs constituted fractures at the connectors or multiple cracks of the pontic. The fatigue resistance with 1% fracture probability was estimated to be 428 N for the Zenostar MT FPDs without colouring liquid, 393N for the coloured Zenostar MT FPDs and 306N for the e.max Press FPDs. There was no statistically significant difference between both the Zenostar FPD groups ($p = 0.26$), which were, however, statistically significant compared to the e.max Press FPDs ($p < 0.05$).

Conclusions: The fatigue resistance of the translucent zirconia 3-unit FPDs was about 30% higher than that of the lithium-disilicate 3-unit FPDs which may justify their use for molar replacement given that a minimal connector size of 4 × 4 mm is observed. Infiltration of the zirconia with colouring liquids reduced the fatigue resistance by about 8%.

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Evaluation of an anatomic dual-laminate composite resin shade guide

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Purpose/Aim: This study aims to compare different shade selection techniques and determine the suitability of a prefabricated anatomic dual-laminate shade guide and its best mode of use compared to a conventional guide and a layered custom guide.

Materials and methods: CIELab coordinates of different shade guides were assessed: Vitapan Classical (tab A2; Vita); Miris2 prefabricated anatomic dual-laminate shade guide, enamel WR tab on top of dentin S3 tab and nothing in-between (M2air) or glycerin gel (M2gly) or water (M2w); custom shade guide using prefabricated silicon moulds, Miris2 enamel WR composite resin moulded directly on dentin S3 prepolymerised base (M2cus). The average values were obtained to calculate DE and compare the different shade selection techniques. Additional samples and measurements were made to compare Vitapan Classical shade tabs A1, A2 and A3 and all possible combinations of Miris2 and establish the closest matching shade ($\Delta E \leq 3.3$).

Results: High DE values were found (6.51–9.11) when comparing M2air to Vita, M2gly M2w M2cus. Differences appeared acceptable (ΔE 2.09–2.99) between Vita, M2gly and M2w and M2cus. Seven combinations of M2 were found to match Vita tab A1 and A2 and three Miris2 combinations for Vita A3 ($\Delta E \leq 3.3$).

Conclusions: The use of Miris2 prefabricated anatomic dual-laminate shade guide with interposition of water or glycerin between the enamel–dentin tabs demonstrated acceptable DE values when compared to Vitapan Classical and custom guides. A chart for matching Vita shades with various combinations of Miris2 enamel/dentin shades was produced to assist the clinician in obtaining acceptable restorations.

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Observation of enamel micro-cracks under fluorescence-staining using near-infrared light transillumi

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Purpose/Aim: Enamel micro-cracks can be seen at many patients frequently, in clinical situation. Many researchers and clinicians reported that there were serious risks of the dental carious, dental pulp disease and tooth fracture at micro-crack teeth. However, identification and early diagnosis were often difficult because initial enamel micro-cracks were not visibly at chair-side routine inspection. The purpose of this study was to examine the observation method of enamel micro-cracks under fluorescence staining using near-infrared light transillumination.

Materials and methods: 10 extracted human upper incisors with typical enamel micro-cracks were selected and observed by three different methods. Labial surface of all tooth specimens were polished with low speed rotary brush and widths of micro-cracks were measured under light microscope (MM-11, Nikon). And tooth specimens were taken grayscale picture by digital camera (D-5100 with 105 mm Micro-Nikkor, Nikon) under visible light (VL) as control. Then tooth specimens were observed by near-infrared light transillumination device (DIAGNOcam, KaVo) (IR). And then each tooth specimens were stained by 0.25% indocyanine green solution 10 s and also observed by DIAGNOcam (ST). Each 10 micro-cracks of observed digital image were compared with background enamel surface. The difference of gray value was measured using image analysis software (Image J, v1.50, NIH, USA) and then data were analyzed using one-way ANOVA and Tukey's q-test (Microsoft Excel 2010, Microsoft, USA).

Results: The mean value of micro-cracks width was 15.9 (3.1) μm. The differences of gray value (S.D.) under three methods were VL: 42.0 (8.0), IR: 92.5 (11.5) and ST: 120.8 (17.9). The observation methods influenced the difference of gray value significantly at $p < 0.01$. The differences of gray value of ST were statistically larger than IR and VL ($p < 0.01$) and also IR showed large value than that of VL ($p < 0.01$). Green shade could not be seen at stained tooth specimen surfaces with naked eyes.

Conclusions: It concluded that the near-infrared light transillumination device was useful for detect of micro-cracks. Also it could be suggested that the near-infrared light transillumination with additional fluorescence staining was effective observation method of enamel micro-cracks as non-invasive chair-side early diagnosis in this study.

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Effect of light transmission on hybrid ceramic-resin-Ti adhesion system

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Purpose/Aim: To evaluate the light transmission effects on the enclosed mold shear bond strength (EM-SBS) of resin-Ti covered with various hybrid CAD/CAM ceramics in different thickness.

Materials and methods: Three CAD/CAM hybrid ceramics, Lava Ultimate™, Cerasmart™ and Vita™ Enamic were cut into specimens with a size of 0.2 mm × 0.5 mm and thickness 0.2–1.0 mm. Ti sheets were cut, grit blasted and silanized with Monobond® Plus. Polypropylene molds (2 mm in height) were used for resin stub preparation using Multilink® Automix resin composite cement. An external mold was prepared to cover every sample and to make sure light only passes through ceramic. EM-SBS was measured using a universal testing machine.

Results: Resin-Ti bonding cured under Lava Ultimate™, showed higher EM-SBS than Cerasmart™ and Vita™ Enamic. The EM-SBS decreased with increasing thickness of each ceramic from 0.2 to1.0 mm. SEM, AFM and surface roughness profilometer showed surface roughness for Lava Ultimate™, and Cerasmart™ was decreased after polishing and for Vita™ Enamic it was slightly increased.

Conclusions: Resin-Ti bonding cured under Lava Ultimate™, showed higher EM-SBS than Cerasmart™ and Vita™ Enamic. The EM-SBS decreased with increasing thickness of each ceramic from 0.2 to1.0 mm. SEM, AFM and surface roughness profilometer showed surface roughness for Lava Ultimate™, and Cerasmart™ was decreased after polishing and for Vita™ Enamic it was slightly increased.

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Graphene as a substrate to enhance neurogenic differentiation of DPSC

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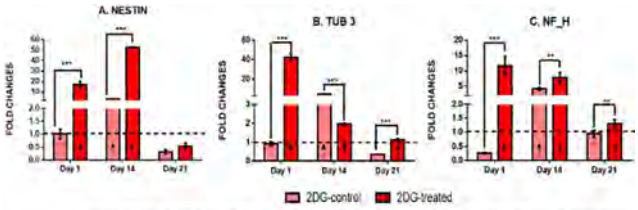
Purpose/Aim: To assess the potential of graphene as a substrate to promote neurogenic differentiation of dental pulp stem cells (DPSCs).

Materials and methods: Graphene was produced by chemical vapour deposition and transferred to glass (2DG). DPSC was seeded on 2DG and left undisturbed for 24 hours. Cells were treated with either basal culture media or neural induction media (neurobasal-A medium, 1× B-27 supplements, 20 ng/ml EGF and 40 ng/ml bFGF) for 21 days. Cell proliferation was assessed for 5 days with MTS assay. Cell morphology with Live&Dead assay for 1, 14 and 21 days. The expressions of neurogenic differentiation markers (nestin, Tub-3 and NF-H) were evaluated by qPCR (1, 14 and 21 days) and FACS (21 days). Glass with neurogenic medium (Gl) was used as control. All tests were performed in triplicates. Statistical analysis was performed with either one-way ANOVA and post hoc Tukey's or two-way ANOVA with post hoc Bonferroni test ($\alpha = 0.05$).

Results: There was no difference in cells proliferation for both Gl and 2DG after 5 days. Cells on 2DG with neurogenic medium presented neuron-like morphology similar to the control. In general, 2DG increased the expression of the genes studied in the presence of neurogenic genes compared to the control (Fig. 1). After 21 days, the protein expression observed for control, 2DG and 2DG treated with NM were 79.8, 11.2 and 96.7 for nestin; 97.5, 3.4 and 97.2 for Tub-3; 26.3, 0.6 and 45.4 for NF-H, respectively.



Gene expression for neuronal markes of DPSCs on 2DG without (2DG-control) and with neurogenic medium (2DG-treated). [*** = p≤0.001, ** = p≤0.01, * = p≤0.05. Dashed line = DPSC on glass treated with neurogenic medium (control). ↑ = significantly higher than the control (p<0.05)]

Fig. 1

Conclusions: 2DG failed to induce spontaneous neurogenic differentiation of DPSC. After 21 days, cells on 2DG treated with neurogenic medium presented higher expression of NF-H compared to the control confirming the capability of 2DG to enhance neurogenic differentiation of DPSC.

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Silica-coating protocols on Y-Tzp: Roughness and fractal geometry

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Purpose/Aim: To determine the surface roughness (Ra) and calculate the fractal dimensional increment (D*) of Y-TZP structures after different silica-coating (SC) protocols.

Materials and methods: Y-TZP bar-shaped specimens were divided according to the SC protocol. The control group (a) did not receive any surface treatment. Groups (b) to (e) received SC with silica-modified alumina particles with different sizes either before or after final sintering, as follows (particle size/SC protocol): (b) 30 μm/before sintering, (c) 110 μm/before sintering, (d) 30 μm/after sintering, and (e) 110 μm/after sintering. Atomic force microscopy (AFM) was used to examine the surface roughness (Ra) of each group. The height data from the treated surfaces were imported into a custom MathCAD script,

and FRACTALS software was used to determine D* by the Minkowski cover technique.

Results: Results are shown in Table 1. Silica-coating before sintering always resulted in rougher surface compared to silica coating after sintering, and this difference was statistically significant for both 30 μm and 100 μm particles. Silica coating after sintering always resulted in greater D* compared to silica coating before sintering, but this difference was significant only when 30 μm particles were used.

Table 1

SC protocol	Ra (μm) (SD)	D*(SD)
Control (after final sintering)	0.28 (0.12)[b]	0.31 (0.04)[a,b]
30 μm/after sintering	0.27 (0.05)[b]	0.35 (0.02)[a]
110 μm/after sintering	0.34 (0.01)[b]	0.28 (0.01)[a,b,c]
30 μm/before sintering	0.62 (0.07)[a]	0.22 (0.02)[c]
110 μm/before sintering	0.57 (0.07)[a]	0.23 (0.04)[b,c]

Conclusions: Fractal analysis was capable of revealing differences in tortuosity for the different SC protocols used. The highest roughness values were obtained when SC was performed before final sintering. As to tortuosity, only surfaces that were silica-coated with 30 μm particles after final sintering had significantly higher contact area than those that were silica-coated before sintering.

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Surface finishing effect on biaxial strength of CAD/CAM materials

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Purpose/Aim: Intraoral adjustment of indirect restorations is a critical step, as introduction of defects in the material's surface might lead to premature failure. Different diamond polishing systems have been introduced to remove the induced damage, although their impact on the final strength of the restoration has not been yet thoroughly described. Therefore, this study assessed the effect of different surface finishing procedures on the biaxial strength of three different dental CAD/CAM blocks and a direct resin composite.

Materials and methods: Thin plates ($12 \times 12 \times 1.2\,mm^3$) were cut off from Celtra Duo (Dentsply), Enamic (Vita Zahnfabrik) and e.max CAD (Ivoclar-Vivadent) CAD/CAM blocks or prepared by condensing and polymerizing Clearfil Majesty Posterior (Kuraray) resin composite in tungsten carbide molds. One of the specimen's surfaces was then grinded using SiC paper with grain sizes Grit 120, 320 or 1200 in order to simulate surface finishing with a red, yellow or white code diamond bur ($n = 30$). The treated surface of half of the specimens ($n = 15$) was then polished using 2-step diamond-impregnate silicon polisher systems. A control group ($n = 15$), sequentially polished with SiC paper (Grit 600 down to 4000), was prepared for each material. The roughness depth (Rz) of all surfaces was measured using optical profilometry. Biaxial strength of the specimens was determined using the ball-on-three-balls fixture with the roughened/polished surface loaded in tension.

Results: An inverse relationship between surface roughness and biaxial strength was found for the ceramic materials Celtra Duo and e.max CAD. A sustained increase of strength was observed with decreasing roughness. The differences among groups were, however, only significant up to a certain roughness depth ($R_z < 1.5\,\mu m$). Polishing with the diamond-impregnate silicon polishers increased the biaxial strength up to the control group values, except for the e.max CAD group with Grit 120 surface, whose strength values did not improve after polishing. The biaxial strength of resin composite materials (Clearfil Majesty Posterior and Enamic) was not affected by the surface roughness, being statistically similar after each surface finishing procedure.

Conclusions: Surface finishing of dental ceramics after intraoral adjustment has a critical effect on the material's strength. Utilization of fine diamond burs (white code) as well as polishing systems are successful strategies to overcome this problem. Still, caution must be observed to completely remove the induced damage. This issue seems not to affect the resin composites studied here.

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Bulk-fill resin composites: Mechanical properties *vs* depth of cure

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Purpose/Aim: To evaluate the Knoop hardness (KH) and the biaxial flexural strength (BFS) of bulk-fill resin composites (high-viscosity and flowable) and compare them to conventional resin composites in different depth of cure.

Materials and methods: Five bulk-fill (x-tra fill, Filtek bulk-fill Posterior, Tetric N-Ceram bulk-Fill, Surefil SDR flow e Filtek bulk-fill flow) and two conventional composites (Premise e Filtek Z 350 XT) were used. Resin composite increment was packed into silicone molds (4mmx4 mm) then light-cured for 20 s ($20\,J/cm^2$). Five specimens from each material were built-up. The Knoop hardness (0.49 N/5 s) was then measured at the top surface and at every 0.5 mm down from the surface. Subsequently KH values, bottom-top-ratios percentage (KH Bottom-Top-Ratio) were calculated. For BFS test, composite discs (0.5 mm thick) were made and stacked between Mylar sheets until completing 4.0 mm in thickness (one disk per each 0.5 mm). The top of each stack was exposed to the light for

20 s (20 J/cm^2). Seventy discs from each material were built for each 0.5 mm thick and then were placed into a biaxial-flexure jig and a vertical load was applied (1.27 mm/min) on a universal testing machine (model 1144, Instron Corp., Norwood, MA) until failure. The KH and BSF values were analyzed using two-way ANOVA and Tukey's post-hoc test at 0.05.

Results: The KH values were statistically lower in 4 mm when compared to top (0.5 mm) for Surefil SDR flow e Tetric N-Ceram bulk-Fill. All resin composites presented adequate curing in 4 mm (at least 80% hardness recorded at the top of the surface), except to Premise. No statistical differences were found for BFS values between specimens at 4 mm compared to top (0.5 mm) in all materials. Tetric N-Ceram bulk-Fill presented statistically lower BSF values compared to all other composites at every measured depth, except to Premise in 3.5 mm depth.

Conclusions: All investigated resin composites obtained proper BSF at 4 mm depth, while KH values were material dependent.

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Evaluation of optical and physical properties for initial LiSi press

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Purpose/Aim: To avoid the risk of metal allergy and gum discoloration, there has been a movement away from metal-based restorations toward all-ceramics. Dental restorations require physical strength above all. From this aspect, zirconia has been used well lately. However, when aesthetic property was also required, zirconia restorations aren't as aesthetic as lithium-disilicate glass-ceramics which has much more translucency and fluorescence. New pressable lithium-disilicate glass-ceramics, initial LiSi Press, is developed with focusing on optical and physical properties. The objective of this study is evaluation of these properties.

Materials and methods: *Color and translucency measurement*: Initial LiSi Press with different level of translucency (HT-E58, MT-A2, MO-1) were pressed according to the manufacturer's instructions and polished with waterproof abrasive papers (#1000) to obtain discoid samples with a thickness of about 1.2 mm. A colorimeter (CM-3610d, MINOLTA) and a turbidity meter (NDH-5000, Nippon Denshoku) were used to measure color of samples and compare translucency of samples, respectively. *Biaxial bending test*: According to ISO 6872:2015 "Dentistry-Ceramic materials", mirror polished samples were prepared (a thickness of 1.2 mm, a diameter of 15 mm), fracture load was measured by using universal testing machine AG-50kNG (Shimadzu) (a crosshead speed of 1.0 mm/min). Biaxial bending strength was calculated ($n = 12$) and one-way ANOVA was used to assess the significance difference.

Results: Fig. 1 shows that total-light transmittance (one dot) of each grade of translucency and biaxial bending strength (bar graph). According as the designed level of translucency was higher, the value of total-light transmittance got higher correlatively. On the other hand, there is no significant difference in biaxial bending strength for various shades ($p > 0.05$).

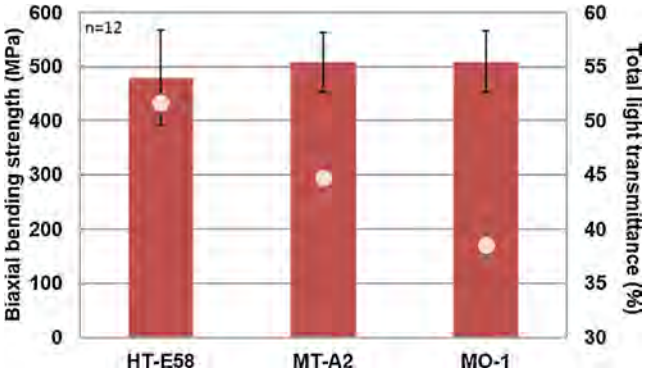

Fig. 1

Conclusions: While initial LiSi Press system has gradual translucency, different levels of translucency didn't affect physical strength. That is, these results suggest that initial LiSi Press system is useful to satisfy the aesthetic demands of each clinical case without any anxiety about insufficient strength.

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Decreasing glass refractive index for development of light-curable bioactive composites

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Purpose/Aim: Bioactive glasses (BGs) can promote bone and tooth remineralisation and may offer potential biological improvements for current resin based composites (RBCs). However, the refractive index (RI) mismatch between the organic polymer resin-matrix of conventional RBCs and conventional BG materials may lead to increased light scattering, reducing light transmission (LT) and degree of conversion (DC) through depth. The aim of this research was to develop and characterize novel BGs which exhibit a similar RI to the resin-matrix in order to improve LT and DC.

Materials and methods: Experimental resins (60:40 and 70:30 wt% BisGMA/TEGDMA) containing camphoroquinone/amine photoinitiator (0.2/0.8 wt%) were formulated. Glass fillers were manufactured based on the commercial 45S5 BG (control; 45 wt% SiO_2 and 5:1 ratio of calcium oxide (CaO) and phosphorus pentoxide (P_2O_5)) using the melt-quenched technique (10 °C/min up to 1450 °C). Modified 45S5 glasses were made by substituting the CaO component with up to 15.0 mol% calcium fluoride (CaF_2). Resins were either tested unfilled or loaded with glass fillers (30 wt%). RI was determined using an Abbé refractometer (475 nm, 23 °C; Carl Zeiss 32487, Germany). Specimens were cured (20 s, 1200 mW/cm^2; S10 Elipar, 3M ESPE, USA) and real time LT and DC were assessed using a MARC resin calibrator (2 mm

thick, 4 mm diameter; BlueLight Analytics Inc., Canada) and Fourier transform near infrared spectroscopy (FT-NIRS) (1 mm thick, 12 mm diameter, $-CH_2$ bond at 6164 cm^{-1}; Nicolet 6700, Thermoscientific, USA). Multivariate analysis of variance (ANOVA) and post-hoc Tukey comparisons ($P < 0.05$) were used to assess the differences.

Results: Multivariate analysis of variance revealed that CaF_2 loading significantly reduced the RI of glass fillers ($P < 0.05$) compared with 45S5 (45S5: 1.568 > 15.0% CaF_2: 1.524). Substitution of CaO with CaF_2 achieved a better RI match between resin and filler for 70/30 (pre-cure and post-cure RI = 1.523 and 1.551) and 60/40 (pre-cure and post-cure RI = 1.513 and 1.545, respectively). 70/30 and 60/40 Bis-GMA/TEGDMA resins resulted in a 5 and 6-fold increase in LT, respectively ($P < 0.05$) compared with controls. Substitution of CaO for CaF_2 also resulted in a significant increase in DC for both resin blends compared with controls ($P < 0.05$) and was dependent on the amount substituted and the resin blend.

Conclusions: The RI of BGs could be altered by partial substitution of CaO with CaF_2. Since increasing CaF_2 loading decreased RI, the RI of BG could be appropriately matched to that of the resin matrix which could improve LT leading to improved DC.

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Catechol functionalized nanopolymersome films on titanium implants to promote osseointegration

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Purpose/Aim: Osseointegration is one of important markers for the success of dental implants, and it can be enhanced by coating polyelectrolyte multilayer films (PEM) on implant surfaces. An ideal PEM should have suitable film-substrate bonding strength to guarantee films against peeling or damage during implanting, suitable stiffness to modulate the biological behaviors of cells on implant surfaces, and biological activity. Herein, a new PEM with suitable mechanical and biological properties was constructed on titanium (Ti) implants to promote osseointegration.

Materials and methods: Lipopolysaccharide-amine nanopolymersomes (NPs) and gene-loaded NPs (pNPs) were prepared following our reported method. Catechol functionalized NPs (cNPs) and Hyaluronic acid (cHA) with different catechol substitution degree (CSD) were synthesized following the reported method. Via layer-by-layer self-assembly technique, the PEM was constructed with cNPs as an initiating layer, cHA as an anionic polyelectrolyte and pNPs as a cationic polyelectrolyte. The built PEM was defined as Ti-cNPs-(cHA/pNPs)n, where n is the number of assembly cycle. It should be noted that NPs and pNPs were chosen because NPs are efficient cytosolic delivery vectors, and pNPs have high transfection efficiency (>95%) in mesenchymal stem cells (MSCs) and induce significant angiogenesis in zebrafish. The assembly process of PEM was analyzed by quartz crystal microbalance with dissipation, XPS and IR. The film-substrate bonding strength and stiffness of films were characterized by a nanoscratch and nanoindentation test, respectively. Their biological effects were evaluated by growing MSCs on films.

Results: Ti-cNPs-(cHA/pNPs)n can be constructed successfully, and it grows exponentially. The film-substrate bonding strength and stiffness of Ti-cNPs-(cHA/pNPs)n can be adjusted by changing CSD in cNPs and cHA. When CSD in cNPs and cHA is 40% and 10% respectively, the bonding strength of Ti-cNPs-$(cHA/pNPs)_3$ is 44.99 ± 12.49 mN, which may be enough to resist the mechanical destroy during implanting because the maximum force applied on dental implants is usually less than 40 mN; and its stiffness is 12.17 ± 3.79 GPa, which is exactly in the range of the bone stiffness (1–27 GPa); in our studied range, this film behaves optimum in facilitating the adhesion, proliferation and differentiation of MSCs.

Conclusions: These preliminary data indicate that the constructed PEM may have suitable mechanical and biological properties to promote osseointegration, and have a potential in the clinic. Next, we will study the resistance of films against the mechanical destroy during implanting by simulating the clinical implantation process, their in situ gene transfection, and in vivo osseointegration.

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Experimental composites of polyacrilonitrile-electrospun nanofibers containing nanocrystal cellulose

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Purpose/Aim: The use of nanofibers containing nanoparticles is a possible and novel approach to reinforce dental resins. In this study we tested the effects of the addition of PAN nanofibers and NCC-containing PAN nanofibers on flexural properties of experimental dental composites.

Materials and methods: 11 wt% PAN (MW 150,000) in dimethylformamide (DMF) solution was electrospun (Kato Tech, Japan) at 17.2Kva and 20 cm from the collector drum. Non-functionalized NCC was added to the solution at 3 wt%. Fiber mats were produced in triplicates, collected on an aluminum foil and tested as-spun. Strips (5 cm × 0.5 cm) were cut from the mat in an orientation parallel (par) and perpendicular (per) to the rotational direction of the collector drum. Tensile tests were performed (KES-G1, Kato Tech, Japan) and ultimate tensile strength (UTS), Elastic Modulus (E) and Elongation at maximum stress (%) were calculated

Table 1 – Effects of the presence of NCC in PAN nanofiber reinforced dental composites.

Groups	Flexural strength, MPa (SD)	Flexural modulus, GPa (SD)	Work of fracture, KJ/m^2 (SD)
Resin blend (No Fiber) ($n=18$)	94.91 (11.43)^{B}	2.85 (0.21)^{A}	5.46 (1.88)^{C}
0% NCC – PAN ($n=47$)	99.03 (8.80)^{AB}	2.62 (0.21)^{B}	7.11 (2.09)^{B}
3% NCC – PAN ($n=42$)	101.39 (5.92)^{A}	2.60 (0.17)^{B}	8.05 (1.62)^{A}

from stress/strain plots. Fiber mats were then infiltrated by resin monomers (50/50 BisGMA/TEGDMA wt%), stacked in a mold (2 × 15 × 25) and light cured. Beams (2 × 2 × 25 mm) were cut from the slabs and tested according to ISO 10477 in a universal testing machine (Shimadzu Corp., Kyoto, Japan). Data were analyzed by multiple t-test and one-way ANOVA ($\alpha=0.05$).

Results: Addition of 3% NCC resulted in higher tensile properties of the fibers. Fibers presented anisotropic behavior with higher UTS and E when tested in perpendicular orientation. Composites were fabricated only with fibers oriented in the perpendicular direction (Table 1).

Conclusions: The presence of NCC in PAN nanofibers resulted in significant increase in work of fracture and flexural strength of experimental dental composite beams. This fundamental study warrants future investigation in the use of electrospun nanofibers with nanoparticles. NCC was found to be a suitable nanoparticle to reinforce experimental dental composites by incorporation via nanofiber.

Different letters (one-way ANOVA) indicate significant difference between the values for each property ($p<0.05$).

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Long-term antimicrobial poly(methyl methacrylate) incorporated with silver releasable nanocarriers

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Purpose/Aim: Since the introduction of poly(methyl methacrylate) (PMMA) as a removable restorative material, it has suffered from having relatively poor antimicrobial properties, which accelerated oral infection and induced unfavorable odor. Mesoporous silica nanoparticles (MSN) have been highlighted as a potential additive due to loading capacity with their large surface area and great total pore volume. Here, we present silver (Ag) releasable drug loaded MSN (Ag-MSN) incorporated PMMA for inducing antimicrobial effects.

Materials and methods: After Ag-sulfidiazine loaded MSN (Ag-MSN) was characterized in terms of morphology, particle and pore size, pore volume, and total surface area, mechanical and antimicrobial properties of Ag-MSN incorporated PMMA were evaluated.

Results: Typical spherical morphology with well-ordered mesoporous structure was visualized by transmission electron microscopy before and after loading Ag-sulfadiazine in MSN. Ag-MSN had 392.6 m^2/g of surface area with 3.54 nm of pore size and 0.42 cm^3/g of pore volume, which are beneficial for releasing ions and being combined in matrix. Existence of Ag releasable drug on the surface of Ag-MSN incorporated PMMA was confirmed by scanning electron microscopy with energy dispersive spectroscopy. Ag-MSN incorporation in PMMA (0.5%, 1%, 2.5% and 5%) did not decrease flexural strength ($p>0.05$) in rectangular specimen (1.4 × 3.0 × 19.0 mm) by 3 point flexural test at a speed of 1 mm/min while flexural modulus was significantly increased in 2.5% and 5% incorporation ($p<0.05$). Anti-adherent effect after 1 hr attachment with *Candida albicans* and *Streptococcus oralis* was observed in Ag-MSN incorporation PMMA disk ($\emptyset=11.5$ mm and $d=1.5$ mm) compared to MSN counterpart ($P<0.05$) without cytotoxicity to oral keratinocyte ($P>0.05$) and this effect was increasing depending on the increase of Ag-MSN incorporation. Continuous anti-microbial effects up to 21 days incubation or reloading Ag drugs on Ag-MSN incorporated PMMA were observed. Antimicrobial effects were majorly caused by released Ag ions and partially change in surface hydrophilicity.

Conclusions: Within the limitation of this study, these aspects of mechanical, continuous anti-adherent performances even after reloading drugs suggest potential usefulness of the Ag-MSN incorporation, especially Ag-MSN 5%, in PMMA resin for inducing antimicrobial effect.

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Incremental and bulk filling techniques in different cavity configurations

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Purpose/Aim: The aim of this study was to compare the micro-tensile bond strength by incremental and bulk fill techniques under conditions of different C-factor and compliance.

Materials and methods: Extracted human third molar teeth were randomly divided in 3 experimental groups. For group I, Class I cavities were prepared. For group II, MOD cavities of the same size were prepared. For group III, the same cavities as group II were made except high compliance cavity wall. Each group was divided into four subgroups. They were restored with two materials: TB, Tetric N-ceram Bulk-Fill (Ivoclar vivadent); VB, Venus Bulk-Fill (Heraeus Kulzer)

and by two methods: incremental or bulk filling technique. The specimens were measured for cavity wall compliance and micro-tensile bond strength (μ-TBS) to the cavity floor. The polymerization stresses of the composites (TB and VB) were gauged with a custom-made device. The results were statistically analyzed using a Kruskal–Wallis test followed by Mann–Whitney U test. The μ-TBS data were also analyzed by Weibull analysis.

Results: In group I, the μ-TBS by incremental technique was significantly higher than that by bulk fill technique ($p < 0.05$). In group II and III, there was no difference by the two techniques. The μ-TBS measured in group I turned out to be lower than that in group II or III ($p < 0.05$). The polymerization stress of TB and VB was different, however, no statistical difference was found in μ-TBS when it was filled with either TB or VB ($p > 0.05$).

Conclusions: In high the C-factor cavity, there was a difference between the incremental and bulk-filling technique in bonding strength on the cavity floor. In the low C-factor cavity, there was no difference between them. The bond strength in the high C-factor cavity was significantly lower than that in the low C-factor cavity.

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MRI artifacts and radiopacity of CAD/CAM composite resin blocks

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Purpose/Aim: Recently several CAD/CAM composite resin block have been introduced. However, MRI artifacts and radiopacity of CAD/CAM composite resin blocks have not been clearly elucidated. The aim of the present study was to evaluate MRI artifacts and radiopacity of CAD/CAM composite resin blocks comparing composite resin for fabrication of crown and bridge (CRCB).

Materials and methods: Four CAD/CAM composite resin blocks (Cerasmart, GC; Estelite Block, Tokuyama Dental; Katana Avencia, Kuraray Noritake Dental; KZR HR2, Yamamoto Precious Metal) and 4 composite resin systems for fabrication of crown and bridge. (Estenia, Kuraray Noritake Dental; Gradia Fort, GC; Pearl Este, Tokuyama Dental; Symphony, 3 M ESPE) were examined. Six crown-shaped specimen (8.0 mm in outer diameter, 8.0 mm in height, 5.0 mm in inner diameter, 6.0 mm in inner depth) of each material were prepared according manufacturer's instructions. Specimens were placed in water tank and transverse images with a 2.5-mm slice thickness were acquired using 3T MRI scanner. The artifacts of images by turbo-spin echo and gradient echo sequences were determined using image process and analyzing software (Image J). Five disc-shaped specimens, 5.0 mm in diameter and 1.0 mm in thickness of each component of each system were prepared. An x-ray image of each specimen with an aluminum step wedge from 0.5 to 6.0 mm in steps of 0.5 mm was obtained by intraoral X-ray apparatus and imaging plate. An aluminum equivalent thickness was determined using an aluminum thickness-radiopacity plot. The sizes of artifact and aluminum equivalent thickness were analyzed with one-way ANOVA and Tukey's multiple comparisons.

Results: The size of MRI artifacts at the crown top portion of CAD/CAM blocks and CRCB were 10.1–10.7 mm and 9.9–13.1 mm, respectively; those at the axial portion of CAD/CAM blocks and CRCB were 9.7–9.8 mm and 9.5–9.8 mm, respectively. One-way ANOVA revealed that the sizes of MRI artifacts at the crown top were not significantly different but those at the axial portion were not. The MRI artifacts of CAD/CAM blocks did not cause severe problem, but those of CRCB did show a slight disturbance. The aluminum equivalent thicknesses of CAD/CAM blocks and CRCB components were 0.01–2.11 mm and −0.05 to 2.95 mm, respectively. The radiopacities of CAD/CAM composite resin blocks were significantly different among products. The products with greater radiopacity did not show a greater MRI artifact.

Conclusions: Comparing CBCR, CAD/CAM composite resin blocks did not show severe MRI artifact, but their radiopacities varied among products.

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Effect of CAD/CAM fabricated framework on complete denture deformation

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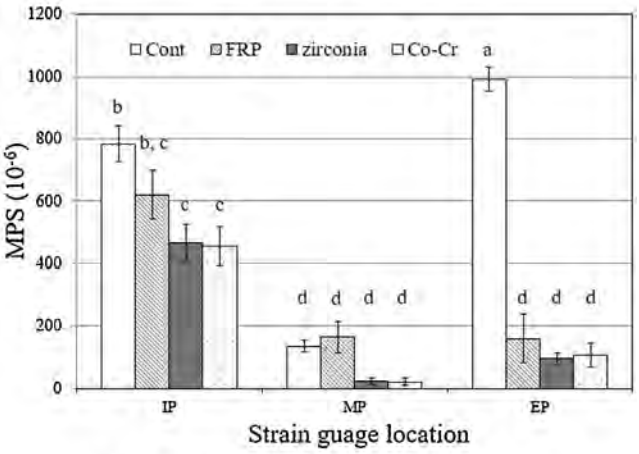
Purpose/Aim: Recently several CAD/CAM systems have been available to fabricate a denture framework using various materials, however, their efficiencies of these framework have not been clearly elucidated. In this study, effects of CAD/CAM fabricated framework on the complete denture deformation during loading were evaluated.

Materials and methods: Four types of the maxillary denture were prepared with and without a denture framework (0.5-mm thickness). Group 1: conventional denture with a palatal plate thickness of 1 mm (without denture framework) (Cont), Group 2: denture with a milled fiberglass reinforced plastic (FRP) (TRINIA, Bicon) framework, Group 3: denture with a milled zirconia (C-Pro Nano-Zr, Panasonic Healthcare) framework, Group 4: denture with laser sintered Co-Cr (SP2, EOS) framework. These frameworks were fabricated with corresponding CAD/CAM system using the same STL files. The framework was located not to cover incisive papilla and upper lip frenulum. The complete denture was processed using an injection molding technique (Fit denture system, Shofu). Three strain gauges were cemented onto the mid-line of oral surface in each denture; incisive papilla (IP), end point of the denture (EP) and middle point (MP) between IP and EP. An occlusal load was applied by a universal-testing machine at a loading rate of 20 N/s to 200 N. The load and strain were recorded through sensor interface to a personal computer, and the maximum principle stress (MPS) was calculated. The MPSs at 200-N load



Result of MPS at 200-N loading. Bars with the same letter were not significantly different (p>0.05).

Fig. 1

were statistically analyzed with 2-way ANOVA and Tukey's multiple comparison ($\alpha = 0.05$).

Results: Generally, the MPS of all groups increased with an increase of applied load. The MPSs at 200-N load were summarized in Fig. 1. The locations of MPS ranking were EP > IP > MP for Cont, IP > MP = EP for FRP, and IP > EP = MP for Co-Cr and zirconia. Regarding EP and IP, Cont were significantly greater than the others. Although the thickness of palatal part of frameworks was half of that of Cont, the MPS with the framework was smaller than that without framework. The MPS of zirconia and Co-Cr showed a similar tendency probably because of their similar elastic modulus. Due to interwoven fiberglass layered structure, this FRP material showed anisotropic mechanical properties. Therefore stress distribution pattern of the FRP was different form the others.

Conclusions: Within the limitation of this study, the framework fabricated with CAD/CAM systems significantly reduced the deformation of the maxillary complete denture.

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Liposome adsorption to restorative materials

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Purpose/Aim: To investigate the in vitro adsorption and retention of liposomes onto four common types of dental restorative materials.

Materials and methods: Uncoated liposomes (positively and negatively charged) and pectin coated liposomes (low- and high-methoxylated) were prepared and characterized in terms of particle size and zeta potential. Four common restorative materials were included: conventional (Tetric EvoCeram) and silorane-based (Filtek Silorane) resin composites, as well as conventional (Fuji II) and resin-modified (Fuji II LC Improved) glassionomer cements (GIC). The adsorption of liposomes was performed by immersion, quantified by fluorescence detection, and visualized by fluorescence imaging and atomic force microscopy.

Results: Positive liposomes demonstrated the highest adsorption on all four types of materials likely due to their attractive surface charge. They also retained well (minimum 40% after 60 min) on both conventional resin composite and GIC even when exposed to simulated salivary flow. Although an intermediate initial level of adsorption was found for the pectin coated liposomes, at least 70% high methoxylated-pectin coated liposomes still remained on the conventional resin composite after 60 min flow exposure. This indicates significant contribution of hydrophobic interactions in the prolonged binding of liposomes to resin composites.

Conclusions: Based on these results, two new possible applications of liposomes in the preservation of dental restorations are suggested: (1) liposomal adsorption may inhibit, reduce, or delay the harmful effects of bacterial adherence and the development of biofilm on the restorative materials and (2) liposomal retention may seal marginal gaps at the tooth tissue-dental material interface preventing microleakage.

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Effects of acidity of "Bissap" (*Hibiscus sabdariffa*) on dental ceramics

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Purpose/Aim: Various studies showed the effect of pH variations from oral environment on dental ceramics. This environment can be modified by foods and drinks consumption. Studies showed evidence of ions releases in acidic medium especially in presence of acetic acid whose pH is near commonly consumed drinks. "Bissap," an acidic drink widely consumed in Côte d'Ivoire is obtained from Hibiscus sabdariffa flowers' infusion. The purpose of this study is to analyze ions releases after ceramics immersion in "Bissap" and determine the impact of glazing.

Materials and methods: 96 ceramic disks were manufactured from 2 types of ceramics. Each type is subdivided into 2 groups: with glazing and glazing-free. After pH measurement from "Bissap", the ceramic samples are immerged in it. Sodium, potassium and aluminum ions are dosed by spectrometry at T_0, after 24 h, 48 h, 72 h, then after 7 days, 14 days, 21 days and 28 days. Then, we have renewed each bath by adding, every day, 60 ml of bissap in the initial bath. The "Bissap" solution pH is also measured.

Results: The results showed an increase of pH which tends to stabilize upon the 2nd day. One-way repeated ANOVA, showed the following results: The composition of the juice of the different groups of ceramics immersion showed no significant difference ($p > 0.05$) compared to initial bissap juice, after

different times of our study. However, when bath was renewed daily, by adding 60 ml of bissap, we got a significant increase ($p < 0.001$), rates of aluminum ions and potassium ions from the 14th day, while sodium increased from 21th day ($p < 0.05$). The comparison of groups with glazing and without glazing, showed no significant difference ($p > 0.05$).

Conclusions: Acids contained in "Bissap" lead to an elution of alkaline ions in acidic medium. Renewing the bath solution seems to accelerate the process while glazing delays it.

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Calcium & fluoride recharge of resin cements

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Purpose/Aim: The objective of this study was to determine the recharge ability of calcium and fluoride ions of dual-cured resin cements.

Materials and methods: Calcium and fluoride release and recharge were measured in Bisco's experimental cement ExpCem, Bisco's self-adhesive cement BisCem, and NuSmile's cement BioCem. Disks were prepared (2 cm diameter and 0.1 cm high) with each cement, and self-cured for 15 min/37 °C. Disks were then stored in 10 ml of 0.2 M NaCl solution (fluoride release/recharge) or 20 ml of deionized water (calcium release/recharge) at 37 °C. After the ion release reached to the base line, the disks were recharged (brushing the disk's surface with a gloved finger for 1 minute, and left undisturbed for 10 minutes, rinsed and dried) with either MI Paste (calcium recharge) or Clinpro 5000 (1.1% sodium fluoride, fluoride recharge). Release of ions was measured on Orion Model 710A+ 1 day, 3 days and 7 days after recharge.

Results: After 56 days of calcium release, or 70 days of fluoride release, the disks were recharged. Ion release results after recharge is shown in Fig. 1.

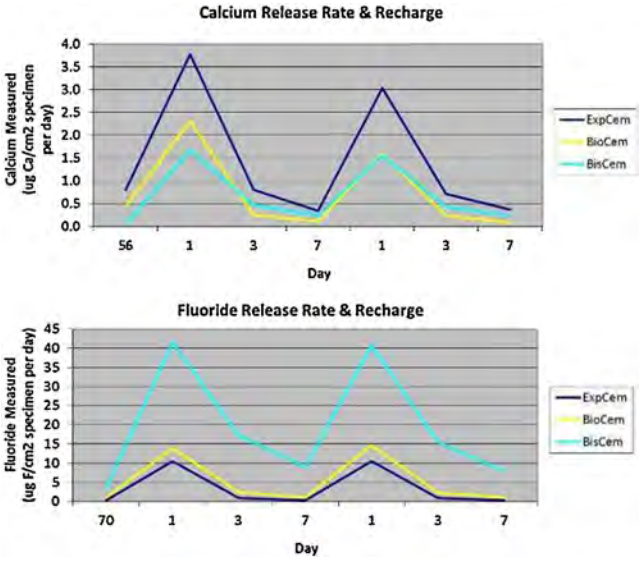

Fig. 1

Conclusions: All three of the cements demonstrated release and recharge of calcium and fluoride.

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Distance's influence on exposure reciprocity and the degree of conversion

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Purpose/Aim: To investigate the influence of distance on exposure reciprocity using multiple light-curing units (LCUs), evaluate exposure reciprocity effect on the degree of conversion (DC) of a RMC using multiple LCUs at two distances, and explore the correlation among irradiance (Irr), radiant exposure (RE) and DC.

Materials and methods: Six LCUs were explored: one quartz-tungsten-halogen (Optilux 401-O), three polywave light-emitting-diode (LED) (Bluephase Style-BS, SmartLite Max-SM and Valo cordless-V) and two monowave LED LCUs (Demi-D and Demi Ultra-DU). The RE (10 J/cm^2) was controlled and the Irr, RE and curing time for each LCU was measured using a Managing Accurate Resin Curing-Resin Calibrator (MARC-RC) at 0-, 2-, 4-, 6- and 8-mm ($n = 6$). RMC samples ($5 \times 5 \times 2$ mm) were fabricated ($n = 3$) using a nano-hybrid dual photoinitiator RMC and polymerized at 2- or 8-mm distance on a MARC-RC to measure the Irr, RE and DC at the top and bottom surfaces. Micro-Raman spectroscopy was used to measure the DC ($n = 50$/surface). UV-spectroscopy was used to measure molar absorptivity of camphorquinone (CQ) and diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (TPO) photoinitiators present in the RMC assessed. The effect of the LCUs and distance from the sample (2- or 8-mm) on Irr, RE and DC were analyzed using ANOVA. Pearson correlation coefficients were calculated to evaluate the linear associations among Irr, RE and DC ($\alpha = 0.05$).

Results: With increasing the distance between the LCU and the surface, Irr decreased and the time needed to reach 10 J/cm2 increased. The %change in curing time and Irr between 2- and 8-mm was distinct for each LCU and exposure reciprocity was not followed. Regardless of the surface, DC for SM and Irr of all LCUs were significantly higher at 2-mm compared to 8-mm. On the bottom surface, Irr and RE were significantly lower compared to the top. Polywave LED LCUs showed significant differences between the top and bottom except BS and V at 8-mm. However, all DC bottom/top ratios were >93%. The LCU had a significant effect on Irr, RE or DC irrespective of the surface. The molar absorptivity of TPO was approximately 20x more than CQ. A moderate correlation was displayed among Irr, RE and DC.

Conclusions: Exposure reciprocity was not followed for the LCUs explored and distance had an effect on Irr and curing time that was distinct for each LCU. Also, surface and distance had a significant influence on Irr, RE and DC. A moderate correlation was demonstrated among Irr, RE and DC.

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Effect of thermal cycle stress on universal adhesive systems

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Purpose/Aim: The purpose of this study was to evaluate the tensile bond strength of the universal adhesive systems to repair ceramic restorations after thermal cycle stress.

Materials and methods: Three types of the universal adhesive systems (SU, Scotchbond Universal Adhesive, 3M; GPB, G-Premio bond, GC; UP, Universal Primer, Tokuyama Dental) and a conventional adhesive system (RK, C&B Repair Kit, GC) for repairing of ceramic restoration were tested in this study. Surfaces of the ceramic blocks (GN Ceram Block, GC) were grinded with SiC paper, grit #600, and ultra-sonicated for 10 min. Each adhesive was applied on the surface according the manufacturer's instructions. Then a cylindrical stainless mold (3.0 mm height, 4.0 mm diameter) was put on the surface and resin composite was filled (MI LOW FLOW, GC). The specimens were stored in distilled water at 37 °C for 24 hrs. After the specimens were thermal-cycled, 5 °C for 30 s and 60 °C for 30 s, at 0, 1000 or 5000 cycles. Tensile bond strength, TBS, was measured by universal testing machine (cross head speed at 0.5 mm/min). The data were statistically analyzed by One-way ANOVA and Student–Newman–Keuls. ($P < 0.05$, $n = 12$).

Results: Overall, TBS seemed to be decreased with thermal cycle stress. TBS after 5,000 thermal cycles showed 2.5 ± 4.5 MPa for SU, 10.6 ± 7.1 MPa for GPB, 12.2 ± 5.5 MPa for UP, and 16.4 ± 3.1 MPa for RK. SU revealed significantly lower TBS than the other adhesives tested. For the interface observations, the failure modes showed to be various.

Conclusions: The results in this study suggested that the tensile bond strength of the one-bottle type universal adhesives systems had affected by thermal cycle stress.

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MTBS between zirconium ceramics and composite using various resin cements

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Purpose/Aim: The high bonding strength of ceramic to substrate can lead to the high clinical success rate of restorations. Although zirconium ceramics mainly contain zirconium oxide powder, various products of them are produced with different fabricating process. In addition, there are many resin cements available in the market nowadays, Therefore, this study aimed to evaluate the microtensile bond strength (MTBS) of zirconium ceramic from three manufacturers bonded to the resin composite using different resin cements.

Materials and methods: Nine groups of microtensile specimens were prepared from the zirconia-composite block bonded with resin cement. Three fully-sintered zirconium ceramic blocks ($5 \times 5 \times 10$ mm) from Katana (Nuvodent, Japan), Lava (3M ESPE,Germany) and Cercon (Degudent GmbH, Germany) were fabricated. The surfaces were finished with silicon-carbide abrasive and sandblasted with 50 μm alumina particles, followed by ultrasonic cleaning in distilled water. Three resin composite blocks (Filtek Z250, 3M ESPE, USA), were fabricated and finished with the same size. The ceramic and composite blocks were bonded using one of the three different resin cements: Panavia F 2.0 (Kuraray Dental, USA), Superbond C&B (Sun Medical, Japan) and RelyX™ Unicem (3 M ESPE, USA). After 24 hr, each block was cut under water coolant to produce microbar specimens, with bonding area 1 ± 0.1 mm^2. The MTBS was tested with universal testing machine (SHIMADZU EZ S, Japan) at a crosshead speed of 0.5 mm/min. The interface failures were examined for using a scanning electron microscope. Two-way ANOVA and multiple comparisons were conducted using Tukey's tests at P-value = 0.05.

Results: The MTBS of nine tested group range from 43.3 to 53.9 N/mm^2. Lava/Superbond produced the highest MTBS (53.87 N/mm^2), while Cercon/Panavia (43.28 N/mm^2) produced the lowest MTBS. The statistical analysis showed that types of zirconium-oxide ceramic ($P = 0.043$) and type of resin cement ($P = 0.047$) had an effect on MTBS, while the interaction between zirconium ceramic and types of resin cement ($P = 0.056$) was not significant. The fracture surfaces at magnification 100× and 2000×, revealed that Panavia F 2.0 and RelyX Unicem demonstrated predominantly cohesive failure in resin cement. Superbond C&B showed mixed of adhesive and cohesive failure. No adhesive failure was observed on the ceramic/cement or the composite/cement interface in all groups.

Conclusions: The MTBS of zirconium ceramic bond to composite using resin cement depended on the brand of zirconia and also types of resin cement.

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Inverse gas chromatography in composites-tooth hard tissues adhesion experiments

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Purpose/Aim: The aim of this study was to determine, by means of inverse gas chromatography, surface energy values and the work of adhesion between an exemplary composite restorative material and dentin and enamel before and after preparation with use of commercially available 4-step bonding systems. The value of surface energy expresses the level of

material or tissue activity and impacts the strength of adhesion between two bonded solid surfaces.

Materials and methods: The experiments were carried out on conventional gas chromatograph. A group of polar and non-polar test compounds was applied to define the ability of examined solids to interact in the way of dispersive or specific (acid–base) interactions. Tooth hard tissues fragments were prepared according to standard dental procedures with use of commercially available bonding system. Surface energy was also examined for one of the commercial restorative composites.

Results: Surface energy of tooth tissues changed under the influence of etchant, primer and bond. The values of the total surface energy before and after preparation were in the range of 29.15–150.79 (enamel), 48.92–104.28 (root dentin) and 64.12–124.73 (crown dentin) $mJ\,m^{-2}$. In case of enamel these differences were statistically significant ($p > 0.05$). In case of dentin, both crown and root, significant changes were observed after application of primer, with no further statistical changes after application of bonding agent. The work of adhesion between commercial composite and tooth tissues was changing according to surface energy changes. The values of this parameter were in the range of 78.22–177.76 (enamel), 104.97–148.29 (root dentin) and 120.13–163.63 (crown dentin) $mJ\,m^{-2}$.

Conclusions: Inverse gas chromatography allowed for fast and uncomplicated indirect examination of work of adhesion between tooth hard tissues (dentin, enamel) and exemplary restorative composite materials. The method is appropriate to estimate the adhesion strength between two bonded solid surfaces only on the basis of their surface activity. The values of surface energy and work of adhesion between dental composite and tooth hard tissues change according to tissue type and place of occurring (root, crown) and after preparation with use of bonding system. Application of etchant, primer and bonding agent changes significantly the values of surface energy of dentin and enamel, which is directly correlated with the bond strength between composite and hard tissues.

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Repaired anterior composite restorations: up to 15 years clinical survival

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Purpose/Aim: This retrospective clinical study investigated the influence of repair on the survival of resin composite restorations in anterior teeth with follow-up times between 4 and 15 years.

Materials and methods: The database was extracted from 396 records of 138 patients who attended a private dental office. In total, 628 anterior restorations carried out between 1994 and 2009 using hybrid, microhybrid, and microfill composites were included. The main outcome of the study was survival of the restorations, considering repair either restoration failure or success. Fisher's exact test was used to analyze the differences in failures within each category ($\alpha = 0.05$). Kaplan–Meier method was used to generate survival curves. A multivariate Cox regression analysis was used to investigate the influence of variables (age, gender, tooth type and position, cavity type, material) on restoration survival.

Results: In total, 198 repairs were performed (55 Class III and IV, 143 veneers), of which 65.4% (Class III and IV) and 63.6% (veneers) remained intact during the evaluation period. Age and gender were not significant factors affecting restoration longevity. The annual failure rate (AFR) of Class III and IV restorations up to 15 years was 3.2% when the repair was considered success, while it increased to 4.9% when the repair was considered failure. AFR for veneers was 10.4% after 10 years when the repair was considered success, and 17.8% when it was considered failure. When repair was considered success, survival rates increased between ~10% (Class III and IV) and ~20% (veneers). Higher survival rates were observed for mandibular restorations. Restorations in central incisors generally had lower survival rates compared to restorations in canines. The composite type influenced the survival of restorations only when the repair was considered success. In this scenario, restorations placed with microhybrid composites had lower survival rates compared to microfill restorations.

Conclusions: In conclusion, repair in anterior composite restorations may significantly increase the survival rates of restorations. Composite repair seems to be a viable alternative for treating failures in anterior restorations.

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Evaluation of bonding properties of G-Cem linkforce to ceramic restorations

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Purpose/Aim: All ceramic restorations have been coming under the spotlight from the aspect of their esthetics and the market for glass ceramics is expanding every year. For all ceramic restorations, resin cement with high bond strength is demanded. This year, G-CEM LinkForce was released as a primer typed resin cement having high bond strength. It is suitable for ceramic restorations. In this study, the bonding properties of G-CEM LinkForce to ceramic restorations including new pressable lithium-silicate glass ceramics "Initial LiSi press" were evaluated.

Materials and methods: Initial LiSi press (GC) and IPS e.max press (Ivoclar Vivadent) were prepared as adherent. Initial LiSi press and IPS e.max press were pressed into the shape which was diameter of 15 mm and thickness of 4 mm. G-CEM

LinkForce and G-Multi primer (GC), Multilink Automix and Monobond plus (Ivoclar Vivadent), Calibra and Calibra Silane Coupling Agent (Dentsply), NX3 and OptiBond XTR Adhesive (Kerr), and RelyX Ultimate and Scotchbond Universal Adhesive (3 M) were prepared as resin cement and primer. Each of pressed ceramics was polished in #1500 SiC abrasive paper. Primer was applied to the polished surface. The bonding area was prescribed in 3 mm diameter by a teflon tape with a hole. Stainless rod was sandblasted and applied Metal Primer Z (GC). A stainless rod and the ceramic specimen were bonded with resin cement (load; 10N, time; 10 s). The cement was cured for 24 h at 37 °C. Half the specimens were subjected to thermo-cycling (5–55 °C, 5000 times). Bonding tests were performed to each test group (crosshead speed; 1 mm/min.) ($n = 10$). The test results were analyzed statistically by Tukey-Kramer Method and One Way ANOVA ($p < 0.01$).

Results: G-CEM LinkForce showed the highest tensile bond strength to the glass ceramics after thermo-cycling. Those of Calibra, RelyX Ultimate and NX3 dropped to almost zero. The reason is G-Multi primer contains silane coupling agent 'γ-MPTS' and it bonds well chemically to the glass ceramics. On the other hand, Silane coupling agent in Calibra Silane Coupling Agent and Scotchbond Universal Adhesive don't work, OptiBond XTR Adhesive doesn't contain it.

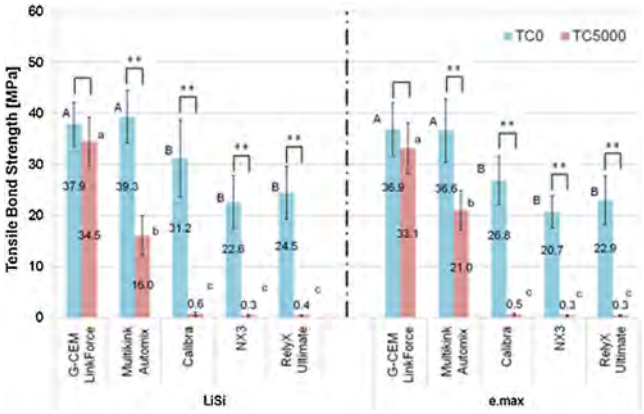

Fig. 1

Conclusions: This study indicated that only G-CEM LinkForce had high bond strength both before and after thermo-cycling, and suggested that G-CEM LinkForce was the most suitable resin cement for ceramic restorations. It is expected G-CEM LinkForce is superior to other resin cement in clinical cases.

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Drug release property of mucoadhesive hydroxypropyl methylcellulose based buccal patches

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Purpose/Aim: Topical steroid is the drug of choice for the treatment of inflammatory oral lesions. However, topical applications have low retention at the oral mucosa. Mucoadhesive hydroxypropyl methylcellulose (HPMC) polymers are well-known and widely used for buccal drug delivery systems. They enhance drug's ability to adhere onto the oral mucosa. The aim of this study was to prepare a new buccal mucoadhesive polymer based on HPMC for the delivery of topical steroid.

Materials and methods: Mucoadhesion and drug release property of the newly developed mucoadhesive polymer (3% HPMC) were tested. Testing of the mucoadhesion of the buccal patches were carried out using a texture analyzer (TA.XT plus, Stable Micro Systems, UK), equipped with a 50-N load cell and a bioadhesive holder. The chromatographic system (Shimadzu Corporation, Japan) was utilized for the measurement of drug delivery. The patches were tested at 2, 4, 6, 8 and up to 10 h and the eluate was monitored by UV absorbance at 252 nm. Each test was repeated for five times. A commercial product (Traful Direct, Kyu kyu Pharmaceutical Co Ltd., Japan) was used as a positive control. Triamcinolone acetonide was the topical steroid used for drug delivery experiments.

Results: It was found that 3% HPMC had significantly higher dissolution time and water absorption when compared to the commercial product. For mucoadhesion, 3% HPMC had comparable mucoadhesive force as the commercial product. And for drug release property, 3% HPMC had significantly higher release of topical steroid at every time point.

Conclusions: This study produced a buccal mucoadhesive patch as an alternative treatment of oral ulceration. The newly developed patch has mucoadhesion and drug release properties that are comparable and more favorable than a commercial patch.

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Color stability of novel bulk-fill composites

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Purpose/Aim: The aim of the present study was to evaluate the color stability of novel bulk-fill composites and to determine their color change after polymerization.

Materials and methods: A nanohybrid resin composite and five novel bulk-fill composites were included in the present study as follows; 3M Z550 (Z550), 3M Bulkfill Posterior (BLK), Voco Admira Fusion X-tra (ADM), Voco X-tra fil (XRF), Voco X-tra base (XRB) and Dentsply SDR (SDR). Forty-five specimens (6-mm in diameter, 2-mm in thickness) were prepared for each restorative material. The first color measurements of specimens were performed before polymerization. The specimens were filled into a Teflon mold on a Mylar strip whilst another strip was laid on top of the uncured composite surface and color measurements (T0) were carried out. The uncured specimens were cured in accordance with manufacturer directions. After polymerization, color measurements were repeated (T1) and samples were divided randomly into three groups accord-

ing to immersion media; distilled water, red wine and coffee. The color was recorded after 24 hour (T2), 1 week (T3) and 3 weeks (T4) of immersion and color change values were calculated using CIEDE2000 (ΔE00*) between T1 and T0 as well as between T2, T3, T4 and T1. The data were analyzed with Kruskal–Wallis and Mann–Whitney U test to represent the differences between groups, and Friedman and Wilcoxon tests were used to determine the changes in time. A confidence level of 95% was considered.

Results: Color difference value for each composite range from 3.29 to 13.00. All of the resin composite showed perceptible color change (ΔE00 > 2.25) after setting. In distilled water, Z550 and ADM showed higher color change than clinically acceptable threshold 2.25 after 24 h. In addition, 3 w immersions in distilled water exposed statistically increased color changes values for all composites except BLK and XRF. The ΔE00 values range from 1.94 to 10.91 at the end of the first day of immersion, whilst 3 weeks later values range from 6.28 to 20.39 as shown. Similarly, resin composite discolorations increased in time for coffee groups. This discoloration was found statistically significant ($p < 0.05$).

Conclusions: All of the bulk-fill composites, which were used in this study, were sensitive to staining in beverages. Additionally, in limitations of present study, it can be concluded that setting reaction significantly caused discoloration within bulk-fill composites.

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Effect of polar solvents on the elastic modulus of dentin

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Purpose/Aim: The purpose of this study was to determine the elastic modulus of demineralized dentin beams immersed in various concentrations of dimethyl sulfoxide (DMSO) or ethanol for different time intervals.

Materials and methods: Dentin beams (1 mm × 2 mm × 6 mm) were completely demineralized in 10% phosphoric acid. After baseline measurements of elastic modulus (E) (3-point bending, 15% strain) the beams were divided into 9 groups ($n = 10$/group). Beams were immersed in 1, 10, 50 or 100% of DMSO or ethanol for10, 30, and 60 min. The beams immersed in distilled water only served as control. After each time interval, modulus of elasticity of each specimen was re-measured using the universal testing machine (Shimadzu corporation, Japan). After solvent incubation, each group was incubated in distilled water for 24 hrs and modulus of elasticity was reassessed. Data were statistically analyzed using ANOVA at $\alpha = 0.05$.

Results: Both the solvent type and the application time showed significant effects on E ($p < 0.05$). Modulus of elasticity of DMSO treated beams ranged between 2.01 and 13.44 MPa for 0, 100% DMSO treated beams respectively. Higher concentration of DMSO (50,100%) showed significantly higher E compared to lower concentrations ($p < 0.05$). The modulus of elasticity of ethanol treated beams ranged between 2.38, 17.8 MPa. Significant increase was observed with only 100% ethanol treated beams ($p < 0.05$). The change in E was fully reversible upon 24 hrs rehydration.

Conclusions: The elastic modulus of the dentin was increased with concentration of the solvent and immersion time but the effect was reversible upon rehydration in water.

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Bacterial adhesion and treatment efficacy on 5 modified titanium surfaces

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Purpose/Aim: To evaluate the bacteria adhesion behavior and antibiotics treatment efficacy of pure titanium surfaces modified with 5 different techniques, explore the effect of surface properties on these characters.

Materials and methods: Pure titanium foils were treated with 5 surface modification techniques, namely mechanically polishing (MP), dual acid-etching (DAE), sandblast-large grit and acid-etching (SLA), micro-arc oxidation (MAO) and anodize oxidation (titania nanotube with pure anatase, TNT-A) respectively. Surface topography was observed by scanning electron microscopy (SEM). Surface roughness was detected by laser scanning confocal microscope (LSCM). The surface water static contact angle was measured by contact angle analyzer. The microcosm were co-cultered on the above 5 groups of specimens respectively. The bacteria adhesion was measured by colony forming units (CFUs) counting and crystal violet staining. The remaining cell viability after chlorhexidine (CHX) treatment was evaluated by microbial viability kit.

Results: MP, DAE, SLA and MAO surfaces demonstrated a structure of a micro scale between 1 and 10 μm, while TNT-A surfaces demonstrated a nano-structure with nanotubes which were about 80 nm in diameter. The surface roughness of MP and TNT-A was the smallest, followed by DAE and SLA consequently, and MAO owned the highest roughness. All 5 surfaces were hydrophilic. Among them, the smaller water static angles of MAO and TNT-A revealed better hydrophilia. The amount of viable organisms all increased with the incubation time on 5 surfaces. At 1 d, the amount of viable microcosm on MAO surfaces was far above that on MP, DAE and TNT-A surfaces; at 5 d, that of MAO surfaces was the highest with no significant difference among other four surfaces. The biomass of microcosm on 5 surfaces at different incubation periods were almost in accordance with the corresponding attachment amounts of viable organisms. At 1 d, after the treatment of CHX, the rank of the microcosm biofilm remaining viability on five surfaces was MAO > DAE/SLA > MP/TNT-A; at 5 d, the

rank was MAO > SLA> MP/TNT-A, and DAE demonstrated no significant difference with neither MAO nor SLA.

Conclusions: Surface topography and roughness both affect the early adhesion of bacteria. However, this effect can be weakened with the maturity of the biofilm. Surface topography and roughness significantly affect the antibiotics treatment efficacy of the biofilms on the surfaces. The nano-structured TNT-A surface reveals lower adhesion of microcosm and higher antibiotics treatment efficacy.

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Synthesis and biofunctionalization of extracellular matrix hidrogel for bone regeneration

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Purpose/Aim: Untreated periodontal disease leads to tooth loss through the destruction of the attachment apparatus and supporting structures. The ultimate goal of periodontal therapy is the regeneration of the destroyed tissues, including bones. Bone is the second most transplanted tissue after blood. Autologous implant is the gold standard for bone repair and reconstruction. However, the material is taken from another site of the patient, having a double surgical approach, and may lead to pain and infections at these healthy areas. Therefore, new materials are needed in bone healing therapy. The extracellular matrix (ECM) is composed of a diverse mix of proteins, glycoproteins and glycosaminoglycans, and could be an excellent bone scaffold. The objective of this study was to synthesize and biofunctionalize an extracellular matrix hydrogel of porcine bladder with calcium salts for bone regeneration.

Materials and methods: The ECM was decellularized using two techniques with different detergents (Triton X-100 and SDS), lyophilized, pulverized and digested with pepsin for gelatinization, different concentrations (20, 50, 100 mg/mL) of hydroxyapatite (HA) and tricalcium phosphate (TP) were added and rheology tests and evaluation of growth factors (BMP-4, TGF-β, BFGF) were conducted. In addition, osteoblast proliferation, cytotoxicity, cell viability and morphology were evaluated.

Results: Proteins from complete ECM and processed with Triton X-100 or SDS showed concentrations of 208.31 ± 10.0 mg/g, 124.74 ± 38 and 113.39 ± 11.0 respectively. The biofunctionalized hydroxyapatite group (HA50) showed the better performance in rheology assays with 43.3 Pa, in comparison to non-biofunctionalized ECM (26.1 Pa). No differences were detected for growth factors among the groups. In the proliferation assays the HA20 and non-biofunctionalized ECM groups showed a higher number of osteoblasts (109.66 ± 1.98% and 103.0 ± 1.30%) with normal morphology and viability.

Conclusions: A hybrid scaffold was formulated which showed optimal characteristics for bone regeneration. However, it is necessary to perform *in vivo* studies that corroborate these results.

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In vivo evaluation of ceramic-ECM scaffolds for bone regeneration

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Purpose/Aim: An ideal bone substitute should be compatible and resorbable, provide calcium and phosphate, inexpensive and readily available; searching this characteristics, different bone substitutes have been assessed during the last 60 years in three clearly marked generations: bioinert materials (first generation), bioactive and biodegradable materials (second generation) and materials designed to stimulate specific cellular responses at the molecular level (third generation). In this last generation of bone implant, the goal is to mimic the extracellular matrix (ECM) environment to provide the development of specific cell response. The ECM is a complex mixture of structural and functional proteins, glycoproteins and proteoglycans arranged in a unique three-dimensional network that envelopes all the organs, tissues and cells of the body. Biological scaffolds derived of these materials have been successfully used in clinical applications so we believe that could be used in bone regeneration as a biphasic construct with osteoinductive materials such hydroxyapatite and tricalcium phosphate.

Materials and methods: We decellularized ECM from porcine urinary bladder and biofuctionalized it with hydroxyapatite (HAP) or tricalcium phosphate (TCP). We evaluate their effect on in vivo regeneration of bone critical size damage in ratio orthopedic surgery of 4 month-old female Wistar rats, by means of radiographic image, histological and serological evaluations.

Results: There was a better regeneration in ECM groups compared with negative and positive control as shown in the radiographic and histological evaluation. The inflammatory reaction was tested by TNF-α and IL-1β, the ECM groups presented a similar activity to the positive control. At the first month TNF-α concentrations were 8.24 pg/ml for positive control, 9.85 for negative control, 9.19 pg/ml for ECM, 6.82 pg/ml for ECM + HAP and 10.69 pg/ml for ECM + FTC; at the same period IL-1β concentrations were 27.72 pg/ml for positive control, 34.63 for negative control, 36.26 pg/ml for ECM, 29.67 pg/ml for ECM + HAP and 33.98 pg/ml for ECM + FTC.

Conclusions: The evaluated biofuctionalized scaffold successfully promotes bone regeneration, without causing important immunological reactions.

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DPCs/hydrogel 3D-setup for materials biocompatibility analysis

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Purpose/Aim: The in-vitro evaluation of dental resin composites biocompatibility is commonly carried out using a monolayer cell model with conditioned medium. Such an indirect and discontinuous method renders difficult the analysis of the impact of monomer leaching kinetics and intensity on cells organized in a 3D tissue. The objectives of this work were (1) to determine an appropriate alginate/fibrin hydrogel for dental pulp cells (DPC) viability and (2) to test the biocompatibility of two experimental resin composites using the hydrogel model compared to a monolayer cell model.

Materials and methods: Four alginate-fibrin (alg-fib) formulations were prepared: 100–0, 50–50, 25–75 and 0–100 alg-fib wt%. The "alginate" and the "fibrin" fractions consisted of [alginate_$CaCO_3$_glucono-delta-lactone] and [fibrinogen_thrombin], to which DPC's were added (25000 cells/70 μL gel). Viability, cell adhesion and proliferation was assessed 1 and 7 days after gel formation using confocal microscopy ($n = 2$, Hoechst/calcein staining). Following freeze-drying, the gels structures were observed by scanning electron microscopy.

Model experimental resin composites (70/30 Bis-GMA/TegDMA, 75 wt% fillers) were prepared based on either camphorquinone (CQ) or monoacylphophine oxide (MAPO) as photoinitiator, following previous works (conversion is 50 or 60%, respectively). Disks (5 × 0.5 mm) were light-cured (3 or 20 s at 1000 mW/cm^2) and then embedded in 1.5 mm-thick 50–50 alg-fib gels. Confocal microscopy was then used, as described above ($n = 3$). Gels without material were used as controls. Cell monolayers were formed in 96-well plates and incubated with pre-conditioned medium (prepared in advance with one disk/mL medium, incubation 1 week). Cell viability was assessed at the same timepoints using an MTS test (absorbance at 490 nm A_490 nm, $n = 4$).

Results: The viability (intensity of calcein staining) increased in all formulations between day one and seven. Viability at day seven was highest in the 50–50 hydrogel and lowest in the 100–0 one. Confocal microscopy showed adherent cells, spreading in the fibrin-containing formulations, while cells remained spherical in 100–0 alg-fib. All gels appeared highly porous when analyzed by SEM. Cell viability in proximity with the materials was the lowest in gels with the – low conversion – CQ-composite. After one week of incubation, cell adhesion and proliferation was observed in both groups but was clearest with MAPO-composite. In contrast, no difference between groups was noted at either timepoint in the monolayer model (A_490 nm).

Conclusions: A 3D model DPC-loaded fibrin/alginate hydrogel is suitable for analyzing the biocompatibility of restorative materials: increasing conversion and replacing CQ by TPO was associated with reduced toxicity. The 3D setup appeared more sensitive and relevant than the monolayer model commonly used in-vitro.

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Sodium trimetaphosphate as protease inhibitors/remineralizing agent of artificial caries-like dentin

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Purpose/Aim: Since evidences have been pointed out sodium trimetaphosphate (STMP) as an effective remineralizing agent for enamel, its use in dentin has been also investigated associated with its biomimetic potential. Therefore, the aim of this study was to evaluate the anti-proteolytic and biomimetization potencial of STMP in dentin previously submitted to acidic challenge.

Materials and methods: The evaluation of anti-proteolytic potential of STMP at 0.5, 1.5, 2.5, 3.5 and 5% over matrix metalloproteinase 2 and 9 (MMP-2 and 9) was assessed by zymography. For mechanical analysis, 64 bovine root blocks (6 × 4 × 4 mm) were randomly divided in 4 groups ($n = 8$) accordingly to the concentration of STMP that had the capacity to inhibit 100% the MMPs activity. All dentin blocks surface were divided in 3 areas: 1-control- no treatment; 2-demineralized- submitted to a demineralized solution (7d/37 °C); 3-treated- the area was demineralized and then submitted to a pH-cycling using treatment solution (H2O, 1.5, 3.5 or 5% STMP) (10 min), demineralized/remineralized solution (8 h and 16 h, respectively) (7d/37 °C). After the pH-cycling, dentin blocks were analyzed by superficial (SH) and cross-sectional (CH) hardness in different depth (10, 30, 50, 70, 90, 110 and 220 μm) using a Knoop penetrator (10 g/10 s). To quantify the relative inhibition of gelatinases by STMP, the gels were scanned electrophoretic bands and analyzed the band intensity. SH and CH data were statistically analyzed by ANOVA and Tukey tests ($p < 0.05$).

Results: The zymography analysis showed that the 1.5, 2.5, 3.5 and 5% STMP solutions were able to promote 100% inhibition of gelatinolytic activity of both MMPs. The concentration

of 0.5% was not able to completely inhibit the enzyme activity. Only the solution containing 1.5% STMP was able to enhance both the SH and CH.

Conclusions: 1.5% STMP solution was effective as anti-proteolytic activity against MMP-2 and 9 and to promote remineralization of demineralized dentin.

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Fatigue resistance and damage modes of lithium-disilicate and nanoceramic resin

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Purpose/Aim: Bulk fractures or cohesive fractures are frequent complications for ceramic crowns. The use of resin-based materials, in contrast to the brittle nature of ceramics, seems to be an alternative to posterior crowns, since these materials present simplified manufacturing and repair techniques. This study evaluates the fatigue resistance of monolithic CAD/CAM crowns made of resin nanoceramic and lithium disilicate reinforced ceramic, as well as the damage modes caused by the application of cyclic loading.

Materials and methods: Twenty-six monolithic crowns ($n = 13$) were cemented to a composite resin replica of a prepared molar and subjected to cyclic load over 2 million cycles under maximum load of 350 N, at a frequency of 2 Hz. After loading, the crowns were embedded in epoxy resin and gradually grinded for damage analysis.

Results: The crowns presented no catastrophic failures or cohesive fractures, resulting in survival of 100% for both materials. Nanoceramic resin crowns presented no damage, outer cone cracks, inner cone cracks and radial cracks. Lithium disilicate crowns showed outer and inner cone cracks, some of the latter reaching the inner surface. Specimens were scored according to the severity of damage. Data was subjected to Mann–Whitney test ($p = 0.462$), which revealed no statistical difference.

Conclusions: It was concluded that lithium disilicate and resin nanoceramic monolithic crowns can be used in the posterior area, since they presented comparable fatigue resistance, with no statistical difference between damage modes.

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Behavior of CAD/CAM materials after long thermocycling process

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Purpose/Aim: The recent development of new CAD/CAM blocks made of polymer-based materials creates the possibility of independent studies on these materials. The aim of this study was to determine the flexural strength and flexural modulus behavior after long thermocycling process.

Materials and methods: The materials chosen to be included in this research were the newly polymer-based materials; Vita Enamic (Vita Zahnfabrik), Lava Ultimate (3M ESPE), and the dental ceramics already well established; Vitablocs Mark II (Vita Zahnfabrik), and IPS e.max CAD (Ivoclar-Vivadent). Beam specimens were prepared using a precision slow-speed cutting machine; after several cuts to have the bars on the correct size ($2.5 \times 3.0 \times 14$ mm), they were polished to avoid any edge chipping that could interfere with the 3-point bending test. Thirty-six beam shaped bars were made for each material and divided into four subgroups ($n = 9$): (1) desiccator for 7 days, (2) 37 °C for 7 days, (3) 60,000 thermocycling, and (4) 120,000 thermocycling (5 °C and 55 °C with a dwell time of 52 s). The specimens were subjected to a 3-point flexural test on a 12 mm span with a crosshead speed of 0.5 mm/min. The results were analyzed by two-way ANOVA and post-hoc Tukey's test was performed for both variables.

Results: The mean of flexural strength (SD) and flexural modulus (SD) among the materials were the highest for IPS e.max CAD 396.19 (75.65) MPa and 84.97 (11.12) GPa, respectively. Vitablocs Mark II presented the lowest mean for flexural strength 125.18 (10.07) MPa, while Lava Ultimate had the lowest for flexural modulus 12.90 (3.73) GPa. From the subgroup (1) to subgroup (4) on the flexural strength, the values always ranged from Vitablocs Mark II with lowest means and IPS e.max CAD with the highest ones, while the flexural modulus presented the lowest values for Lava Ultimate and the highest for IPS e.max CAD. SEM fractography identifies a plastic behavior for Lava Ultimate, however, the others materials had brittle fractures.

Conclusions: The development of dental materials allows the recent polymer-based materials perform better than some ceramics, however, one of the major problems related to polymer-based materials still remain irresolvable. The drop of the values in flexural strength made it clear the weakness of the materials under moisture conditions.

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Notchless trianguler prism fracture toughness of new indirect composite

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Purpose/Aim: We developed new indirect composite system, GRADIA PLUS LB (Light Body)/HB (Heavy body). GRADIA PLUS overcomes weaknesses of Micro-Filled Resin (MFR) by adopting nano-filler technology same as G-aenial universal flo (GC) and CERASMART (GC), demonstrating high wear resistance and high mechanical properties. Fracture toughness of dental materials is evaluated as a method to measure a resistance level of the destruction. Recently, notchless trianguler prism (NTP) fracture toughness test has been paid attention as an effective method for measuring fracture toughness of composite resin. The purpose of this study is to evaluate the NTP fracture toughness of GRADIA PLUS and other indirect composites.

Materials and methods: GRADIA PLUS LB/HB, GRADIA (GC), Signum ceramis (Heraeus Kulzer), crea.lign flow (Bredent), crea.lign paste (Bredent) and SR nexco paste (Ivoclar/Vivadent) as indirect composites were examined in this study. NTP specimens of each indirect composite were formed for each material using metal mold and cured according to the manufacturers' instructions for use. All specimens were stored in 37 °C water for 24 hours. NTP fracture toughness test was performed with multi testing machine (Autograph, SHIMADZU), and a fracture morphology of each prism was inspected by scanning electron microscope (SEM, HITACHI). KIC was calculated by following equation, KIC = Pmax × Y*min/(DW0.5) where P_{max} = the maximum load at fracture (N), D = the specimen diameter (12 mm), W = the specimen length (10.5 mm), and Y*min = the minimum of the dimensionless stress intensity factor coefficient (= 28). Results were analyzed by one-way ANOVA ($p < 0.05$).

Results: Evaluation of NTP fracture toughness needs that the fracture morphology is indicative of plane strain conditions, so fracture surface was observed by SEM. GRADIA PLUS LB and HB exhibited significantly higher NTP fracture toughness compared to the other indirect composite (shown in Fig. 1). Fracture toughness was influenced with many factors which are by size, form and content rate of glass filler and strength of resin matrix in composite resin. Results of this study suggested that GRADIA PLUS shows higher fracture toughness due to nano-filler technology.

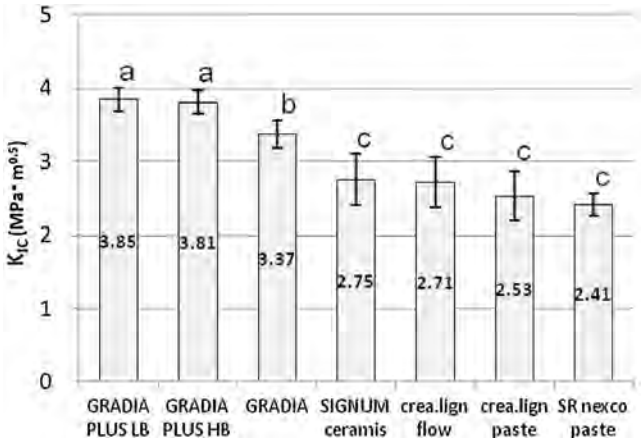

Fig. 1

Conclusions: New indirect composite, GRADIA PLUS LB and HB, showed higher NTP fracture toughness than the other indirect composite, and it may suggest that GRADIA PLUS is not easily fractured for clinical use.

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New calcium sulphate powder-binder system for 3D printing

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Purpose/Aim: Develop a new powder-binder system for 3D printing used in maxillofacial prosthesis.

Materials and methods: The SEM and EDS study of zp® 151 3DSYSTEMS® was performed with the scanning electron microscopy low vacuum. The infrared spectroscopy study of zp® 151 powder and liquid ZB ™ 61 Clear Binder Solution 3DSYSTEMS® were performed on a spectrometer Fourier transform. For the thermogravimetric analysis the sample was placed in the thermobalance in air atmosphere and heated at a rate of 10 °C per minute. Subsequently the bands databases that marks the literature were compared. For the synthesis of the binder solution, polyacrylic acid (PAA) at 1.5%, 1%, 0.75%, 0.5% and 0.25% and deionized water were mixed using a magnetic stirrer until the polymer completely dissolved. For the powder, Dental® mdc, NICSTONE, and Zhermack®, dental stones elite®, were tested. Subsequently, samples in stainless steel molds, 4 mm diameter by 6 mm height were made and it allowed dry the samples at room temperature, 24 exact times were subjected to a load of 50 ± 16 N/min, the resistance and modulus of elasticity values were obtained as averages.

Results: The composition of the sample according EDS were: 48.10% O, 27.32% Ca, 8.14% S, 9.02% Si, 1.16% Na, and 0.32% Mg. Also the shape of the powder particles was: mostly irregular prisms of 50–80 μm. In the IR analysis of the powder, concordant bands with molecules of calcium sulphate as the link S-O between 1100 and 1400 were detected. In the IR liquid the three water bands characteristics (3225, 1625, 994) were found, and a small band 2124 corresponding to a triple bond who corresponds to an acetylene-based acrylic polymer. As a result of the mechanical tests an average value of compressive strength of the original material of 15.4 MPa and an elastic modulus of 1997 MPa was achieved, taking into account the different formulations were tested, which came closest to the original powder and binder was dental gypsum elite® with PAA to 0.25%.

Conclusions: With this study the components and characteristics of the material used in stereolithography were identified, the powder is a calcium sulphate and the binder comprises a polymer dissolved in water. With the previously mentioned data, was developed a new formula for the benefit

of the institution and the community in general. Also new lines of research for develop new materials arise.

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Biological properties of fiber reinforced composites (FRCS): A systematic review

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Purpose/Aim: Recently, Fiber Reinforced Composites (FRCs) have received much attention due to their potential use in dental implants or as bone fracture fixation applications. The aim of this study was to investigate the biological properties of FRCs through systematically reviewing and assessing the existing literature.

Materials and methods: A systematic search of five databases (PubMed/MEDLINE, Scopus, Web of Science, and Cochrane library) was performed to identify all relevant studies, published between 1962 and 2016. The search was limited to in-vitro testing studies published in English. Citation mining was also performed through cross-referencing of included studies and hand searching of relevant journals.

Results: One thousand and ninety-one articles were initially identified, out of which twenty-seven articles were full-text screened, and eleven included for review. Three studies investigated bacterial adherence properties and growth ability on FRCs, three studies investigated the cytotoxicity of different surface-treated FRCs on fibroblasts, and five studies investigated the osseointegration between bone and FRCs. There was obvious heterogeneity between the included studies evident through the testing of varying fiber types, the use of different FRC-coating approaches, and the lack of standardized testing protocols. As such, a meta-analysis was not feasible. The included studies indicated that bacterial binding was present significantly more on ceramic and polyethylene FRCs, when compared to glass FRCs, and aramid FRCs, due to their surface physicochemical properties, capacity of agglutinin adsorption from saliva, and water storage time. Furthermore, E-glass FRCs exhibited the lowest fibroblast cytotoxicity followed by Silicate glass FRCs and S-2 glass FRCs. Additionally, 95% of cells on FRCs were viable, with a comparable cellular response to that on titanium, especially when FRCs were coated with bioactive glass or phosphate.

Conclusions: The findings of this review demonstrated that FRC's are cytocompatible materials, possessing satisfactory biological properties that support their use in dental implants or as bone fixation appliances. Further research is necessary to regulate matrix ion release/degradation of FRCs in order to prolong the initially demonstrated properties. Introduction of antibacterial components, into the FRC matrix system, can also be considered in future studies to improve FRC-response to various microbial/biofilm challenges.

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WITHDRAWN

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Effect of bioactive glass on the remineralization of demineralized dentin

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Purpose/Aim: The demineralization of dentin is the initial pathologic changes that can lead to destruction of the dentin. Bioactive glass (BAG) is an excellent biocompatible material for tissue mineralization. So, the aim of this study is to evaluate the possibility of remineralization of completely demineralized dentin using bioactive glass.

Materials and methods: Ten caries-free extracted human 3rd molars were obtained under protocol approved by the Institutional Review Board committee of Kyung Hee Medical Center (KHD IRB 1507-4). Two dentin blocks ($2 \times 7 \times 0.9$ mm) were obtained from each tooth using a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water-cooling. They were completely demineralized in 0.2 M formic acid solution for 10 days, and thoroughly washed with distilled water. After that, they were randomly divided into two groups: Deminerlized dentin (DE) group ($n = 10$), Bioactive glass-treated (BAG) group ($n = 10$). The specimens of DE group were stored in deionized water, while those of BAG group were immersed in the slurry of BAG 45S5 (Mol, SCI, USA)/deionized water (1:1 mixture) for 7 days. Following modalities were performed to analyze the remineralization effect of BAG: (1) FE-SEM/EDX: The specimens were fixed with 2.5% glutaraldehyde(4 h) and dehydrated in ascending order with 25%, 50%, 75%, 90%, and 100% ethanol and dried with hexamethyldisilazane (Sigma–Aldrich, USA). Micro-morphologic structures of the specimens were evaluated using FE-SEM (Leo Supra 55, Carl Zeiss, Germany); (2) Raman Spectroscopy: The mineral phase of specimens were examined using a computer-controlled confocal laser Raman apparatus equipped with a Leica DM/LM optical microscope and CCD detector attached to a modular research spectrograph; (3) XRD Analysis: Optical emission spectrometry technique (ICP-Spectrometer Plasma 2000, Perkin Elmer, Germany) was used for elemental analysis.

Results: In the FE-SEM/EDX, BAG particles were observed on the completely demineralized collagen matrix in the BAG group. The Crystalline phase of the dentin surface was confirmed through XRD and RAMAN in the BAG-treated group. It is similar to the crystalline phase in mineralized dentin.

Conclusions: Within the limitation of this study, BAG is able to remineralize the demineralized dentin although it is completely demineralized.

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Bonding durability of two universal dentin adhesives

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Purpose/Aim: The purpose of this study was to evaluate bonding durability of two universal dentin adhesives with NaOCl artificial aging. The adhesive interface was analyzed with scanning electron microscope (SEM). Field-emission scanning electron microscope (FE-SEM) analysis was used to evaluate the degradation effect by NaOCl aging after various dentin adhesives treatments.

Materials and methods: 36 extracted 3rd molars had their occlusal surfaces removed and were polished serially with 320- to 600-grit silicon carbide abrasive paper to produce a standard smear layer. They were divided randomly into 6 groups according to the application mode of four dentin adhesives [XP-Bond (total-etch), SE-Bond (self-etch), All-Bond Universal (total-etch or self-etch), Single Bond Universal (total-etch or self-etch)]. After adhesive application following the manufacturers' instructions, 4-mm composite build-ups were incrementally placed in 2-mm increments. After 24 h of storage in distilled water, 30 composite-dentin beams of $1.0 \times 1.0\,mm^2$ were obtained for each group. These beams were divided into two sub-groups. No treatment was performed to first 15 beams. Other 15 beams were stored in 10% NaOCl solution for 1 hour. Microtensile bond strength (μTBS) was measured by the crosshead speed of 1 mm/min. Results were analyzed with two-way ANOVA and Tukey HSD post-hoc test at 95% significance level. Sectioned tooth slabs with 1.0 mm thickness were produced for each experimental group. The adhesive interfaces were observed under SEM before and after NaOCl aging. Demineralized dentin blocks were stored in 10% NaOCl for 3 min after various dentin adhesives application. Six differently treated dentin blocks were observed with FE-SEM.

Results: In no treatment groups, μTBSs of All-Bond Universal (total-etch) and SE-Bond are significantly higher than other experimental groups ($p < 0.05$). In NaOCl aging groups, μTBS of XP-Bond was significantly lower than other experimental groups ($p < 0.05$). μTBSs of XP-Bond, All-Bond Universal (total-etch) and SE-Bond significantly decreased after NaOCl aging ($p < 0.05$). SEM analysis revealed that more erosive pattern of demineralized collagen in total-etch mode. It was predominant around resin tags. In self-etch mode, erosive pattern was not clear. FE-SEM analysis revealed that unprotected collagen fibers effectively degraded by NaOCl. When collagen fibers were protected with dentin adhesive, the degradation effect of NaOCl decreased.

Conclusions: Universal adhesives used in this study showed similar μTBS to conventional dentin adhesives before aging. They showed stable adhesive performance after NaOCl aging.

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Phosphate based glass as bone graft and scaffold material

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Purpose/Aim: The main aim of this study is developing osteoconductive and osteoinductive material that can enhance bone repair and has a potential in dental associated bone regeneration and repair procedures. This was done by: (1) the development of glass beads as primary material for scaffold formation. (2) The assessment of biocompatability of the glass beads on cellular basis by using hMSCs (human mesenchymal stem cells. 3- Preparation of scaffold that has both mechanical and feature properties that may be used for bone related dental procedures.

Materials and methods: Glass beads production: four compositions of glass beads were prepared by melt-quench technique followed by spheriodization. These glass compositions were SrO17.5%, SrO35%, ZnO_5% and ZnO_{10}% where the glass composition was $(50P_2O_5 - 10Na_2O - 5TiO_2 - (35-x)\,CaO-x$ (SrO or ZnO)), x was the percentage of zinc and strontium oxides. These glass beads were used to make 2 mm thickness of multilayers in tissue culture inserts which were used as scaffold for hMSCs later on. The biological effects of glass beads scaffolds were assessed by Ca concentration and alkaline phosphate assays which are osteodifferntiation biological markers.

Scaffold construction: the most appropriate Zn and Sr compositions from the cell studies were used in scaffold construction by using glass powder particles size (850 μm–1000 mm). Powder was compacted in 5 mm diameter cylindrical graphite mold then heated for 1.5 hour at 3

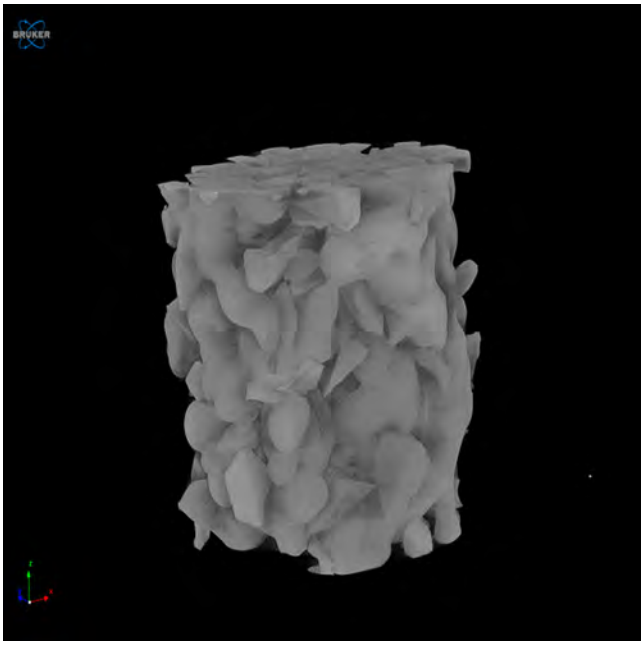

Fig. 1

different temperatures in the glass transition temperature period to optimize the best degree for scaffold production. The optimization was done by: (1) examining the compressive strength, (2) calculation of porosity percentage of glass and the range of porous size that are suitable for bone growth by using microCT.

Results: Biological effect: Both Ca and alkaline phosphate assays data for hMSCs showed that all the zinc and strontium phosphate glass can enhance the osteogenic differentiation in comparison to the control group. Scaffold construction optimization: compressive strength results showed that the mechanical properties are enhanced with temperature increase in scaffold making. Conversely, porosity was decreased with temperature rise.

Conclusions: Glass beads preparation was successful as a step to make glass in suitable features that can be used suitably as scaffold material. Glass beads enhance the differentiation of stem cells to osteoblastic lineage which was shown clearly in SrO17.5 and ZnO 5 in comparison to the control and other groups. Production of Cylindrical Bone scaffold from phosphate based glass was successful and fulfilled the spatial and porosity requirements for bone growth.

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Effect of conditioning solutions on dentin bond strength of adhesives

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Purpose/Aim: The aim of this study was to evaluate the influence of experimental dentin conditioners on bond strength of two adhesive systems.

Materials and methods: Eighty human third molars were obtained after approval by the review board of the Institutional Ethics Committee (#091/2015) and stored in thymol solution. The occlusal enamel was removed with a diamond blade to expose the dentin surfaces. The teeth were polished with a silicon carbide paper (600-grit) and randomly divided into ten experimental groups ($n = 8$): (1) 37% phosphoric acid (PA) + XP Bond (Dentsply) – wet dentin; (2) PA + XP Bond–dry dentin; (3) PA + One Step (Bisco)–wet dentin; (4) PA + One Step–dry dentin; (5) 10^{-3} solution + XP Bond; (6) 10^{-3} solution + One Step; (7) 6.8% ferric oxalate + XP Bond; (8) 6.8% ferric oxalate + One Step; (9) 1.4% nitric acid + XP Bond and (10) 1.4% nitric acid + One Step. The conditioners were applied on dentin surfaces for 15 s, except for ferric oxalate, which was applied for 60 s. The etched surfaces were washed and air-dried for all groups, excepted for groups 1 and 3, in which the dentin was kept moist. After bonding, three layers (2 mm thick) of a composite resin (EsthetX, Dentsply) were placed onto dentin surfaces. Each layer was photoactivated separately. The teeth were sectioned to obtain specimens of 1 mm2 at cross section for the microtensile bond strength test. The specimens were tested in a universal testing machine (EZ Test, Shimadzu) after 24 h of storage in distilled water.

Results: The bond strength results are presented in Table 1.

Table 1 – Mean (SD) microtensile bond strength values of each group (MPa) according to conditioners and bonding agent.

Etchant Agent	XP bond	One step
37% Phosphoric Acid–Wet Dentin	54.5 (6.6) Aa	35.9 (9.2) Bab
37% Phosphoric Acid–Dry Dentin	46.6 (5.2) Aab	19.5 (6.0) Bc
10-3 Solution–Dry Dentin	40.2 (8.7) Abc	41.2 (7.6) Aa
6.8% Ferric Oxalate–Dry Dentin	21.9 (3.0) Ad	16.2 (3.0) Ac
1.4% Nitric Acid–Dry Dentin	36.8 (7.5) Ac	30.0 (8.3) Ab

Uppercase letters compare adhesives for the same etchant (rows) and lowercase letters compare etchants for the same adhesive (columns) ($p > 0.05$).

Conclusions: For XP Bond, the alternative dentinal conditioner solutions yielded lower bond strength than PA, while for One Step, 10-3 solution and the nitric acid groups showed no significant difference compared to PA with wet dentin. This work was supported by São Paulo Research Foundation (#2015/03927-3).

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Static and cyclic flexural strength of various dental composite resins

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Purpose/Aim: Resistance to fatigue (cyclic loading) of dental restorative materials has been considered more relevant to clinical longevity than monotonic flexural strength. The purpose of this study was to investigate the flexural fatigue strength of several composite resins and the interrelation with their static flexural strength.

Materials and methods: Eight dental composite resins including nanohybrids and bulk fill composites were investigated; one microfill (Metalfil CX), one packable (Surefil), two bulkfills (Tetric-N Ceram, Sonicfill), and four hybrids (Z100, Filtek Z350, Synergy D6, Nexcomp). Flexural specimens ($2 \times 2 \times 25$ mm) were prepared in conformity with the ISO 4049. The light-polymerization was performed with a LED curing unit in overlapping mode on upper and lower side. All the resin flash was trimmed using #800 SiC paper and stored in 37 °C distilled water for 24 h (static) or at least 48 h (cyclic) before testing. Static flexural strength of specimens was determined with a three-point flexural test according to ISO 4049 ($n = 20$). Additionally, four-point flexural strength was also determined with outer span 20 mm and inner span 10 mm ($n = 20$). Static flexural strength and Young's modulus values were calculated for each group. Flexure fatigue specimens were loaded in cyclic 4-point flexure to failure within distilled water using an experimental electromagnetic cyclic loading machine at a stress ratio and frequency of 0.1 and 5 Hz, respectively. The fatigue strength distributions were evaluated from 100 cycles to 1200 cycles and Basquin-type power law models were developed for the mean fatigue responses ($n = 25 \sim 28$). The fatigue strength distribution for specimen groups was compared over the defined range of cycles using a nonparametric analysis.

Results: Mean three-point flexural strength (FS) and modulus (FM) were ranged 66–161 MPa, 2.53–15.30 GPa and 64–147 MPa, 4.61–23.02 GPa for four-point flexure strength and modulus, respectively. The apparent flexural fatigue limits (FFL) (defined at 1E7 cycles) for groups were ranged from 6.0 MPa Metafill CX)to 27 MPa (Sonicfill), respectively, which were dropped by >80% from their mean static flexure strength. There was a highly positive correlation between FFL and mean three and four-point flexural strength and flexural modulus; $R^2 = 0.66$ (3PFS), 0.85 (3PFM), $R^2 = 0.82$ (4PFS), 0.88 (4PFM), respectively, for the composite resins examined ($n = 8$).

Conclusions: Although the results of fatigue limit strength indicated a decrease over 80% in flexural strength, the cyclic flexural fatigue resistance of the dental composite resins examined could be predictable with their static flexural mechanical properties (flexural strength and modulus).

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Remineralizing and antibacterial composites

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Purpose/Aim: The study aim was to develop light cured composites with added monocalcium phosphate monohydrate (MCPM), tristrontium phosphate (TSrP) and antimicrobial polylysine (PLS). The composites were required to exhibit high monomer conversion, hygroscopic expansion to compensate polymerization shrinkage, promote apatite precipitation and have high antibacterial release whilst maintaining strength.

Materials and methods: All composite formulations consisted of fluid light activated dimethacrylate monomers combined with 80 wt% powder. The powder phase contained a dental glass with and without PLS (2.5 wt%) and/or reactive phosphate (PO) fillers (TSrP (15 wt%) and MCPM (10 wt%)). Monomer conversion and shrinkage, mass and volume changes in water versus simulated body fluid (SBF) (due to water sorption, mineral loss/gain), surface apatite precipitation and PLS release were assessed using FTIR, gravimetric studies, Raman/SEM, and UV spectroscopy respectively. Furthermore, biaxial flexural strengths after 24 h SBF immersion were compared with that obtained for the commercial composite, Z250.

Results: With experimental composites, monomer conversion was higher than that of Z250 (54%) but decreased upon the addition of phosphate fillers (from 76% to 64%). Phosphate addition increased water sorption induced expansion from 2 to 4% balancing the ~3.4% calculated polymerization shrinkage. Phosphate addition also enhanced apatite precipitation in SBF. Gravimetric, Raman and SEM examinations also indicated that PLS increased the apatite layer thickness from ~10 to 20 μm after 4 weeks. The composites showed a burst release of PLS (3.7%) followed by diffusion-controlled release. PLS and PO decreased strength from 154 MPa on average by 17% and 18% but this was still above the ISO 4049 requirement of >80 MPa.

Conclusions: Swelling to compensate shrinkage, with promotion of mineral precipitation and antibacterial release would help prevent bacterial microleakage which is the current main cause of composite tooth restoration failure. These properties would also enable a more minimally invasive approach to tooth restoration.

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PLGA-based nanoparticles for sustained release of Ca^{++} for apexification

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Purpose/Aim: Calcium hydroxide (CH) is a biomaterial widely used to promote apexification. However, the compound releases all its Ca^{++} and OH^- in few weeks, thus the clinician needs multiple appointments to apply new CH to induce an apical closing. Ca^{++} sustained release systems are of great interest to release Ca^{++} for long time with a single application into the root canal. Poly (lactide-co-glycolide) acid (PLGA) based nanoparticles (NP) are proposed to entrap CH and then release the ions for a long time. The aim of this study was to compare the Ca^{++} release profile induced by PLGA-NP loaded with CH and by CH powder.

Materials and methods: PLGA-based NP's loaded with calcium hydroxide were formulated with a spray drying technique. Then, the NP (100 mg) was put into a dialysis membrane that was suspended into a 5 mL tube with deionized water (4 mL); the sample was incubated at 37 °C to promote the degradation of the NP and the release of Ca^{++}. CH (20 mg) was employed as a control; it was also introduced into a dialysis membrane submerged into a 5 mL tube with deionized water. Samples were done by triplicate. The Ca^{++} concentration and pH were measured at several evaluation times (24 hr–42 days) with a pH/ISE Multiparameter Meter (Orion A324). A Kruskal–Wallis test was done to identify possible statistical differences between the Ca^{++} concentration released from the PLGA-NP and the CH.

Results: The PLGA-NP released Ca^{++} for 42 days, while the CH released Ca^{++} for 14 days. At 48 hr, significantly statistical differences were noticed between the Ca^{++} concentration released by the NP and the CH, the differences were observed until the day 14. The Ca^{++} release profile for the NP showed a biphasic behavior. The NP released

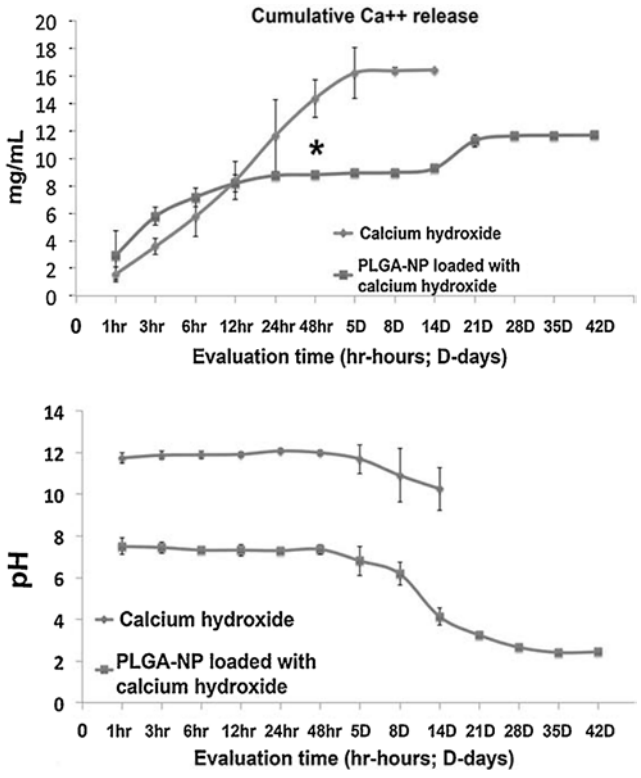

Fig. 1

11.68 mg/ml as a total Ca^{++} concentration, while the HC released 16.42 mg/ml as a total Ca^{++} concentration. The NP induced an initial neutral pH (7.51 pH), and induced an acidic pH at the end of the experiment (2.44 pH). The HC induced an initial alkaline pH (11.76 pH) and induced a 10.26 pH at 14 days.

Conclusions: The PLGA-NP showed a release of Ca^{++} that was longer than the release of Ca^{++}induced by the CH. The Ca^{++} release profile was biphasic for the NP, and they induced an acidic pH.

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Depth of cure and bond strength of bulk-fill composite

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Purpose/Aim: The aim of this in vitro study was to evaluate the depths of cure and bond strengths at different depths of bulk-fill composites.

Materials and methods: Two bulk-fill composites were tested in this study. Depth of cure was measured by ISO 4090 with a stainless steel mode (4 mm diameter). The bulk-fill composites were light-polymerized (40 s at 500 mW/cm^2). The uncured materials were removed with plastic spatula. The height was measured and the result was divided by two to give a depth of cure value ($n = 3$). Shear bond strength was tested on zirconia surface. Yttria-stabilized zirconia ceramics were subjected to air abrading and ultrasonic cleansing, and treated with 1 coat of MDP-containing universal adhesive (All-Bond Universal, Bisco), which was light-polymerized (10 s/500 mW/cm^2). A stainless steel mold (4 mm diameter, 2 or 5 mm height) was placed on the zirconia surface, and then bulk-fill composite was filled up in the mold. The composite was light-polymerized (40 s/500 mW/cm^2). The specimens were then stored in water at 37 °C for 2 h, and tested by Universal Testing Machine (Instron; crosshead-speed 5 mm/min) ($n = 5$). The data were analyzed statistically by two-way ANOVA and Student-t Tests.

Table 1

Composite	Bulk-Fill A	Bulk-Fill B
Bond strength at 2-mm deep	19.7 (1.3) a, 1	22.8 (3.5) a, 1
Bond strength at 5-mm deep	14.7 (3.5) a, 2	19.2 (5.0) a, 1
Bond strength change%	−25%	−16%
Depth of cure (mm)	3.8 (0.0) a	6.1 (0.0) b

Results: Mean shear bond strength and depth of cure (standard deviation) are shown in Table 1. Means within the same row with different letters, or within the same column with different numbers (1,2) are statistically different ($p < 0.05$).

Conclusions: Bulk-fill composite A had depth of cure less than 4 mm, and at 5-mm depth, its bond strength was significantly reduced.

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3-D hygroscopic expansion of resin-composites

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Purpose/Aim: To evaluate the hygroscopic expansion of current resin-composites after storage in distilled water at 37 °C at two time intervals.

Materials and methods: Seven restorative materials were examined: 5 commercial resin-composites (Filtek Supreme XTE, XTE, 3M ESPE; Spectrum TPH3, TPH, Dentsply; Tetric EvoCeram, TEC, Ivoclar Vivadent; Filtek Z250, Z250, 3M ESPE; Admira Fusion, AOR, Voco; and Herculite XRV Ultra, XRV, Kerr), 1 experimental resin-composite (EXP, Voco). Five disks (12 mm × 2.5 mm) of each material were prepared. Baseline measurements were recorded and then specimens were measured after 1-week and 1-month storage in distilled water at 37 ± 1 °C with a custom-built non-contact laser micrometer. Data were re-expressed in volumetric terms and analysed by repeated measures ANOVA, one-way ANOVA and Tukey's post hoc test ($p = 0.05$).

Results: The volumetric hygroscopic expansion ranged from 0.46 to 1.86 after 1 week and from 0.60 to 2.70 after 1 month. For most materials, there was a significant increase after 1 month storage.

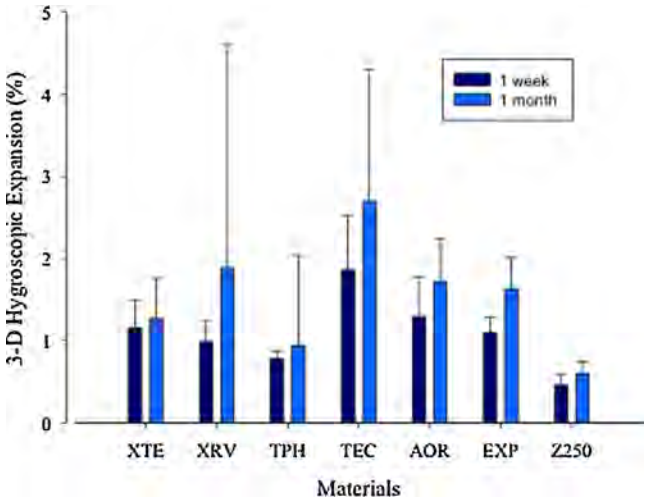

Fig. 1

Conclusions: The composition had a significant effect on hygroscopic expansion. A minimum storage time of 1 month is recommended since most materials kept expanding after 1 week.

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Multispecies biofilm onto plasma-treated titanium surface for dental applications

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Purpose/Aim: In this study, titanium (Ti) was modified with biofunctional and novel surface by micro-arc oxidation (MAO) and glow discharge plasma (GDP) and tested the development of a three-species periodontopatogenic biofilm onto the treated commercially-pure titanium (cpTi) surfaces.

Materials and methods: Several techniques were performed for surface characterizations. A multispecies biofilm composed of *Streptococcus sanguinis*, *Actinomyces naeslundii* and *Fusobacterium nucleatum* was developed onto cpTi discs for 16.5 h (early biofilm) and 64.5 h (mature biofilm). The number of viable microorganisms and the composition of the extracellular matrix (proteins and carbohydrates) were determined. The biofilm organization was analyzed by scanning electron microscopy (SEM) and Confocal laser scanning microscopy (CLSM). MAO treatment produced oxide films rich in calcium and phosphorus with a volcano appearance while GDP treatment produced silicon-based smooth thin-film.

Results: Plasma treatments were able to increase the wettability of cpTi ($p < 05$). An increase of surface roughness ($p < 05$) and formation of anatase and rutile structures were noted after MAO treatment. GDP had the greatest surface free energy ($p < 05$) while maintaining the surface roughness compared to the machined control ($p > 05$). Plasma treatment did not affect the viable microorganisms counts, but the counts of Fusobacterium nucleatum was lower for MAO treatment at early biofilm phase. The extracellular matrix composition was similar among groups, excepted for GDP that had the greatest carbohydrates content.

Conclusions: The findings indicate that plasma treatments is a viable and promising technology to treat bone-integrated dental implants as the new surfaces displayed improved surface properties with no increase in biofilm proliferation.

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Bonding agents improve bond strength of RMGI

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Purpose/Aim: Resin-modified glass ionomer cement (RMGI) is the most popular choice for cementation. However, RMGI normally has low bond strength to ceramics and teeth. The purpose of this study is to investigate whether bonding agents could improve bond strengths of RMGI on zirconia ceramic, lithium disilicate, and dentin.

Materials and methods: Yttria-stabilized zirconia ceramic was sandblasted with alumina sand, rinsed and dried. Lithium disilicate was either polished or etched with 4%-HF etchant for 25 s, rinsed and dried. Human dentin was polished with 320-grit SiC paper, rinsed and blot-dried. Half of the zirconia, lithium disilicate ($LiSi_2$) and dentin were treated with a zirconia primer (ZPrime Plus, Bisco), silane primer (Porcelain Primer, Bisco), and universal adhesive (All-Bond Universal, Bisco), respectively. The other half of specimens was not treated with any bonding agents (as control groups). Shear bond strength was tested using the notched-edge shear bond strength test method (ISO 29022:2013). RMGI (RelyX Luting Plus, 3 M Oral) was placed on the substrate surface,

Table 1

Substrate	Dentin	Zirconia	$LiSi_2$ (etched)	$LiSi_2$ (polished)
No primer/ adhesive	6.9 (1.4) a	7.7 (1.7) a	9.4 (0.7) a	0.3 (0.4) a
With primer/ adhesive	11.1 (0.6) b All-Bond U	12.6 (1.1) b ZPrime Plus	11.5 (0.6) b Porcelain P	4.0 (1.7) b Porcelain P
Bond increase	61%	64%	22%	1233%

light-polymerized (40 s@500 mW/cm^2), and continued to self-polymerized in 37 °C-oven for 15 min. The specimens were then stored in water at 37 °C for 24 h, and tested by universal testing machine (Instron, crosshead-speed 1 mm/min). The data were analyzed statistically by one-way ANOVA and Student-*t* test.

Results: Mean shear bond strengths in MPa (standard deviation) are shown in Table 1. Means with different letters in the same column are statistically different ($n = 5$, $p < 0.05$).

Conclusions: Bond strengths of resin-modified glass ionomer cement on dentin, zirconia, and lithium disilicate were improved by universal adhesive (All-Bond Universal), zirconia primer (ZPrime Plus), and silane Primer (Porcelain Primer).

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Effect of cavity design on gap formation observed by OCT

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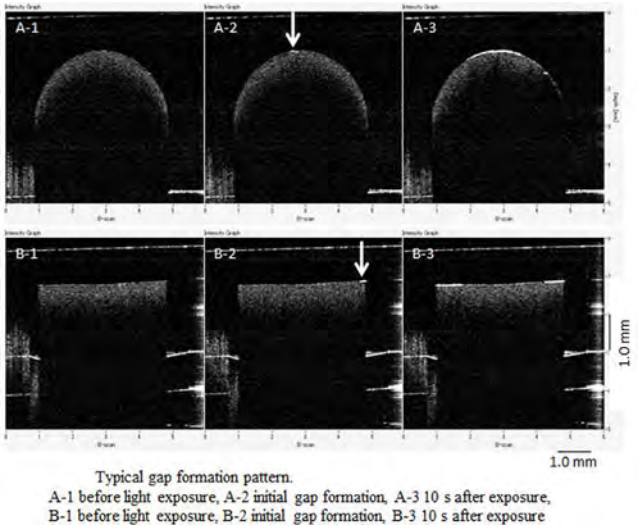
Purpose/Aim: The patterns of gap formation at composite resin polymerization are not clearly elucidated. The purpose of the present study was to evaluate the effects of cavity design on gap formation of composite resins observed by a dental swept-source-optical coherence tomography (OCT).

Materials and methods: Four different cavity-designs were used; a semi-sphere and cylindrical cavity with a diameter of 4.2 mm and a depth of 2 mm and 4 mm was prepared in a 3-mm thickness and 5-mm thickness PMMA plate, respectively. Four composite resins, three bulk fill flowable composites (Bulk fill Medium flow, Sun Medical; SDR, Dentply; Beauty fil Bulk flow, Shofu) and one conventional flowable composites (MI flow, GC) were examined. Paste of the composite resin was placed into the cavity and polymerized with an LED curing light for 20 s. Marginal gap formation was observed from the cavity bottom side using a dental swept-source-OCT. The cross-sectional image at the center of the cavity was acquired at 0.1 s interval until 20 s after the start of light exposure. The time and location at the initial gap formation observed and the time at complete gap formation were recorded. When the gap formation was not detected in 20-s observation, the time of gap formation was considered at 20 s. At least 5 measurements of each condition were performed. The times at initial and complete gap formation were analyzed with 2-way ANOVA selecting cavity designs and composite resin brands as main factors ($\alpha = 0.05$).

Results: Fig. 1 shows two typical gap formation patterns that the gaps were initiated at the cavity margin and the center of cavity bottom. The initial gap formation of the 2-mm cavity except SDR occurred from the cavity margin, but that of the 4-mm cavity sometimes occurred at the center of the cavity bottom. The gap formation of the 2-mm cavity of SDR could not be detected within 20 s after the start of light exposure, but those of the others detected within 6 s. The complete gap formation of the 2-mm cavity of SDR and Beauty fil were later than 18 s, but those of 2-mm cylindrical cavity of all composites were less than 12 s. Two-way ANOVA of initial and complete gap formation time revealed that two main factors and their interactions were significant.



Typical gap formation pattern.
A-1 before light exposure, A-2 initial gap formation, A-3 10 s after exposure,
B-1 before light exposure, B-2 initial gap formation, B-3 10 s after exposure

Fig. 1

Conclusions: These results suggest that gap formation patterns are influenced by not only cavity design but also composite resins.

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Study of the oxygen-inhibited layer of three universal dental adhesives

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Purpose/Aim: This study compares the oxygen-inhibition effect on the physiochemical properties of three universal dental adhesives: Prime & Bond Elect (PBE) [Dentsply], iBond Universal (IBU) [Kulzer], and Scotchbond Universal (SU) [3 M]. The physicochemical properties studied were degree of conversion (DC), and the chemical composition of the outermost layers.

Materials and methods: The effect of the oxygen on the DC for each sample was determined by using FTIR [Thermo Scientific Nicolet^{TM} 6700, $n = 4$. with two experimental conditions: using a Mylar strip (S) before polymerization and without Mylar strip (NS). For NS, a drop was placed on a glass slab side and the solvent was allowed to evaporate for 10 s before curing the sample with a LED (Valo Cordless, 1000 mW/cm^2) for 20 s. For S: the same procedure was used for samples with a Mylar strip, to minimize the oxygen-inhibiting layer formation. The carbonyl peaks at 1720 cm^{-1} and the aliphatic double carbon bond at 1640 cm^{-1} were used to calculate the DC for Scotchbond, while the peaks at 1610 cm^{-1} and the peak at 1640 cm^{-1} were used for iBond and Elect. The chemical analyses of the

effect of the oxygen on adhesive surface were performed using the X-Ray Photoelectron Spectroscopy (XPS). The samples ($n = 3$) were analyzed by XPS (Kratos Axis, vacuum of $2 \times 10\text{-}9$ torr). XPS surveys were taken from the surface (depth = 0 nm, (D0)) and sub-surface layers (depth = 20 nm (D20)) and 70 nm (D70) after removing the external surface with Argon etching. The obtained mean data was subjected to 2-way ANOVA and Tukey's post hoc test at 5% significance level.

Results: DC results displayed a discrepancy for the groups S and NS. Oxygen-inhibition effect reduced the DC of all of adhesives: PBE (- 19.2%), IBU (-22.9%) and SU (−15.4%).

XPS survey: Table below displays the carbon and oxygen percentages, which were found at D0, D20 and D70 for all adhesives. All adhesives displayed carbon impurities for C1s peak at 285 eV at D0, but not in the other layers.

Conclusions: Oxygen-inhibited layer was different quantitatively and qualitatively depending of the adhesive chemical composition.

Adhesives	Element	Atomic percentage according to the depth		
		0 nm	20 nm	70 nm
PBE	Carbon	71.55	95.44	95.14
	Oxygen	23.91	1.69	1.53
IBU	Carbon	87.20	94.49	95.54
	Oxygen	11.86	3.67	3.24
SU	Carbon	74.52	77.99	77.83
	Oxygen	18.60	11.59	10.34

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Engineered peptide for adhesive/dentin interfacial integrity

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Purpose/Aim: Recurrent decay is the primary reason for failure of composite restorations and 80–90% of recurrent decay is located at the gingival margin. At this margin, the adhesive and its bond with dentin is the barrier between the restored tooth and the oral environment. In vivo degradation of the bond formed at the adhesive/dentin (a/d) interface follows a cascade of events that ultimately lead to an undermining of the composite restoration. The objective of this work was to mineralize deficient dentin matrices at the a/d interface using engineered peptides; the engineered peptides were conjugated to fluorescence protein for ease of monitoring.

Materials and methods: A/D interface specimens, prepared according to published protocols, were analyzed using micro-Raman spectroscopy (μRS) to determine the distribution of adhesive, collagen and mineral. Following initial μRS analysis, the exposed a/d interface was treated for 20 s with 5% NaOCl followed by 5 N HCl for 30 min. Anchoring peptides that are designed with an inherent capacity to self-assemble and induce selective binding on the defective dentin matrices, i.e. hydroxyapatite binding peptide (HABP) were absorbed on the a/d interface. The peptide was coupled to biomarker protein, GFP, to track its selective binding. Remineralization was initiated by adding alkaline phosphatase at a final concentration of 1.4×10^{-6} g/mL, samples were incubated at 37 °C for 24 h, and examined using fluorescent microscopy (FM) and μRS.

Results: Based on the absence of spectral features associated the mineral (PO_4^{-2} at 960 cm^{-1}), the μRS images indicated ~20 μm demineralization at the NaOCL/HCl-treated a/d interface. Following functionalization of the a/d interface with HABP, the intensity of the P-O group suggests distribution of mineral throughout the interface. Complementary FM analysis revealed mineralization of the GFP-HABP-treated a/d interface.

Conclusions: Comparison of the μRS images demonstrates remineralization of the exposed collagen layer (deficient dentin matrices) at the a/d interface following functionalization with HABP. HABP incorporation to the demineralized dentin surface produced extensive integrated remineralization of the a/d interface.

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Simulated gap wear of resin luting cements

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Purpose/Aim: One of the primary areas of concerns with luting agents is marginal gap erosion and attrition. The purpose of this laboratory study was to use a new model to simulate marginal gap wear and examine the loss of material during simulated generalized wear.

Materials and methods: Five resin luting cements were used in this study: (1) G-CEM LinkForce (GC, Tokyo, Japan), (2) RelyX Ultimate (3M ESPE, USA), (3) Multilink Automix (Ivoclar Vivadent), (4) Panavia V5 (Kuraray Noritake Dental, Tokyo, Japan) and (5) NX3 (Dentsply, USA). A two-piece stainless steel custom fixture was designed with a gap 300 μm wide and 4 mm in length. Twenty specimens for each of the adhesive cements were made with both light cure and chemical cure techniques. The cured cements were polished with a series a carbide papers to a 4000 grit surface and subjected to 100,000 cycles in a Leinfelder-Suzuki (Alabama) wear simulator (maximum load of 80 N) using a stainless steel flat-ended antagonist in a water slurry of PMMA beads for simulation of generalized contact free area (CFA) wear. Before and after the wear challenges, the specimens were profiled with a Proscan 2100 non-contact

Table 1 – Simulated gap wear of resin luting cements.

Resin luting cement	Volume loss (mm^3)		Facet depth (μm)	
	Light cure	Chemical cure	Light cure	Chemical cure
LF	0.023 (0.007)^{a,A}	0.028 (0.006)^{a,A}	35.4 (9.2)^{a,A}	44.4 (11.7)^{a,A}
RU	0. 030 (0.007)^{b,A}	0.044 (0.006)^{b,B}	45.2 (9.6)^{b,A}	69.5 (10.4)^{b,B}
MA	0.026 (0.010)^{a,A}	0.039 (0.008)^{b,B}	38.2 (13.0)^{a,b,A}	62.7 (11.9)^{c,B}
PV	0. 029 (0.014)^{a,A}	0.033 (0.008)^{a,b,A}	42.9 (7.2)^{a,b,A}	52.3 (9.9)^{a,c,A}
NX	0.028 (0.007)^{a,A}	0.042 (0.009)^{b,B}	40.4 (8.3)^{a,b,A}	64.3 (9.7)^{b,B}

Values in parenthesis are standard deviations. Same small letter in same vertical column indicates no significant difference ($p > 0.05$). Same capital letter within individual rows indicates no significant difference ($p > 0.05$).

profilometer and wear (volume loss and mean gap depth) was determined using AnSur 3D software. A two-way ANOVA and Tukey's post hoc test were used for data analysis.

Results: The two-way ANOVA showed a significant effect among the five resin cement materials for the factors of resin cement ($p < 0.001$) and cure method ($p < 0.001$) and for the interaction of resin cement and cure method ($p < 0.001$). Table 1 shows the volume loss (mm3) and mean gap depth (μm) after wear simulation and the significant differences ($p < 0.05$) among the cements and cure methods evaluated.

Conclusions: A new gap model for evaluation of simulated generalized wear of resin luting cements demonstrated significant differences ($p < 0.05$) in wear among five resin luting cements and between visible light and chemical cure techniques.

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Selective etching in NCCL for self-etch adhesives: SR and meta-analysis

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Purpose/Aim: To identify if selective etching of enamel margins improves the retention rates and marginal discoloration of cervical composite restorations in non-carious cervical lesions (NCCLs) of adult patients performed by systematic review and meta-analysis.

Materials and methods: A comprehensive search was performed in the MEDLINE via PubMeb, Scopus, Web of Science, LILACS, BBO and Cochrane Library and SIGLE without restrictions. The abstracts of the annual conference of the IADR, unpublished and ongoing trials registry were also searched. Dissertations and theses were searched using the ProQuest Dissertations and Periodicos Capes Theses databases. We included randomized clinical trials that compared the clinical effectiveness of selective enamel etching using self-etch adhesive for direct composite resin restorations in NCCLs in permanent dentition of adult patients. The risk/intensity of POS was the primary outcome. The risk of bias tool of the Cochrane Collaboration was used. The meta-analysis was performed on the studies considered 'low' risk of bias.

Results: After duplicates removal, 2689 articles were identified. After abstract screening, 11 studies remained in the qualitative synthesis. Seven were considered to be 'low' risk of bias. The report of the studies varied to 1–5 years. Except for one-year follow-up, there was a significant lower marginal discoloration and marginal adaptation during all follow-up times. Also, it was observed a significant lower loss of retention to the restorations at the 3-year follow up for selective enamel etching.

Conclusions: Selective enamel prior to application of self-etch adhesive systems in NCCLs might improve the clinical performance of resin composite cervical restorations.

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Bond strength and sealing ability between fiber posts to dentin

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Purpose/Aim: To evaluate the push-out bond strength and sealing ability of glass-fiber posts in different regions of post space (cervical, middle, and apical) submitted to different conditioning agents.

Materials and methods: Ninety extracted single rooted human teeth were used in this study. After endodontic filling, teeth were divided into five groups ($n = 18$), according to the conditioning agents: distilled water, 5.25% sodium hypochlorite, 25% polyacrylic acid, 2% chlorhexidine and 23 ppm silver nanoparticles dispersion. The groups were divided into 3 subgroups ($n = 6$) according to the materials used for adhesive cementation: SBU group: Scotchbond Universal adhesive system + RelyX ARC resin cement; U200 groups: RelyX U200 self-adhesive resin cement; MCE group: MaxCem Elite self-adhesive resin cement. The bond strength was measured using the push-out test and the sealing ability was measured using Flodec in different areas of the post space (cervical, middle, and apical). The data were subjected to statistical tests of normality and analyzed by ANOVA and Fisher's test ($\alpha = 0.05$). Images of representative sample were obtained in an scanning electron microscopy.

Results: The silver nanoparticle solution showed the highest bond strength values in all thirds analyzed for the SBU group. In the U200 group, the highest values were found for sodium hypochlorite solution, with difference to the polyacrylic acid. The MCE group generally showed lower bond

strength values. There was a decrease in push-out bond strength in the cervico-apical direction.

Conclusions: The different conditioning solutions and the intraradicular depth influenced the bond strength of adhesives materials to dentin substrate. The silver nanoparticle can be used as an irrigant agent in the post space previously to glass fiber post cementation process, since there is an antibacterial effect, and it does not cause interference in the bonding process between fiber post and intrarradicular dentin.

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Monomer conversion and mechanical properties of contemporary bulk-fill composites

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Purpose/Aim: The objective was to evaluate the degree of conversion and selected mechanical properties of bulk-fill composites (BFCs) as a function of the composition and the processing method (with or without additional thermal treatment, TT).

Materials and methods: The following composites were tested: four low-viscosity BFCs, Filtek Bulk Fill Flowable (3M ESPE) [FBFF], Fill-Up! (Coltene Whaledent) [FU], Surefil SDR (Dentsply Caulk) [SDR], Venus Bulk Fill (Heraeus Kulzer) [VBF], three high-viscosity BFCs, everX posterior (GC) [EXP], SonicFill (Kerr) [SF], Tetric EvoCeram Bulk Fill (Ivoclar Vivadent) [TECBF], and one high-viscosity conventional composite, Charisma Diamond (Heraeus Kulzer) [CD]. Specimens were light-cured (20 J) and degree of conversion (DC, via Fourier-transformed near-infrared spectroscopy, $n=3$) was determined for composite discs (⊖8 mm × 1 mm-thick). Knoop hardness (KHN, $n=3$) was evaluated at top and bottom surfaces of blocks (2.5 × 2.5 × 4 mm), flexural strength (FS, three-point-bending, $n=10$) and fracture toughness (K_{IC}, single-edge-notched-beam, $n=10$) were measured in bars (10 × 2 × 1 mm). All tests were performed in two conditions: with or without additional TT (170 °C/10 min). Data were submitted to ANOVA and Tukey's test (alpha = 0.05) or Kruskal–Wallis test ($p=0.05$).

Results: Results are shown in Table 1. BFCs showed similar or higher DC than that of the conventional composite. Except for CD, all materials were sufficiently polymerized at depth of 4 mm, as confirmed by visual inspection of the bottom surfaces of all specimens. All BFCs obtained lower top KHN compared to CD, except for SF, which showed similar top KHN. EXP, FU, SDR and VBF showed similar KHN for top and bottom surfaces. In comparison to CD, high-viscosity BFCs showed similar or better FS; and EXP showed better KIC. TT improved all tested properties for most tested materials.

Conclusions: The fact that all tested composites (conventional and bulk-fill) had their DC and mechanical properties improved by TT indicates that the technological innovation related to the development of BFCs does not represent an improvement in terms of polymerization behavior.

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Table 1 – Mean and standard deviation for degree of conversion (%), top and bottom Knoop hardness (KHN), flexural strength (MPa) and fracture toughness (MPa.mm0.5) before (only light-cured) and after thermal treatment (TT). Different letters indicates significant differences within the test.

		Before TT	After TT	Before TT		After TT		Before TT	After TT	Before TT	After TT
				Top	Bottom	Top	Bottom				
Conventional composite	CD	54.5 (1.9) H	87.4 (3.1) C,D	85.63 (2.0) B	–	116.47 (3.06) A	–	99.15 (8.59) E,H	189.85 (20.22) A	2.01 (0.14) E,F,G	2.91 (0.06) A,B,C
Low-viscosity bulk-fill composite	FBFF	56.6 (1.9) G,H	88.3 (3.1) B,C,D	27.18 (1.14) N	18.51 (1.6) O	48.52 (0.82) L	37.15 (5.51) M	60.85 (4.76) I	132.96 (16.41) B,C	1.37 (0.09) I	2.62 (0.14) B,C,D,E
	FU	60.0 (4.3) F,G,H	88.0 (6.9) C,D	53.06 (2.03) J,KL	51.53 (0.6) K,L	63.27 (1.76) H	63.15 (1.79) H	83.86 (12.74) F,G,H,I	120.95 (21.31) C,D,E	1.54 (0.09) H,I	1.74 (0.11) F,G,H
	SDR	62.8 (0.6) F,G	98.3 (1.0) A	36.2 (0.51) M	35.56 (0.67) M	53.5 (0.91) I,J,K	55.4 (1.1) I,J,K	65.18 (5.09) H,I	144.47 (13.92) A,B,C	1.74 (0.04) F,G,H	2.86 (0.09) A,B,C
	VBF	66.5 (0.9) F	94.0 (0.8) A,B,C	25.06 (0.99) N	26.38 (1.93) N	39.27 (3.99) M	39.75 (2.38) M	59.90 (5.06) I	155.17 (12.54) A,B,C	1.59 (0.06) G,H,I	2.92 (0.10) A,B,C
High-viscosity bulk-fill composite	EXP	66.3 (0.2) F	88.7 (0.9) B,C,D	63.51 (1.31) G,H	59.5 (0.93) H,I,J	78.09 (1.58) C,D,E	76.22 (0.72) D,E,F	122.54 (18.33) C,D,E	138.29 (10.04) A,B,C	3.35 (0.52) A,B	3.84 (0.52) A
	SF	75.7 (0.7) E	95.8 (0.8) A,B	81.65 (1.33) B,C,D	53.04 (0.26) J,K,L	83.08 (0.91) B,C	74.91 (1.1) E,F	101.09 (14.38) D,E,F,G,H	161.10 (24.77) A,B	2.42 (0.14) C,D,E,F	2.93 (0.17) A,B,C
	TECBF	56.6 (2.1) G,H	83.4 (0.4) D,E	53.74 (0.63) K,L	36.23 (3.83) M	69.95 (2.07) F,G	61.13 (3.29) H,I	82.71 (6.68) G,H,I	116.22 (11.24) C,D,E,F	1.57 (0.07) H,I	2.13 (0.12) D,E,F

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Effect of cranberry and proanthocyanidin in dentin erosion prevention

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Purpose/Aim: The literature recognizes that one of the most effective strategies in minimizing the dentin tissue loss by erosive agents is the maintenance of its organic matrix (DOM), because it acts as a diffusion barrier to acid. However, the collagen tends to be degraded and it is important the search for agents that can inhibit this process. There are evidences that proanthocyanidin-rich agents can inhibit matrix metalloproteinases (MMPs) which degrade collagen, but these agents have not been adequately evaluated in dental erosion. Thus, the aim of this study was to evaluate the effect of Cranberry and Proanthocyanidin in inhibiting wear and demineralized organic matrix (DOM) degradation.

Materials and methods: For this, 120 bovine dentin blocks (4 × 4 mm) were randomized into 3 groups: 40 treated with 10% purified proanthocyanidin gel, 40 with 10% Cranberry extract gel (proanthocyanidin-rich agent) and 40 with placebo gel (no active agent). Before the treatments, samples were demineralized by immersion in 0.87 M citric acid, pH 2.3 (36 h at 4 °C) to expose the DOM. Then, the studied gels were applied once over dentin for 1 minute. Next, the samples were immersed in artificial saliva containing collagenase obtained from Clostridium histolyticum for 5 days (37 °C). The response variable was depth of dentin loss (μm) measured by profilometry. Thus, 3 analysis were performed: initial (profile of sound specimens), after demineralization (for quantification of initial wear) and after-immersion in saliva with collagenase (to assess the overall wear and DOM degradation). Data were analyzed by ANOVA followed by Tukey's test ($p < 0.05$).

Results: Results showed that the purified proanthocyanidin gel had the best ability to reduce the DOM degradation (69.0 ± 34.8a), followed by Cranberry extract gel (120.2 ± 44.8b) and Placebo gel (161.8 ± 60.1c).

Conclusions: Cranberry extract was able to reduce the dentin wear and collagen degradation, but the purified proanthocyanidin obtained the best results, confirming its effectiveness in minimizing the negative effects of dentin erosion.

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Influence of biosilicate incorporation on the mechanical properties of GIC

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Purpose/Aim: It is known that the addition of components into glass ionomer cements can change its mechanical properties, especially the resin-modified ones that have resin matrix in their composition. However, bioactive glasses may help in the remineralization of the dentin underlying the decay. Therefore, the aim of this study was to evaluate the influence of the incorporation of different proportions (0: control; 2% and 6.5% wt%) of functionalized Biosilicate® in the flexural (FS) and compressive (CS) strengths of resin-modified glass ionomer cements (RMGIC).

Materials and methods: For the FS test, specimens were obtained at bar shape (2 × 2 × 25 mm), while for the CS test they were obtained at a cylindrical shape (ø 4 × 6 mm), either for the control groups (Vitrebond® e Vitremer® – 3M ESPE – without Biosilicate®) or for the ones modified by the incorporation of micrometric silanized particles of bioglass. The samples were subjected to the tests in a mechanical testing machine (EMIC) with a speed of 0.5 mm/min.

Results: The means analysis (1-way ANOVA, Tukey, $p < .05$) revealed that the groups with Biosilicate®, silanized or not, showed lower ($p < .05$) FS and CS values, for all groups.

Conclusions: We conclude that the functionalization of the Biosilicate® particle was not enough to maintain the mechanical properties of the tested resin-modified glass ionomer cements.

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Flexural strength and contrast ratio comparison for translucent zirconia

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Purpose/Aim: The demand for natural translucent monolithic restoration induced manufacturer to develop polycrystalline ceramics with increased translucency. Aim of this study was to compare three different compositions of Partially Stabilized Zirconia (PSZ) with a reinforced glass ceramic as the Lithium Disilicate in term of flexural resistance and translucency.

Materials and methods: Three pre-sintered zirconia disks (GC Tech, Leuven, Belgium) with different composition and reported translucency were tested. Aadva ST (Standard

Translucency – ST), Aadva EI (Enamel Intensive – EI) and Aadva NT (Natural Translucent - NT) were cut by a slow speed diamond saw in bars and tabs shapes in order to obtain after sintering the dimensions of 1.2 × 4.0 × 13.0 mm and 15.0 × 15.0 × 1.0 mm for bars and tabs respectively. Blocks of IPS e.max CAD LT (Ivoclar Vivadent AG, Schaan, Liechtenstein – LD) were cut and crystallized in the same shapes and dimensions. Bars ($n = 15$) were finished and polished until the desired dimensions were reached (according with ISO 6872:2015) and then submitted to a three-point bending test (span = 13 mm, speed = 1 mm/min) in a universal testing machine. Critical fracture load was recorded in N; flexural strength (σ in MPa), Weibull modulus (m) and Weibull characteristic strength ($\sigma 0$ in MPa) were then calculated. Tabs ($n = 10$ per group) were polished and measured with a spectrophotometer (OceanOptics PSD1000) equipped with an integrating sphere. For translucency evaluation, contrast ratios (CR) were calculated as Yb/Yw. Significance for variables Translucency and Flexural strength were tested by separate One-Way ANOVA, followed by the Tukey test for post-hoc comparison.

Results: Data and statistical significance were summarized in Table 1. Differences in translucency result to be statistically

Table 1

Translucency - CR		Flexural Strength - 3PBT		
Groups	Mean (SD)	Mean σ (MPa) (SD)	m	σ^0 (MPa)
Aadva ST	0.74 (0.01)^{d}	1215 (190)	7.45	1291^{a}
Aadva EI	0.69 (0.01)^{c}	983 (182)	6.54	1056^{b}
Aadva NT	0.65 (0.01)^{b}	539 (66)	9.68	566^{c}
IPS e.max LT	0.56 (0.02)^{a}	377 (39)	11.27	394^{d}

significant. CR increased on the following order (lower CR) LD > NT > EI > ST (higher CR). The flexural strength statistically differed between groups. The flexural strength decreased on the following order: (higher strength) ST > EI > NT > LD (lower strength).

Conclusions: The statistically significant increase in translucency resulted in a statistically significant decrease of mechanical proprieties. According to ISO 6872:2015 the clinical indications for Zirconia Aadva NT differs from the other zirconia compositions and should be limited up to three-unit span bridges in molar region.

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Bond strength of composite resin to nickel-chrome alloy using primers

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Purpose/Aim: To evaluate the effects of three adhesive primers and one multi-mode adhesive system on the shear bond strength (SBS) of a flowable composite to nickel-chrome alloy (Ni-Cr).

Materials and methods: Ninety rectangular plates were cast from Ni-Cr and randomly divided into nine groups ($n = 10$). The surfaces were sandblasting with Al_2O_3 (50 μm) and

Table 1 – Shear bond strength (MPa) and standard deviation for the experimental groups at 24 hours or after thermocycling.

Groups	24 h	Thermocycling
Control	11.4 ± 1.5 A d	6.9 ± 2.2 A c
AP	19.6 ± 1.7 A bc	11.4 ± 4.3 B c
AP + SB	23.2 ± 1.7 A ab	18.8 ± 3.5 A b
AP + SU	22.9 ± 0.9 A ab	20.1 ± 4.5 A b
UP + SB	22.0 ± 1.4 A ab	19.2 ± 2.3 A b
UP + SU	25.4 ± 1.9 A a	26.0 ± 3.5 A a
CP + SB	12.6 ± 1.3 A d	7.8 ± 1.2 A c
CP + SU	12.3 ± 1.7 A d	6.7 ± 1.4 B c
SU	15.7 ± 0.7 A cd	7.0 ± 1.5 B c

Upper case letters compare specimens tested after 24 hours to thermocycled ones (row) and small case letters compare groups (column).

primed with three metal primers: Alloy Primer (AP), Universal Primer (TP) and RelyX Ceramic Primer (CP), and a multi-mode adhesive (Scotchbond Universal, SU). The Adper Single Bond Plus (SB) and SU adhesives were also combined with metal primers. Control group did not receive any surface treatment. The experimental groups were: AP, AP + SB, AP + SU, TP + SB, TP + SU, CP + SB, CP + SU and SU. Flowable composite cylinders (1 mm high, 1 mm diameter) were built in each plate. After 24 hours, half of the specimens were subjected to SBS and another half were subjected to 5000 thermal cycles before testing. Data were analyzed by two-way ANOVA and Tukey's test ($\alpha = 0.05$). Failure modes were determined by SEM observation.

Results: Higher SBS were obtained with AP and TP combined with adhesives at 24 hours and lowest one for the control group. The thermocycling reduced the SBS for AP, CP + SU and SU. Combination between TP and SU resulted in the highest SBS following the thermocycling. TP groups showed all types of failures and high incidence of mixed failures. Table 1

Conclusions: Results suggested that the combination of a sulfur-containing monomer and phosphate or carboxylic acid functional monomer increased the SBS of flowable composite to Ni-Cr. However, the mixture of three types of monomers showed the highest bond strength after thermocycling.

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Are novel universal adhesives more resilient to salivary contamination?

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Purpose/Aim: To evaluate the influence of salivary contamination and decontamination procedures, during different stages of the restoration on a new universal adhesive system (U), in comparison with a clinically established two step self-etch (SE) adhesive.

Materials and methods: Extracted carious free 3rd molars were sectioned using a low speed saw into 4 or 8 parts (depending on the size of the tooth). The obtained 280 dentin surfaces

were embedded in cold cure acrylic and wet ground with 600grit SiC paper. They were then randomly allocated into 14 groups ($n = 20$) for 2 adhesives; Clearfil SE Bond 2 (SE) and Clearfil Universal (U)(Kuraray). Two control groups with no contamination (NC) for both the adhesive system were prepared. The contamination (C) and decontamination (DC) were implemented at 3 Stages; Stage-1: after surface preparation, Stage-2: after primer application (only for SE) and Stage-3: after adhesive system curing. The area to be tested was delimited using an adhesive sheet with 3.5 mm diameter hole. The dentin surfaces were then contaminated at aforementioned stages with 0.025 ml natural human saliva for 10 s, which was then air-dried (C1-SE, C2-SE, C3-SE, C1-U and C3-U). The decontamination groups involved the aforesaid contamination, followed by either rinsing with water and air-drying (DC1-SE, DC3a-SE, DC1-U and DC3a-U) or by rinsing with water, air-drying and reapplication of adhesive system (DC2-SE, DC3b-SE and DC3b-U). A low shrinkage, bulk-fill resin composite (Admira Fusion X-tra, VOCO) was used as restorative material. Specimens were stored in distilled water at 37 °C for 7 days and then subjected to shear bond strength test at a crosshead speed of 0.5 mm/min.

Results: Data were statistically analyzed using one and multi-way analyses of variance and independent-samples *t*-test ($\alpha = 0.05$). Contamination significantly reduced the bond strength for U, while none of the decontamination procedure could restore the bond strength to control levels (NC-U). SE showed statistically significant reduction in bond strength only when the contamination took place after application of primer (C3-SE), however the decontamination (DC3-SE) improved the bond strength considerably. The multivariate analysis (general linear model with partial-eta-squared statistics) revealed the type of adhesive had a higher influence on bond strength (partial-eta-square= 0.287, p <0.0001) compared to the influence of the contamination and decontamination procedures (partial-eta-square =0.063, $p < 0.0001$). The fracture pattern was predominantly adhesive (71.8%) or mixed (28.2%), and no cohesive or pre-failure was registered.

Conclusions: Universal adhesives may not be as tolerant to contamination as compared to the two step self-etch adhesive.

Stages of influence		Shear bond strength (MPa)		*p*-value
		Self-Etch (SE)	Universal (U)	
No influence	Control (NC)	18.53(5.26)b	16.55(3.91)B	0.186
Stage-1	Contamination (C1)	16.58(3.28)b	9.63(3.84)A	<0.0001
	Decontamination (DC1)	16.99(4.06)b	9.99(4.38)A	<0.0001
Stage-2	Contamination (C2)	12.72(5.27)a	–	–
	Decontamination (DC2)	17.50(3.50)b	–	–
Stage-3	Contamination (C3)	17.43(3.31)b	10.56(3.94)A	<0.0001
	Decontamination (DC3a)	17.85(2.77)b	10.67(4.4)A	<0.0001
	Decontamination (DC3b)	18.74(3.68)b	10.55(4.51)A	<0.0001

a and A: statistically significant difference ($p < 0.05$).

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Fabrication of gradient scaffolds for bone and dental tissue engineering

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Purpose/Aim: The aim of this study was to develop a new protocol to control the pore size and porosity of 3D scaffolds fabricated through freeze-drying technique for bone and dental tissue engineering applications. We intended to produce gradient scaffolds using this method as the best representative model for such a control.

Materials and methods: Gelatin was employed as the main component of the matrix in our formulation while hydroxyapatite (HA) and/or tri-calcium phosphate (TCP) were used as the filler to improve mechanical properties and osteo-conductivity. Scaffolds were prepared by foaming different gelatin/HA/TCP suspensions through mechanical stirrer at different speeds in the range of 0–2000 rpm. The foams were then freeze-dried (−54 °C, 1 Pa) with or without lyoprotectant and cross-linked using 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide/N-Hydroxysuccinimide. Gradient scaffolds were produced through layer-by-layer assembly of the foams prior to freeze-drying. Scanning Electron Microscopy (SEM) was used to study the morphology of scaffolds and pore size distribution. Mechanical properties of the scaffolds were evaluated in compression mode using a Universal Testing Machine (UTM).

Results: The effect of gelatin concentration, stirring speed and presence of lyoprotectants on the final pore size and porosity were investigated. The scaffolds prepared using our method represent a porous interconnected structure that is highly reproducible compared to conventional freeze-drying technique. Our results indicate that increasing the gelatin concentration and stirring speed, the pore size and porosity decreases. Addition of lyoprotectant also decreases the pore size and porosity. Gradient scaffolds were fabricated through

layer by layer assembly of three foams with various pores (i.e. <100 μm, 100–300 μm and >300 μm). SEM images confirmed the pore size and porosity gradient. Compressive modulus of the scaffolds that is in the range of spongy bone varies by the porosity change.

Conclusions: The method developed in this study has the potential to be used to produce gradient scaffolds as well as scaffolds with controlled pore size for bone and dental tissue engineering applications.

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Polymerization properties assessment of bulk fill resin composites

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Purpose/Aim: The objectives of the present study were to assess the depth of cure, the volumetric shrinkage as well as the shrinkage force of different resin composites: Filtek Z350XT (Z350 – control group, 3M ESPE), Surefill SDR (SDR, Dentsply), Xtra Fil (XF, Voco), Admira Fusion Xtra (ADM, Voco), Tetric N Ceram Bulk Fill (TBF, Ivoclar Vivadent) and Filtek Bulk Fill Posterior (FBP, 3M ESPE).

Materials and methods: For depth of cure, longitudinal Knoop microhardness was performed in the specimens ($n = 8$) right after 20 s light curing, using a 50 g weight during 20 s. Values corresponding to at least 80% of the surface microhardness were considered as adequate cured. For volumetric shrinkage, seven specimens of resin composite ($4 \times 4 \times 4$ mm) were scanned in micro-CT (Skyscan, Bruker) before and right after 20 s light curing, the images were processed and analyzed for changes in dimensions. For shrinkage force assessment, the polymerization stress was measured through the deformation of a load cell adapted in an Universal testing machine, up to 300 s after polymerization of the resin composites with 12 mm^3 ($n = 7$). Data was evaluated through one-way ANOVA (volumetric shrinkage and depth of cure), two-way ANOVA (shrinkage force) and Tukey's test, all with $p < 0.05$.

Results: The results showed higher values of depth of cure for SDR (6.93 ± 0.41), followed by ADM (5.43 ± 0.62), XF (5.37 ± 0.69), FBP (5 ± 0.46) and TC (4.87 ± 0.44), with the control Z350 (2.62 ± 0.23) showing the lowest depth of cure. Considering the polymerization shrinkage, lower values were observed for XF (0.84 ± 0.36), ADM (1.24 ± 0.18), TC (1.75 ± 0.12) and FBP (2.19 ± 0.47), with higher dimensional changes for SDR (3.36 ± 0.62) and Z350 (3.07 ± 0.61). Regarding polymerization stress (after 300 s), lower values were observed for TC (3.94 ± 0.4), XF (4.61 ± 0.34) and SDR (4.63 ± 0.25), with the highest stresses generated by Z350 (5.67 ± 0.21), FBP (5.2 ± 0.42) and ADM (5.11 ± 0.31).

Conclusions: It can be concluded that all bulk fill resins are capable of polymerization in large increments (at least 4.5 mm). In addition, they are capable of generating lower shrinkage forces and polymerization shrinkage, but it depends on their composition and polymerization kinetics.

Group	Z350	FBP	TC	ADM	XF	SDR
Depth of cure (mm)	2.62 (0.23)	5 (0.46)	4.87 (0.44)	5.43 (0.62)	5.37 (0.69)	6.93 (0.41)
Polymerization shrinkage (%)	3.07 (0.61)	2.19 (0.47)	1.75 (0.12)	1.24 (0.18)	0.84 (0.36)	3.36 (0.62)
Polymerization stress (N)	5.67 (0.21)	5.20 (0.42)	3.94 (0.4)	5.11 (0.31)	4.61 (0.34)	4.63 (0.25)

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Improving mechanical properties of GICs by adding hydroxyapatite and fluorapatite

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Purpose/Aim: The purpose of this research was to improve the mechanical properties of glass-ionomer cement (GIC) by adding hydroxyapatite (HA) and fluorapatite (FA) nanoparticles, while preserving their potent clinical properties.

Materials and methods: HA and FA were added to the powder component of cement (Fuji II, GC gold label, GC international, Tokyo, Japan) at 5% and 8 wt%. Compressive strength (CS) and diametral tensile strength (DTA) before and after 1, 7 and 28 days storing in deionized water were examined. Surface microhardness after 1 and 7 days storing in deionized water was calculated using Vickers microhardness tester. Setting and working time was determined as specified in the ASTM standard. The surface morphology of the modified GICs was evaluated using SEM observations.

Results: The mechanical results of the modified GICs demonstrated that incorporation of HA and FA (5 and

8 wt%) into the glass ionomer cement after 7 days storing in deionized water displayed statistically higher CS about 107–113.6 MPa and 111–117.8 MPa, respectively, also, higher DTS 13–16 MPa and 14–19 MPa, respectively. The hardness of the glass ionomers including HA and FA nanoparticles (5 wt%) were increase by 2.21% and 11.77%, respectively. In addition, working time and setting time by adding the 5% nanoparticles were reduced about 8.5% and 13.23% for HA and 10.63% and 19.11% for FA, respectively.

Conclusions: It was concluded that HA and FA nanoparticles containing glass ionomer cements are promising restorative dental materials with improved mechanical characteristics.

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Preparation of strontium-containing calcium phosphate cements for maxillofacial bone regeneration

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Purpose/Aim: The purpose of this study was to evaluate the performance of a 3 wt% (10% of calcium in hydroxyapatite was replaced with strontium) additive with calcium phosphate cement (CPC) for maxillofacial bone regeneration.

Materials and methods: The phase composition, thermal analysis, microstructure, setting time of the prepared strontium containing calcium phosphate cement was examined. Also, the in vitro testing of MTT assay and alkaline phosphatase (ALP) activities examinations between three groups of 3 wt% Sr-HA/CPC, CPC and control were evaluated and compared.

Results: X-ray diffraction (XRD) demonstrated that enhancing the ratio of Powder/Liquid (P/L), crystallinity of the prepared cement has enhanced. The substitution of strontium instead of calcium in CPC could also change the crystal structure, containing some structural disorder. However, in the CPC with no strontium hydroxyapatite (Sr-HA), no significant increase in crystallinity was seen. SEM observations displayed CPC with enhancing P/L ratio; the formation of hydroxyapatite crystals arising from the interaction of solid and liquid phase of cement was reduced. Also, addition of Sr within Ca site culminates in a dramatic enhance in crystallinity of hydroxyapatite. In vitro biological characteristics reveled that incorporation of 3 wt. % Sr-HA into CPC could cause to increase MTT assay and ALP activity, which may be due to the presence of strontium ions.

Conclusions: The obtained results cleared that CPC could be a potential candidate as a carrier with strontium additives for maxillofacial bone regeneration.

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3-dimensional scaffolds based on PCL/titania nanotube fabricated via microsphere sintering

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Purpose/Aim: In this research, the novel 3D porous scaffolds made of polycaprolactone (PCL)/TiO2 nanotube (TNT) composite microspheres were prepared and evaluated for potential craniofacial bone tissue engineering.

Materials and methods: We used a microsphere sintering technique to fabricate 3D PCL microspheres/TNT (0, 0.5, 1.25 and 2.5 wt%) composite scaffolds. Then, the structural, mechanical and biological properties of these scaffolds were examined.

Results: The mechanical characteristics of composite scaffolds were regulated by varying parameters, such as sintering time, microsphere diameter range size and PCL/TNT ratio. The obtained results revealed that the PCL/TNT (0.5 wt%) scaffold sintered at 60 °C for 90 min had the highest mechanical characteristics and an appropriate pore structure for bone tissue engineering applications. MTT assay and ALP activity results cleared that a general trend of enhancing in cell viability was observed for PCL/TNT (0.5 wt%) scaffold sintered at 60 °C for 90 min by time with compared to control group. The quantitative RT-PCR data provided the evidence that the PCL scaffold containing titania nanotube constitutes a good substrate for cell differentiation leading to ECM mineralization.

Conclusions: This research ascertained combining the use of PCL and titania nanotube to promote bone-like tissue formation is a promising approach for craniofacial bone tissue engineering.

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Biodegradation properties of PLGA/nano-fluorhydroxyapatite composite microsphere-sintered scaffolds

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Purpose/Aim: The purpose of this work was to evaluate the degradation characteristics of the novel 3D porous

composite scaffold made of poly (lactic-co-glycolic acid (PLGA))/(Nano-fluorhydroxyapatite (nano-FHA)) microsphere by using sintering technique for potential bone regeneration in dentistry.

Materials and methods: We have developed a PLGA/n-FHA composite scaffold based on a sintered microsphere technique in order to study in vitro biodegradation behavior of the prepared scaffolds. Then, we evaluate the biodegradation properties (mechanical properties and weigh loss) of these scaffolds after soaking in simulated body fluid (SBF) for various period times.

Results: The experimental results ascertained that PLGA/n-FHA composite scaffold with ratio of 4:1 sintered at 90 °C for 2 h has the greatest mechanical properties and a proper pore structure for bone repair applications. The average weight of both PLGA/n-FHA and PLGA scaffold revealed a monotonic trend with enhancing degradation time that the addition of n-FHA into polymeric scaffold could lead in weight loss in comparison with that of pure PLGA. The pH change for composite scaffolds displayed that there was a slight reduce until 2 weeks after immersion in SBF, followed by a significant increase in the pH of SBF without a scaffold at the end of immersion time. The mechanical characteristics of composite scaffold were higher than that of PLGA scaffold at total time of incubation in SBF, however it should be stated that the addition of n-FHA into composite scaffold lead in decline relatively significant mechanical strength and modulus during hydrolytic degradation. Additionally, MTT assay and alkaline phosphatase activity (ALP activity) results defined that a general trend of enhancing in cell viability was observed for PLGA/n-FHA scaffold sintered by time with compared to control group.

Conclusions: We displayed that a PLGA/nano-fluorhydroxyapatite composite microsphere-sintered scaffolds has excellent mechanical properties, and biocompatibility, and would be a promising materials to achieve bone regeneration needed in the treatment of bone defects in dentistry.

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Device for automatic evaluation of biomaterials micro-leakage by gas permeability

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Purpose/Aim: To design and standardize an automatic device to evaluate the micro-leakage of dental biomaterials by gas permeability.

Materials and methods: The system was design digitally using CAD software. All original pieces were built at the Physics Faculty of the University of Costa Rica (UCR) (Patent pending). The final assembly and the experiments were done at the Research Center of Atomic, Nuclear and Molecular Sciences (CICANUM) of the UCR. The micro-leakage device is composed of 3 phases. The first phase (P1) is an empty chamber (approximately 30 cm^3) with two entrances; one for nitrogen influx and another were a piezoelectric pressure sensor is connected. The second phase (P2) is a duplicate of chamber one, with a second pressure sensor and an exit valve to regulate the gas release. Between both chambers, there is a cylindrical ring (P3) were the samples are placed once prepared. The samples are cylindrical dental human roots calibrated to 10 mm, were the material to be evaluated was placed. The roots are stabilized inside the inner part of the ring using resin, allowing that between both chambers there is only the material-dentin interphase. The experiments begun by closing the system (P1–P3–P2) hermetically. Once secured, the gas is automatically injected in P1 and the pressure lecture is recorded in customized software. Simultaneously, the sensor in P2 register any pressure change in the chamber, and the software calculates the difference of pressure resulting of gas permeation through the dentin–material interphase.

Results: The system was able to control the vacuum and the gas permeation through the chambers. All the controls indicated that the resin chosen is an acceptable material to avoid leakage bias of the system. The negative and positive controls employed (empty roots and roots containing light-cured composite respectively) are suitable options to perform analysis and comparisons of different kind of materials. The system allows performing 3 different experiments. E1: leakage curves evaluating the controlled increase of pressure up to 30 psi. E2: response of the material to constant pressure peaks of 30 psi; and E3: response of the material to maintained pressure of 30 psi for controlled periods.

Conclusions: The device designed is an optimal option to evaluate the adaptability and sealing capability of different kind of biomaterials. Different advantages such as non-destruction of the samples, and multiple experiments in short term are available with this system.

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Synthesis of fluorine-substituted hydroxyapatite nanopowders for dental applications

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Purpose/Aim: In this study, the effect of fluorine ion addition on the biological and bioactivity behavior of hydroxyapatite was evaluated.

Materials and methods: Fluoride-containing hydroxyapatite nanopowders with the general chemical formula

$Ca_5(PO_4)^3(OH)_{1-x}F_x$ ($0 \leq x \leq 1$), where $x = 0.0$ (hydroxyapatite; HA), $x = 0.68$ (fluorhydroxyapatite; FHA) and $x = 0.97$ (fluorapatite; FA) were synthesized. The obtained powders were characterized using X-ray diffraction (XRD), Fourier transform infra-red (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and F-selective electrode analyses. The in vitro dissolution studies of the samples were carried out at osteoclastic resorption conditions. The biological evaluation of the samples was conducted using osteoblast-like cells and L929 cell line, respectively. The bioactivity evaluation of the obtained samples with different fluorine contents was compared by carrying out alkaline phosphatase (ALP) and in vitro dissolution studies of the samples at osteoclastic resorption conditions.

Results: The results cleared that the incorporation of fluorine ion into hydroxyapatite led to a systematic shift in the (3 0 0) and (2 1 1) peak to a higher angle comparing to pure HA of the XRD pattern as well as modifications in the FTIR spectra. TEM observations exhibited that the increase of fluoride ion concentration will cause a reduction in the aspect ratio of the produced crystallites. In vitro dissolution studies clearly demonstrated that it is possible to fine-tune the solubility and correspondingly the biological lifetime of the samples by varying the amount of fluorine substitutions.

Conclusions: By careful selection of fluoride content, the composition and physicochemical and biological characteristics of fluoride-substituted hydroxyapatite could be controlled and this could have interesting implications for the development of these bioceramics for dental applications.

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Novel glassflake resin composites

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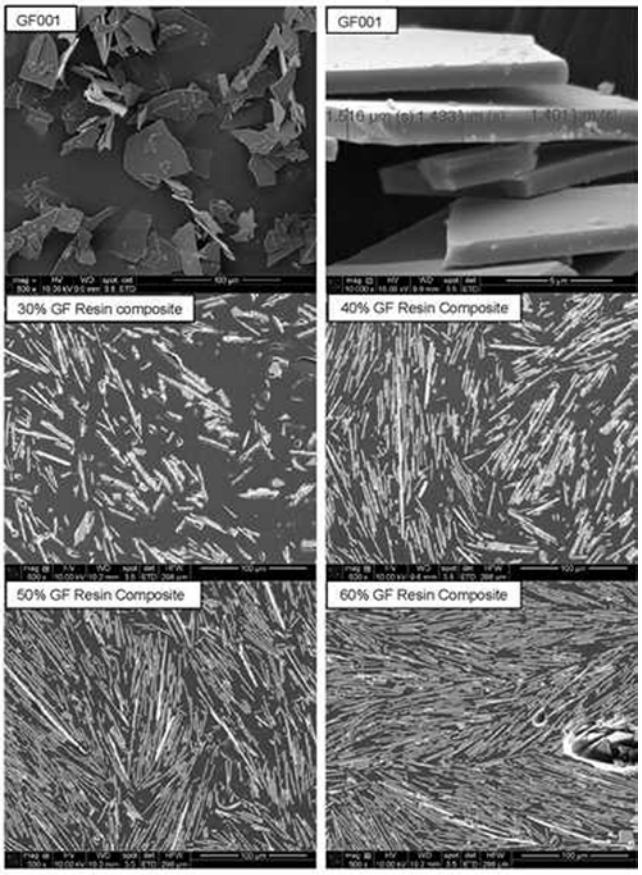
Purpose/Aim: Glassflakes (GFs) are high aspect ratio glass platelets that are widely used in anti-corrosion coatings for the oil industry due to the unique layering characteristic (overlap and align parallel to substrate surfaces as a corrosive barrier). GFs have also been used as planar reinforcements for varies composites and demonstrated better mechanical properties compared with fiber reinforced composites. The aims of the present study were to explore the feasibility of formulating glassflake dental resin composites with suitable depth of cure and glassflake alignment.

Materials and methods: Four glassflake resin composites were formulated by incorporating increasing amount of micronised borosilicate glassflakes GF001 (30–60 wt%) in a 50:50 wt% BisGMA/TEGDMA mixture with a photo-initiator system of CQ and DMAEMA. Cylindrical specimens (4 mm diameter × 4/2 mm thickness, $n = 8$) of each groups were light cured with an Elipar S10 LED curing light and the light output at the bottom surface of the specimen was measured using a laboratory grade NIST-referenced USB-4000 spectrometer. The morphology of the GFs and the resultant GF resin composite were studied by Scanning Electron Microscopy (SEM). Post-irradiance hardness of both top and bottom surfaces (VT and VB) of GF resin composites after 24 h dry storage at 37 °C was measured and the differences between groups were compared using one-way ANOVA ($p < 0.05$) followed by Tukey's post hoc test (Fig. 1).



SEM images of glassflake and the cross-session of glassflake resin composites with different amount of micronised borosilicate glassflakes GF001 as reinforcing filler.

Fig. 1

Results: Glassflakes GF001 demonstrated a high aspect ratio with a thickness between 1.1 and 1.5 μm. Cross sessional SEM images of the GF resin composites showed alignment of GFs orientated along the applied force. GFs were more densely packed and better aligned with an increase in the amount of GFs. All GF resin composites presented lower VB than VT where VB/VT were all above 75%; however, there was no significant difference between the hardness among groups. The light output (irradiance) at the bottom surface of GF resin composites decreased with an increase in the amount of GFs. The bottom irradiance of 4 mm resin composite specimens with lower GFs (30 and 40 wt%) and all 2 mm specimens provided higher irradiance than the minimum clinical acceptable irradiance.

Conclusions: Glassflakes were successfully incorporated in dental resin composites and demonstrated alignment along the applied force. The amount of incorporated glassflakes

showed influences on the depth of cure of glassflakes resin composites and the alignment of the glassflakes.

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Interaction of chlorhexidine with MDP-based dentin bonding systems

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Purpose/Aim: MDP (10-methacryloyloxy-decyl-dihydrogen phosphate) is the main functional monomer used in current commercial self-etching dentin bonding systems. The aim of this study was to investigate its interaction with chlorhexidine in terms of bonding strength (BS) to dentin and degree of conversion (DC) as two independent evaluations.

Materials and methods: Thirty six sound third human molars were selected, prepared and randomly divided into 3 groups according to the dentin bonding system (DBS) ($n=6$): SB – Adper Single Bond-control, SU – Adper Scothbond Universal (self-etching mode) and CSE – Clearfil SE Bond. Half of the teeth were treated with water (control) and the other half with 2% digluconate chlorhexidine aqueous solution (CHX). All specimens were restored with Filtek Z250. Sticks (0.64 mm^2) were obtained and subjected to the microtensile test (μTBS) in a universal testing machine at 0.5 mm/min. Failure mode of interfaces was classified by optical microscopy 40×. For degree of conversion assessments, a drop of each dentin bonding system (≈3.0 μL, $n=3$), combined or not with 2% diacetate of chlorhexidine was assessed using Fourier Transform Infrared Spectroscopy (FT-IR) with attenuated total reflectance (ATR), before and after light curing with Radii Cal/SDI, LED-curing unit (1000 mW/cm^2). For both tests, data was statistically analyzed by two-way ANOVA and Tukey tests ($p<0.05$).

Results: CHX did not impact BS for any DBS: SB – 37.65 + 0.78a; SB-CHX – 31.05 + 3.42a, SU – 50.81 + 9.86b, SU-CHX – 49.34 + 15.28b, CSE – 49.77 + 7.29b, CSE-CHX – 49.88 + 9.41b. SB, MDP-free system, presented the lowest BS compared to MDP-based systems. Adhesive failures were predominant in all groups. Regarding DC, it was influenced by DBS and treatment: SB – 90.57 + 3.59a; SB-CHX – 91.68 + 2.04a, SU – 84.57 + 2.16ab, SU-CHX – 60.01 + 3.53c, CSE – 78.71 + 7.38b, CSE-CHX – 46.14 + 4.74d. Only MDP-based systems were affected by CHX.

Conclusions: MDP-based systems showed satisfactory performance regarding immediate bond strength not compromising their quality when associated with chlorhexidine, although this combination strongly affected their degree of conversion.

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Thermography analysis during laser irradiation on dentin-bonding agents

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Purpose/Aim: Many studies have shown improved physical property with increasing temperature because. The aim of this study was to evaluate the temperature of the dentin bonding agents (DBA) during irradiation of different laser.

Materials and methods: Eight molars were prepared and randomly divided into two groups according to the DBA factor: 2-step total-etch (Adper™ Single Bond 2-[SB]), universal system (Adper™ Single Bond Universal-[SU]) and laser factor: Er:Cr:YSGG laser (2790 nm) and Diode laser (980 nm). A thermographic camera was used to perform the in vitro experiment, in which lasers were used to irradiate tooth samples and the infrared images obtained were stored and analyzed. The beginning of laser irradiation and temperature recording was synchronized but the thermal measuring finished 15 s after the end of the laser irradiation. For later analysis of temperature increments, the standardized point was chosen on the infrared images obtained: one located at the dentin/DBS surface immediately below the laser. Data was statistically analyzed by two-way ANOVA and Tukey tests ($p<0.05$).

Results: Only laser factor ($p<0.05$) was statistically significant. Therefore interactions between factors did not exist. In both DBA, Er:Cr:YSGG laser showed higher heating surface (dentin/DBA) and Diode laser lowest values.

Conclusions: Er:Cr:YSGG laser may be promising and alternative to improve the physical properties of DBA.

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Proteolytic-inhibitors use for bonding carious/eroded dentin: Does it work?

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Purpose/Aim: Dentin is commonly altered through challenges as caries and erosion, modifying their ability to interact with dentin bonding systems (DBSs). Strategies that favor for adequate bonding and improve their durability include the

association with proteolytic inhibitors. The aim of this study was to evaluate the effect of proteolytic inhibitors on bond strength (BS) of a universal adhesive system (etch-and-rinse mode) to pretreated dentin in artificial carious and eroded dentin.

Materials and methods: Ninety molars were prepared and randomly divided into three groups according to the substrate: N – no challenges (stored in artificial saliva), CA – artificial caries simulation (6 h DE + 18h-RE/5 days + 48 RE) and ERO-artificial erosion simulation (3 × 5 min/5 days with orange juice). All groups were redivided into three subgroups according to the dentin pretreatment: W – water, CHX – 2% chlorhexidine and E-64 – 5?M E-64 inhibitor. They constituted 9 groups ($n = 10$): N-W, N-CHX, N-E64, CA-W, CA-CHX, CA-E64, ERO-W, ERO-CHX, ERO-E64. All specimens were restored with Adper Single Bond Universal®/Filtek Z250. Sticks (0.64 mm^2) were obtained and subjected to the microtensile test (μTBS) in a universal testing machine at 0.5 mm/min after 7 days, 6 months and 18 months. Failure mode of interfaces was classified by optical microscopy 40×. Data was statistically analyzed by three-way ANOVA and Tukey tests ($p < 0.05$).

Results: All individual factors ($p < 0.0001$) and interaction between factors were statistically significant. Caries substrate was the most affected substrate. CHX impacted to BS and it was not able to improve their durability over time regarding any substrate.

Conclusions: The use of any proteolytic inhibitors to treat affected dentin seems not to improve BS associated with a universal bonding system after 18 months. Controversially, CHX seems to react negatively to this system.

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Abfraction *versus* erosion: Impact of type-lesion on 1-year restorative performance

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Purpose/Aim: Resin-modified glass-ionomer cement (RMGIC) is recommended for the treatment of non-carious cervical lesions (NCCL) irrespective to their etiological factor. However, as these factors frequently persist even after restorative treatment, this study aimed to evaluate the restorative performance predominantly caused by abfraction or erosion.

Materials and methods: Patients presenting exclusively lesions of abfraction ($n = 38$) or erosion ($n = 14$) were recruited, who were distributed into these two groups. Two calibrated operators performed the restorative procedures (RMGIC – Vitremer/3M) using rubber dam with no cavity preparation. Vitremer was prepared and inserted following manufacturer's instructions. Evaluations were made with a modified USPHS method in the baseline and after 6 and 12 months, by two calibrated examiners regarding the criteria: retention, marginal adaptation, marginal staining, post-operative sensitivity, adjacent caries, color alteration, anatomic form and surface texture. The intra and inter examiner Kappa index were 0.98 and 0.95, respectively. Data was collected and statistically assessed with Mann–Whitney and Friedman/Tukey tests were performed ($p < 0.05$). SEM images were also obtained.

Results: Overall survey revealed predominance of localized abfraction lesions than erosive lesions, which occurred as generalized lesions in few patients. The clinical performance of RMGIC were time and lesion type-dependent. Erosive lesions were more susceptible to alterations from 6-month analyzes regarding marginal adaptation and surface texture. SEM images also pointed out these differences. Comparisons regarding other parameters did not show any alteration irrespective the lesion.

Conclusions: NCCLs etiologic factor determines differences in clinical performance of RMGIC restorations after 1 year.

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Thermally oxidized Ti_6Al_4V alloys enhance the corrosion behavior

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Purpose/Aim: We aimed to study in the current work the structural and electrochemical properties of different heat treated Ti6Al4V substrates under oxygen flow in the temperature range of 600–800 °C.

Materials and methods: The morphology, chemical composition and phase composition of synthesized layers were investigated using field emission scanning electron microscopy (FE-SEM) and X-ray diffraction (XRD). The corrosion behavior of the different oxide layers was evaluated in 0.9 wt% NaCl solution with pH = 6.4 at room temperature by means of open circuit potential (Eoc), potentiodynamic polarization (PDYN) and electrochemical impedance spectroscopy (EIS).

Results: The XRD patterns of thermal oxidized samples exhibit the presence of anatase phase only at 500 °C while rutile phase begins to appear from 600 °C and becomes the predominant phase at 800 °C. Surface morphology of thermal oxidized samples reveals the presence of oxide scales throughout the surface. All thermally oxidized samples exhibit a shifting in open circuit potentials and corrosion potentials toward positive values.

Conclusions: It is found that thermal oxidation can effectively enhance the surface and improve the corrosion resistance of Ti6Al4V alloy. And the optimal oxidation temperature is 700 °C.

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Endocrine disruptors in paediatric patients & *in-vitro* sealant weight variability

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Purpose/Aim: Numerous publications suggest that bisphenol A (BPA) could pose a threat to human health with children being more at risk than adults. Resin-based dental materials are a potential source of exposure to BPA in some clinical situations. Environmental exposures to plasticisers prior to dental treatment however, may be significant. The purpose of this research was to quantify baseline concentrations of urinary endocrine-disrupting compounds in paediatric dental patients and to determine if those patients receiving sealants had elevated urinary BPA/phthalate metabolite concentrations 24-h post-treatment. Subsequently, the variability of applied sealant weight was determined using synthetic teeth.

Materials and methods: Baseline urine samples were collected from patients ($n = 63$), with 11 patients receiving sealants (Delton Light-Cure) based on individual dental care needs. Urinary BPA and phthalate metabolite enzymatically deconjugated concentrations were determined using a validated SPE-HPLC-MS/MS method. Thirty-six synthetic teeth (KaVo); (12 × molars 5, 6 and 7) were weighed before sealant was applied with either a discoid excavator, a brush or microbrush, cured (LED Cure-Light) and re-weighed.

Results: Mean baseline urinary BPA and MEHP concentrations ($n = 63$) were 8 and 14.4 μg/g creatinine. Summary data for treated patients is shown in the table. BPA concentration was elevated in 8 of 11 patients (up to 40-fold) but the difference was not significant ($p = 0.057$). Mean sealant weights ($n = 12$) applied to synthetic teeth for all three tools were: tooth #5 3.27 mg (SD ± 1.09), tooth #6 8.08 mg (SD ± 2.26), tooth #7 6.56 mg (SD ± 1.54).

Conclusions: Baseline urinary concentrations of BPA and phthalate metabolites were comparable with US and German data. BPA and phthalates have similar non-dental sources of exposure i.e. food packaging. However, large increases in BPA concentrations in most of the treated patients were not reflected by increased phthalate concentrations, suggesting a significant non-food packaging source. Twenty-four hour post-treatment samples were collected for practicality and despite an elimination half-life of 5.5 h, increases, although highly variable, were observed in BPA concentrations. There was no correlation between BPA concentration and the number of sealants applied. The variability in applied sealant weights (in-vitro) was higher than expected, given the controlled conditions with identical standardised model teeth and the sealant being applied consecutively by an experienced clinician. In the clinical setting, with different degrees of technical and clinical expertise, and with real teeth, it is likely that variability will be much higher and could account for some of the variation seen in BPA concentrations in the treated patient samples.

	μg/g urinary creatinine			
	Mean	Median	Min	Max
BPA				
Baseline1 ($n = 10$)	9.41	4.55	0.00	82.63
Baseline2 ($n = 11$)	9.36	4.99	0.68	52.59
Post-treatment ($n = 11$)	46.10	13.80	2.40	200.30
MEHP (primary DEHP metabolite)				
Baseline1 ($n = 10$)	41.3	6.50	2.50	237.11
Baseline2 ($n = 11$)	7.83	6.40	1.36	29.16
Post-treatment ($n = 11$)	6.41	4.08	0.60	16.88

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Mechanical, microbiological and copper release from copper nanoparticles-containing adhesives

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Purpose/Aim: To evaluate the effect of addition of copper nanoparticles (CN) at different concentrations into a simplified etch-and-rinse (ER) adhesive on antimicrobial activity (AMA), copper release (CR), ultimate tensile strength (UTS), degree of conversion (DC), water sorption (WS), solubility (SO), as well as, the dentin bonding (μTBS) and nanoleakage (NL).

Materials and methods: Seven experimental ER were formulated according to the addition of CN (0–1 wt%) in ER adhesive. After build-up the specimens, we tested the AMA of experimental adhesives against *Streptococcus mutans* using agar diffusion assay after 24 h. For CR, WS and SO specimens were stored and measured for 28 days. For UTS, specimens were tested after 24 h and 28 days. For DC, specimens were constructed and tested after 24 h by FTIR. The occlusal enamel of thirty-five molars was removed and adhesives were applied to dentin surface after 37% phosphoric acid etching. After composite resin build-ups, sticks were obtained and tested after 24 h (μTBS and NL). The data were submitted to appropriate statistical analysis ($\alpha = 0.05$).

Results: CN provided antimicrobial properties to the adhesives at all concentrations and significant decrease NL. Higher CR was observed in adhesives with higher concentration of CN. UTS, DC, WS and SO were not influenced. For μTBS an increase was observed in 0.1 and 0.5% copper group.

Conclusions: The addition of CN in concentrations up to 1 wt% in the ER adhesive may be an alternative to provide

antimicrobial properties and increase the dentin bonding, without reducing mechanical properties.

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Preventive effect of potassium nitrate-glutaraldehyde gel in the bleaching-induced sensitivity

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Purpose/Aim: Although dental bleaching is one of the cosmetic treatments most requested by patients in dental offices, the in-office bleaching produces a high risk of bleaching-induced tooth sensitivity, which may affect more than 60% of the patients. In face of that, the aim of this randomized, split-mouth, triple-blind study was to evaluate the efficacy of a desensitizing gel based on 5% potassium nitrate and 5% glutaraldehyde applied before in-office bleaching with 35% hydrogen peroxide.

Materials and methods: A total of 42 patients were included in the sample. Each side of the patient's upper dental arch was randomly assigned to either the control group or experimental group. Before in-office bleaching with a 35 percent hydrogen peroxide gel (three applications of 15 min each), clinicians applied a placebo gel and the desensitizing agent (composed of 5% potassium nitrate; 2% sodium fluoride and 5% glutaraldehyde) to in the same manner in the respective randomized arch side. They repeated this protocol one week later. Patients recorded their tooth sensitivity on a 0-to-4 NRS scale and on a 0–10 VAS scale. Color change was evaluated with a spectrophotometer (Vita Easyshade) and two shade guide units (Vita classical and Vita Bleachguide 3D-Master). The risk of bleaching-induced tooth sensitivity of both groups was compared with the McNemar's test and the color change was compared with the paired *t*-test ($\alpha = 5\%$).

Results: The color change that occurred in both sides of the patient's upper dental arch was statistically similar ($p > 0.05$) and occurred towards the lighter end of the shade guide units in all color scales and in the spectrophotometer. The risk of bleaching-induced tooth sensitivity in the control group was [31.7% (95% CI 19.6–46.9)] statistically lower ($p < 0.0001$) than that observed in the experimental group was [70.7 (95% CI 55.5–82.3%)].

Conclusions: It was concluded that the previous application of the experimental desensitizing agent based on 5% potassium nitrate and 5% glutaraldehyde before in-office bleaching reduced the risk and intensity of bleaching-induced tooth sensitivity without jeopardizing the bleaching effectiveness.

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Proteolytic activity of dentin caries-like lesions provided by smutans biofilm

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Purpose/Aim: Several methods have been developed to produce artificial caries lesions, since caries-affected dentin has being used in studies targeting restorative materials bonding to dentin. It has already known that natural caries lesions presents a substantial proteolytic activity by the activation of host matrix metaloproteinases (MMPs) in dentin, however, there is no information about enzymatic activity of dentin when submitted to artificial caries-like methods. Thus, this study evaluated the in situ proteolytic activity of dentin demineralized by an artificial biological caries model.

Materials and methods: Fifteen coronal dentin specimens were randomly allocated into two groups: NC – natural caries ($n = 3$); and BM – dentin caries lesion produced by artificial biological model (12 teeth), which was divided into four subgroups ($n = 3$), according to the time of demineralization: BM3 – three, BM6 – six, BM12 – twelve and BM18 – eighteen days of demineralization. Dentin specimens from BM group were kept in sterile brain-heart infusion (BHI) broth supplemented with 0.5% yeast extract, 1% glucose, and 2% sucrose, where *S. mutans* (UA159) was inoculated with initial concentration of 0.05 at 550 nm (A550). Broth was renewed daily and, at each time point (3, 6, 12 and 18 days) three specimens were removed from the bacterial suspension (pH = 4.0). Dentin specimens from NC group were used as control. After demineralization, slices (200 μm) of each specimen were prepared and carried to in situ Zymography, which was performed with quenched fluorescein-conjugated gelatin as the MMP substrate. Endogenous enzymatic activity was assessed by multi-photon confocal microscope. Polarized light microscopy confirmed the depth of dentin demineralization. Intensity of fluorescence was measured quantitatively and compared between BM groups ($p < 0.05$).

Results: For all BM groups, it was observed higher green fluorescence in the zone corresponding to demineralized dentin than the underlying sound layer zone, indicating that the fluorescein conjugated gelatin was strongly hydrolyzed in the first zone, differently from natural caries group that presented green fluorescence in all extension of specimens. Intensity of fluorescence increased over time in demineralized zone ($p < 0.01$), whereas the fluorescence did not changed after six days in sound area ($p > 0.05$).

Conclusions: Proteolytic activity of artificial caries-like lesions produced by biological model occurred predominantly in the zone where dentin had been demineralized, as result of release collagen bound-MMPs.

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Hydroxyapatite/dicalcium phosphate dihydrate composite for dental bone defect repair

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Purpose/Aim: The purpose of this study was to evaluate a new porous calcium phosphate composite composed of hydroxyapatite (HA) and dicalcium phosphate dihydrate (DCPD) as a bone substitute for dental bone defect repair.

Materials and methods: The HA/DCPD composites were prepared by foam casting method using polyurethane foam, designed for osteoconductive scaffolds. Then, the prepared samples were characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM). The density, porosity and pore size of these samples were measured and finally, the mechanical properties of them were examined in this research.

Results: The obtained results cleared that the elastic modulus, compressive strength and density of the samples decreased with increasing percentage of DCPD phase. The mechanical characteristics of the prepared samples were near the natural compact bone. Biomineralisation studies demonstrated the deposition apatite phase on the surface of the scaffolds exhibiting the bioactive nature of the scaffolds. Additionally, the in vitro results revealed that the scaffolds were biocompatible.

Conclusions: The success of applying biomaterials composed of HA and DCPD as bone substitute for bone defect repair is related to the fact that this combination is biocompatible and forms a favorable 3-dimensional matrix for human osteoblast cells to adhere and spread, associating the advantage of DCPD osteoinduction to the superior bioactivity and osteoconduction of HA. Finally, the prepared composite samples fabricated from HA and DCPD could be considered as highly bioactive bone substitute scaffold materials for dental bone defect repair.

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Biocompatibility of modified glass-ionomer cements on periodontal ligament (PDL) cells

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Purpose/Aim: The purpose of this study was to examine the biocompatibility of the glass-ionomer cement (GIC) by adding hydroxyapatite (HA) and fluorapatite (FA) nanoparticles, while preserving their potent clinical properties.

Materials and methods: HA and FA were incorporated to the powder component of the cement (Fuji II, GC gold label, GC international, Tokyo, Japan) at 5% and 8 wt%. The cytotoxicity of a GIC, 5%HA modified GIC, 8%HA modified GIC, 5%FA modified GIC and 8%FA modified GIC has been examined using cement extracts, a culture media modified by the cement. The pH of each material group in the culture media were determined and correlated to the results of the biocompatibility study.

Results: Among the five group samples, GIC revealed the most cytotoxicity effect, followed by FA modified GIC and HA modified GIC.

Conclusions: Our research demonstrated that the biocompatibility of the GIC can be moderated by incorporating HA and FA based ceramics. The modified GIC could be promising dental restorative cements with improved biological characteristics.

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3D-printing of porous calcium phosphate cements for bone tissue engineering

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Purpose/Aim: The ability of inkjet-based 3D printing to produce bioceramics has made it one of the most reproducible method to create the scaffolds for bone tissue engineering. The main advantage of hydroxyapatite (HA)-forming calcium phosphate cements (CPCs) employed as the scaffold in bone tissue engineering is their setting under physiological conditions without the necessity for heat treatment that permits the addition of biological elements. The aim of this study was to evaluate the biological and mechanical properties of the 3D-printed CPC.

Materials and methods: In this research, we fabricate the biocompatible calcium phosphate cement (CPC) scaffolds by 3D printing of a storable CPC paste based on water-immiscible carrier liquid with defined inner and outer morphology. We revealed the application of the 3D printing technique for the preparation of the CPC-based scaffolds. Printing and hardening of the CPCs were performed under mild and malleable conditions permitting the (precise and local) integration of biological elements.

Results: The CPC pastes displayed an appropriate processability in the 3D printing technique and permitted the preparation of the stable 3D structures with sufficient compressive strength and young modulus. It was found that CPC pastes were stable and their setting reaction is initiated only after soaking in the water. Also, the Biocompatibility and bioactivity of the printed CPC-based scaffolds was ascertained in a cell culture experiment with osteoblast cells.

Conclusions: The malleable and sterile conditions of the all experimental procedure during 3D make this technique highly attractive for production of customized scaffold with respect to patient-specific demands.

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3D-printed β-TCP/collagen scaffolds for bone tissue engineering

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Purpose/Aim: Bone tissue engineering is sought to apply strategies for bone defects healing without limitations and short-comings of using either bone autografts or allografts and xenografts. The aim of this study was to fabricate and characterize the thin layer collagen- coated 3D printed beta-tricalcium phosphate (β-TCP) scaffolds.

Materials and methods: β-TCP scaffolds were fabricated using 3D-printing technique. Scaffolds were prepared from 3D printed paste composed of β-TCP, carboxymethyl cellulose (CMC) as binder and tripolyphosphate (TPP) as viscosity modifier. The printed scaffolds were then sintered at 1100 °C, followed by coating with a thin layer collagen via dip coating. The prepared scaffolds were evaluated by scanning electron microscopy (SEM), X-ray diffraction (XRD) analyses. Also, the mechanical properties of theses printed scaffolds were determined. Finally, the in vitro biological characteristics of the scaffolds such as biocompatibility and bioactivity were assessed using human osteoblast cells (HOB).

Results: The β-TCP/collagen scaffolds were found to display compressive strength near cancellous bone. The mean porosity and pore size of the prepared printed scaffolds was about 87% and 500 micrometers, receptively. The obtained results from in vitro biological properties revealed that the rate of cell proliferation is significantly improved for collagen-coated β-TCP scaffolds than β-TCP.

Conclusions: In conclusion, this study ascertained that optimization of material parameters for 3D printed β-calcium phosphate scaffolds and improvement of material characteristics by collagen incorporation via inkjet printing.

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3-D printed PCL/halloysite scaffolds for craniomaxillofacial bone regeneration

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Purpose/Aim: The purpose of this experimental study is to assess the compressive durability of three-dimensionally printed PCL/Halloysite composite scaffolds in order to optimize their suitability for future clinical attempts at regenerating craniomaxillofacial bone. It is our hope that this study will affirm our hypothesis that the addition of Halloysite will improve the compression strength of PCL.

Materials and methods: Following a solvent casting method to prepare the desired nanocomposite, the PCL/Halloysite was printed by hot extrusion with a commercial bioprinter at an optimal pressure and printing speed of 2.5 bar and 4 mm/s, respectively, to produce scaffolds of 3 wt% and cubic dimensions of 5 × 5 × 5 mm.

Results: The samples exhibited proper bioactivity by being conducive to osteoblast growth and the addition of Halloysite improved, to a certain degree, the compression strength of the scaffolds. Additionally, SEM micrographs displayed excellent morphology.

Conclusions: The experiment demonstrated that Halloysite concentrations can be manipulated to produce nanocomposite scaffolds with optimal mechanical characteristics, while still retaining its osteoconductivity, for craniomaxillofacial bone regeneration. With consistent durability shown in vitro, it is our hope to advance the scaffold's capabilities to in vivo studies and clinical trials in the future.

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Biomimetic analogs/bioactive phosphates based adhesives promote dentin collagen mineralization

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Purpose/Aim: The aim of this study was to use ultra-morphology (TEM) to investigate the remineralization of

resin–dentin interfaces (RDI) created with an experimental multi-mode adhesive system containing biomimetic analogs and bioactive phosphates.

Materials and methods: The adhesive systems included: primer (15% GDMA-P; 10% HEMA, 10% TEG-DMA, 10% UDMA, and 40% ethanol); and adhesive resin (30% UDMA, 27% BisEMA, 10% TEGDMA, 10% GDMA-P and 3% photoinitiator system). The analogs (5% polyacrylic acid and 10% polyvinylphosphonic acid), and the bioactive phosphates (20%) were added to the primer and/or adhesive resin. The following groups were tested for ultra-morphology of the RDI ($n = 24$), according to either an etch-and-rinse (ER) or self-etching (SE) strategy: Control-E&R ($n = 3$)/SE ($n = 3$) (no analogs or bioactive phosphates); Exp-1 E&R ($n = 3$)/SE ($n = 3$) (no analogs but bioactive phosphates), Exp-2 ER ($n = 3$)/SE ($n = 3$) (analogs and no bioactive phosphates), Exp-3 ER ($n = 3$)/SE ($n = 3$) (analogs and bioactive phosphates). Flat sound mid dentin was revealed and a standard smear layer created. For E&R strategy, phosphoric acid was applied for 15 s, rinsed for 15 s and dried leaving dentin moist. A primer solution was applied for 20 s and dried for 15 s. For SE strategy, a primer solution was applied for 20 s, then dried for 10 s. Both strategies received a layer of adhesive and light cured for 20 s. A 0.5 mm layer of a microfilled resin-based composite was added/light cured 30 s. After 24 h and 4 M storage in simulated body fluid, beams of $0.3 \times 0.3 \times 4$ mm were obtained from the resin–dentin interface (RDI) and processed following a conventional TEM protocol. Ultra-thin sections (90 nm) were obtained and the morphology evaluated (Hitachi, H-7600).

Results: Ultra-thin sections demonstrated a well-established RDI for all groups. Phosphates conglomerates were observed in the adhesive layer only for groups containing the bioactive phosphate. In both strategies, only the experimental groups containing analogs showed a higher number of needle-like crystals at the bottom of the RDI.

Conclusions: Incorporation of analogs, such as polyacrylic and polyvinylphosphonic acids, into etch-and-rinse and self-etching adhesive systems facilitates remineralization of depleted collagen present in the RDI.

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Photo-fenton: An alternative for dental bleaching

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Purpose/Aim: Consumption of colored drinks and food has been identified as one of the main causes for the staining of tooth structure. The oxidation of these pigments during dental bleaching treatment is achieved by the use of H_2O_2. Virtually unknown to the dental field are the advanced oxidation processes (AOPs), able to generate a large number of stronger oxidants, and which may be an alternative to conventional dental bleaching. This study evaluated, in vitro, the bleaching of a common food stain through the use of a well-known AOP called Photo-Fenton reaction.

Table 1 – Experimental design.

Group	[%] Ferrous gluconate	[%] H_2O_2	Irradiation time (min)
1	0.0015 (−1)	10 (−1)	15 (−1)
2	0.0045 (+1)	10 (−1)	15 (−1)
3	0.0015 (−1)	25 (+1)	15 (−1)
4	0.0045 (+1)	25 (+1)	15 (−1)
5	0.0015 (−1)	10 (−1)	45 (+1)
6	0.0045 (+1)	10 (−1)	45 (+1)
7	0.0015 (−1)	25 (+1)	45 (+1)
8	0.0045 (+1)	25 (+1)	45 (+1)
9	0.0005 (−1.682)	17.5 (0)	30 (0)
10	0.0055 (+1.682)	17.5 (0)	30 (0)
11	0.0030 (0)	4.9 (−1.682)	30 (0)
12	0.0030 (0)	30.1 (+1.682)	30 (0)
13	0.0030 (0)	17.5 (0)	4.8 (−1.682)
14	0.0030 (0)	17.5 (0)	55.2 (+1.682)
15	0.0030 (0)	17.5 (0)	30 (0)
16	0.0030 (0)	17.5 (0)	30 (0)

Materials and methods: Following the experimental planning (Table 1) 16 aqueous solutions containing cochineal carmine dye (0.17 g/L) (C) + hydrogen peroxide (5–30%) (P) + ferrous gluconate (0.0005–0.0055%) (G) were irradiated by the LED lamp Zoom! (Philips, ZOOM!) for a period of time between 5 and 55 min. With an absorption spectrophotometer UV–vis the remaining dye in the solutions was measured. For each solution three repetitions were carried out ($n = 3$).

Results: The color removal values were analyzed based on a response surface obtained through the software Statigraphics 5.1, which indicated variations in the results dependent upon the experimental condition. The concentration of G and the irradiation time had a positive effect on dye removal, varying from 81% to 96%; nevertheless, the use of P alone showed no major effect on color removal, ranging from 5% to 14%. The hydrogen peroxide for the conditions evaluated did not have a significant improvement on the removal of the dye.

Conclusions: The photo-Fenton reaction presented a high potential for dye removal for the conditions evaluated; the dye removal efficacy was dependent on the concentration of the ferrous gluconate and the irradiation time. Further evaluations should be made with different concentrations of P, G and irradiation time.

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Resistance of resin-based materials against prolonged erosive and abrasive challenges

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Purpose/Aim: This in vitro study evaluated the effects of applying resin-based materials, including resin infiltration, on previously eroded enamel subjected to prolonged erosive and abrasive challenges (30 days).

Materials and methods: For that purpose, 144 bovine enamel blocks were immersed in hydrochloric acid (HCl), 0.01 M (pH 2.3), for 30 sin order to form a softened erosion lesion. The blocks were then randomly assigned to four groups ($n = 36$): C = control without treatment; Hel = pit & fissure resin sealant (Helioseal Clear); Adh = conventional adhesive system (Adper Scotchbond Multi-Purpose Plus Adhesive); and Inf = infiltrant (Icon). The materials were applied on enamel-eroded surface following the manufacturer's instructions. The treated enamel blocks were than subjected to three types of challenges ($n = 12$ for each challenge in each group): erosion, abrasion and erosion + abrasion. Erosive challenge was performed by the immersion of the blocks in HCl for 2 min followed by immersion in artificial saliva for 120 min four times a day for 30 d. Abrasion was simulated by using a tooth brushing machine with the application of slurry of 1450 ppm F^- toothpaste (NaF) and tooth brushing (200 g) during 15 s, two times a day for 30 d. On erosion plus abrasion after the second and forth erosive cycle, the blocks were brushed. Enamel alteration and material thickness were analyzed using profilometry, and the results were submitted to Two Way ANOVA (Factors group and type of challenge) followed by Tukey test ($p < 0.05$).

Results: The groups Hel, Adh and Inf resulted in the formation of a layer of material over the enamel which was effective in inhibiting enamel loss. For the control group abrasion promoted less enamel loss, followed by erosion and erosion plus abrasion, with significant difference among them ($p < 0.05$). However, the impact of erosion, abrasion and erosion + abrasion was similar on the resin based studied groups ($p > 0.05$). The groups Hel, and Inf showed less material losses when compared to Adh group ($p < 0.05$).

Conclusions: In conclusion, all the studied materials were able to protect the enamel against erosion and abrasion wear, however, the adhesive showed the higher material loss.

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Transmittance of ceramics and dual-cure resin cements polymerization

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Purpose/Aim: The aim of the present study was to evaluate the transmittance of different ceramic types and shades and their effects on the degree of conversion (DC) of two dual-cure resin cements.

Materials and methods: Sixty discs were fabricated with IPS e.max ceramic (Ivoclar Vivadent, Schaan, Liechtenstein) and divided into three groups ($n = 20$ in each group): LT, MO and Z. For LT and MO groups discs were fabricated with low translucency (LT) and medium opacity (MO) lithium disilicate ceramic, respectively. Discs from Z group were manufactured of zirconia. Each group was divided into five subgroups ($n = 4$), according to the ceramic shade: A2; A3,5; B2; C2; D3. In the LT group, specimens were heat-pressed in shades cited above and in the MO and Z groups, the discs were initially fabricated as core materials and then veneered with veneer ceramic (IPS e.max Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) in the same shades. A spectrophotometer UV-1800 Shimadzu was used to determine the transmittance percentage of each ceramic specimen (2.0-mm-thick). For DC measurements, the resin cements (Variolink II – Ivoclar Vivadent, Schaan, Liechtenstein and Rely X U200 – 3M ESPE, St. Paul, MN, USA) specimens (thickness: 100 μm) were photocured under the ceramic discs (2.0-mm-thick) for 40 s. Specimens photocured without the ceramics discs were used as control group. ATR/FTIR spectrometry was used to evaluate the extent of polymerization for all cement specimens immediately after photocuring.

Results: The results were submitted to one-way ANOVA and Tukey test ($\alpha = 5\%$). The transmittance percentage was less than 1% and the LT group had the highest transmittance values, followed by MO and Z groups. The %DC of Variolink II cement was not influenced by the ceramic disc interposition. For Rely X U200 cement, the interposition of some ceramics types/shades (LT A3,5, MO A2, MO A3,5 and Z A3,5) significantly decreased the %DC compared to control group.

Conclusions: It was concluded that the transmittance values and %DC were influenced by the ceramics types/shades. However, only the LT group, using the Rely X U200, showed a negative correlation between transmittance and %DC.

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Microfluidic-directed synthesis of polymeric nanoparticles for bone cancer therapy

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Purpose/Aim: Finding an efficient approach for cancer therapy has been always of great interest. Recently, microfluidic-assisted methods for synthesis of polymeric particles have shown great potential to design drug delivery carriers. Here we present microfluidic technique as a promising approach to encapsulate anticancer drugs in fine-tunable polymeric nanoparticles for potential bone cancer treatment.

Materials and methods: Doxorubicin was encapsulated in poly(lactic-co-glycolic acid) (PLGA) nanoparticles trough on-chip nanoprecipitation technique. A cross-junction hydrodynamic flow focusing microfluidic platform with three inlets and one outlet was used in this study.

Results: It is demonstrated that the microfluidic nanoprecipitation method provide improved monodispersity, encapsulation efficiency and well-regulated release profiles of particles compared with conventional bulk synthesis methods. Various flow rates ratio (polymer solution flow to water stream) and solvents were examined to make nanoparticles with different size. The in vitro release results revealed a sustained release profile of Doxorubicin, which could be customized depending on the particle size.

Conclusions: The synthesized PLGA nanoparticles have the potential to be used as carriers for anticancer drug delivery. The sustained release of the loaded doxorubicin in synthesized PLGA nanoparticles promises enhanced efficiency for efficient bone cancer therapy.

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Color stability and flexural strength of 4-meta/MMA-TBB resin

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Purpose/Aim: The color stability and the mechanical property of self-curing resin composite may be affected by different powder-liquid ratios. This study evaluated the effect of three different liquid-powder ratios on the color stability after immersion in staining solution and on the three-point flexural strength of 4-META/MMA-TBB resin (Bondfill SB).

Materials and methods: Materials and methods. For evaluation of the color stability, three different liquid-powder ratios (1:0.8, 1:1, 1:1.2) of 4-META/MMA-TBB resin (Bondfill SB) and a light curing resin composite (Filtek Z350XT) were used. Seventy disk-shape samples ($n=10$ per group) were prepared from three different materials using custom made acrylic mold. The diameter of the specimen is 6 mm and height is 1 mm. The CIE $L^*a^*b^*$ values of specimen were measured using a spectrophotometer over a black and white background. The color difference (ΔE, L^*, a^*, b^*) were measured at the time interval of baseline, after 1 day, 1 week, 2 weeks, 3 weeks, 4 weeks and analyzed by one-way ANOVA, followed by Tukey's HSD test ($\alpha=0.05$). To assess flexural strength according to ISO Standard 4049/2000, thirty-six bar-shaped specimens ($n=12$ per group) were prepared for 3 groups with different powder-liquid ratios (1:0.8 light, 1:1 light, 1:1.2 light) using metallic mold. Three different powder-liquid ratios of 4-META/MMA-TBB resin (Bondfill SB) were used. 2.0 mm width, 2.0 mm height, 25 mm length specimens were prepared for three-point flexural strength test.

Results: The ΔEs suggested that three different powder-liquid ratios do not affect the color stability of 4-META/MMA-TBB resin over all the time interval. In the point of flexural strength, 8 specimens of 1:0.8 group, 7 specimens of 1:1 group, and 2 specimens of 1:1.2 group were not fractured over 15% of compressive strain.

Conclusions: The color stability of 4-META/MMA-TBB resin on staining solution displayed stable regardless liquid-powder ratio. The mechanical property of 4-META/MMA-TBB resin may be affected by liquid-powder ratio.

	D0	D1	D7	D14	D21	D28
1:1L	11.97758	12.024	12.271	12.674	11.558	10.998
1:1.2L	10.42987	10.319	9.847	10.742	10.298	9.580
1:0.8L	11.91611	12.851	12.241	14.363	12.320	11.536
1:1M	12.0827	12.256	11.798	13.279	13.045	12.626
1:1.2M	10.24955	10.580	11.923	10.816	10.545	10.618
1:0.8M	12.15217	12.190	11.497	12.868	13.998	11.396
Z350	12.40571	12.416	11.815	12.135	11.618	11.043

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Flexural strength of dual-cured provisional resin with and without visible-light

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Purpose/Aim: This study examined the effect of light irradiation on flexural strength for light or chemically activated provisional prosthesis material after the thermal cycling.

Materials and methods: A commercially available provisional crown and bridge material TEMPSMART (GC, bis-acrylic dual-cured composite resin) was tested. The composite resin was provided with two pastes mixed with syringe automatically at manufacturers' recommended paste/paste ratio. Rectangular specimens (2 mm wide, 2 mm thick, 25 mm long) were prepared without and with light irradiation (60 s) using curing lamp (NEW LIGHT VL-II, GC). One hour after the start of mixing, each specimen was subjected to thermal cycling between $5\pm2^{\circ}C$ (60 s) and $55\pm2^{\circ}C$ (60 s) of water up to 10,000 cycles for using the thermal cycle equipment (K178, Tokyo-Giken). The flexural strengths of each specimen were determined using a universal testing machine (Instron 3366) at one hour (baseline), 5000 thermal cycles or 10,000 thermal cycles ($n=10$). The results were statistically analyzed using ANOVA/Scheffé's test at $p=0.05$.

Results: Table displays the flexural strength of the provisional prosthesis material ($n=10$, mean $\pm$ SD, MPa). ANOVA indicated that significant effects were found for light irradiation and thermal cycling ($p<0.05$). At baseline the flexural strengths of light cured specimens were significantly greater than those of without light ($p<0.05$). No significant effect on flexural strength was found for the light cured specimens after thermal cycling tested ($p>0.05$).

Conclusions: Under the present experimental conditions, the flexural strength of light cured specimens was stable after thermal cycling compared to those without light irradiation. The light irradiation to the dual-cured resin appeared to accelerate polymerization process, which affects their mechanical property.

	Baseline (MPa)	5000 thermal cycles (MPa)	10,000 thermal cycles (MPa)
Without light	72.5 ± 4.1	89.8 ± 6.9	82.9 ± 6.3
With light	90.8 ± 7.4	93.7 ± 5.0	94.8 ± 4.5

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Thermographic analysis of photo-cured composite in tooth cavity

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Purpose/Aim: The purpose of this study was to evaluate temperature rises in the composite and dentin in class I cavity of extracted human molar when restored with high-viscosity conventional or bulk fill composites using different layering methods and light curing units.

Materials and methods: Class I cavities (Bucco-Lingual: 4 mm, Occluso-Gingival: 3 mm, Mesio-Distal: 3 mm) were prepared in vertically half sectioned twenty-eight extracted human molars with their mesial side of cavity opened to sectioned surface. Cavities were restored with one of the following protocols. Group 1, two horizontal incremental layers of Filtek Z250 (Z250, 3 M ESPE) were cured with Elipar S10 (3 M ESPE); Group 2, two horizontal incremental layers of Filtek Bulk Fill Posterior (BFP, 3 M ESPE) were cured with Elipar S10; Group 3, A bulk filling of BFP was cured with Elipar S10; Group 4, A bulk filling of BFP was cured with BeLite (B&L Biotech) ($n = 7$). Each incremental layers or bulk filling were light cured for 20 s. Thermograms of the specimens during light curing were recorded using an infrared thermal camera (VarioCamhr head 700, InfraTec GmbH) at a rate of 2 frames/s for 100 s. Temperature changes on the composite and dentin were obtained.

Results: The highest temperature increases (ΔT_{max}) were 11.9 ± 1.2 °C (Group 1, 2nd layer), 12.8 ± 1.5 °C (Group 2, 2nd layer), 16.7 ± 1.3 °C (Group 3), and 14.1 ± 0.7 °C (Group 4) at 0.75 mm apical from the top of the cavity ($p < 0.05$). At the floor of the cavity, ΔT_{max} were 9.8 ± 1.2 °C (Group 1, 1st layer), 10.4 ± 0.9 °C (Group 2, 1st layer), 10.1 ± 1.8 °C (Group 3), and 8.8 ± 0.8 °C (Group 4) ($p > 0.05$). In the dentin 1 mm beneath cavity base, ΔT_{max} were 8.4 ± 1.6 °C (Group 1, 1st layer), 8.7 ± 1.4 °C (Group 2, 1st layer), 8.5 ± 1.1 °C (Group 3), and 6.8 ± 0.9 °C (Group 4) ($p > 0.05$).

Conclusions: Bulk filling generated more heat within the composite below the top of the cavity when compared to incremental filling. Heat generation of this region was irrespective of composite materials and the type of light curing units. Maximum temperature rises at the floor of the cavity and the dentin close to the pulp were not influenced by the type of composite materials, layering method, and light curing units.

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Monomer leaching and degree of conversion of bioactive dental composites

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Purpose/Aim: Recently, bioactive materials based dental composites have gained importance due to dual effect of bioactivity and better physical properties. In dental composites it is considered that unreacted monomer left inside the polymeric structure can be leached out and cause destructive effects on body tissues both locally and systemically. Therefore the aim of this study is to evaluate the elution of dental monomer and degree of conversion from experimental bioactive flowable and packable composites and compared with commercial bulkfil and experimental posterior composites.

Materials and methods: Experimental dimethacrylate based resin composites samples which were reinforced with nano-hydroxyapatite (nHA) fillers were prepared. Filler particles were surface treated with a silane coupling agent. Two experimental composites were prepared with concentration of 30% and 45% wt/wt of silanized nHA and considered as flowable and packable composites, where resin without filler was used as control. Two commercially available nano-hybrid bulkfill and micro-hybrid composite resin-based composites i.e. SDR (DENTSPLY, Germany) and Filtek P60 (3 M ESPE, Germany) respectively were also evaluated in this study. The experimental and commercial composites sample discs were prepared in 4mm × 4 mm molds by using standard method of photoactivation. Degree of conversion of these samples was evaluated by using ATR-FTIR (Thermo Nicolet P6700 USA) technique. Multivariate (Principal Component Analysis, PCA) and cluster analysis were performed to differentiate the variances. To evaluate the release of monomer and structural changes of polymerized samples were investigated by immersion in deionized water at 37 °C for periodic time intervals (i.e. 1, 7 and 28 days) and were assessed by HPLC (Schmadzu, Japan; with C-18 column) and FTIR respectively.

Results: Degree of conversion of experiment resins both 30 and 45%wt/wt showed highest conversion rate after polymerization and the sequence was Experiment resins (60%) > SDR (56%) > Filtek P60 (50%). HPLC results showed that both commercial and experimental composites released TEGDMA, UDMA and BisGMA monomer, where, maximum release was observed on day 1 which was subsequently reduced on day 7 and negligible release was observed on day 28. However, significant difference in elution of monomer between these resins based composites (RBCs) was observed. SDR showed significantly high elution of monomers compared to other composite

and structural changes were also observed more in SDR compared to others.

Conclusions: Release of monomer from dental composite after polymerization has potential to cause adverse effect on oral tissues. There is need to develop new dental composite with ideal properties and minimum release of monomer having appropriate interlocking of matrix chains.

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Discontinuous lamellar mesostructured hydroxyapatite formation in sodium dodecyl benzene sulfonate

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Purpose/Aim: Mesostructured hydroxyapatite (HA) is commonly used for the treatment of osseous diseases. The synthesis of mesostructured HA has been less successful as it is hindered by the very different interactions of the calcium and phosphate ions with the common surfactants used for the synthesis of silica mesoporous materials. This study investigated the precipitation of calcium phosphates in aqueous medium in the presence of ionic surfactant, Sodium Dodecyl Benzene Sulfonate (SDBS).

Materials and methods: $CaCl_2{\cdot}2H_2O$ (0.25 M) and $Na_2HPO_4{\cdot}2H_2O$ (0.25 M) solutions were used as calcium and phosphate sources (Ca/P molarity: 1). Synthesized bioceramics were observed at 5 mM to 90 mM of SDBS concentration. Ionic surfactant was assayed to template the calcium phosphate precipitation and the surfactant was dissolved in the $Na_2HPO_4{\cdot}2H_2O$ solution of 60 mM concentration. $CaCl_2{\cdot}2H_2O$ solution was slowly added at 40 °C and the condition was maintained for 1 h. The precipitated calcium phosphate was aged for 24 h at 37 °C, filtered, washed with water and dried at 90 °C for 24 h. The specimens prepared in 60 mM were calcinized at 450 °C to observe the nanostructure, porosity characteristics and phase changes. The obtained material was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy, thermogravimetric (TG) and thermodifferential (DTA) analysis, transmission electron microscopy (TEM) and electron diffraction (ED).

Results: Non-hydroxyapatite (mixture phase) was observed with ≤30 mM SDBS concentration, while hydroxyapatite with lamellar mesophase was observed in specimens prepared with ≥60 mM SDBS concentration, presenting ideal crystalline hydroxyapatite, lamellar mesophase. 5–30 mM SDSB resulted in mainly brushite crystalline. Low crystalline HA was evident for 45–90 mM SDSB. TG/DTA analysis indicated the highest total weight loss of SDSB for 30 mM and 90 mM (48%). In 15–90 mM SDSB concentrations, no Carbon was detected. TEM analysis showed discontinuous lamellar mesostructures of 2 nm of layer (Fig. 1a). The layer repetition did not exceed

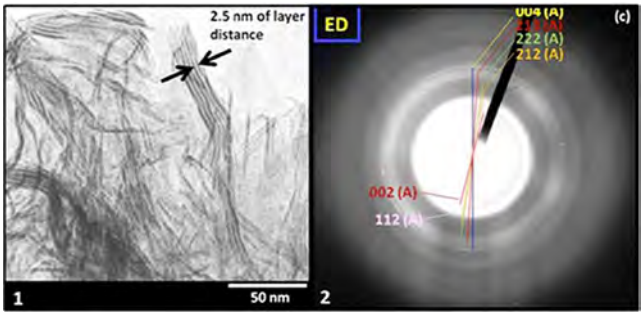

Fig. 1

five layers, presenting curved structure. HA formation was confirmed by ED pattern (Fig. 1b) as discontinuous lamellar mesophase. pH increased with the increase in surfactant concentration.

Conclusions: The presence of the ionic surfactant, SDBS, containing Sulphate in its polar head, could be useful to obtain HA in lamellar mesostructure.

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3D roughness of cranioplasty titanium implants following different surface treatments

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Purpose/Aim: Surface and mechanical properties of titanium alloys are integral for their use in restoring bone defects of skull and face regions (cranioplasty implants). This study aimed to investigate the effects of titanium protocols on the surface roughness of cranioplasty TiAl6V4.

Materials and methods: Seventy square shaped TiAl6V4 specimens (ASTM F68) were divided into seven groups of different surface treatment ($n = 10$). Control (G1); mechanical polishing (G2); specimens immersed in Nitric acid (G3); specimens sandblasted with aluminium oxide (50 μm) (G4); specimens treated using a combination of mechanical polishing and then acid treatment (G5); specimens treated using a combination of sandblasting and then acid treatment (G6); specimens were acid etched in solution of nitric and hydrofluoric acids then electro-chemically treated in solution of orthophosphoric and sulphuric acids (G7). 3D μ-Roughness values evaluated were Sq, Ssk, Sku, Sp, Sv, Sz, and Sa. Data was analyzed using one-way ANOVA and Dunett T3 post-hoc test ($p < 0.05$).

Results: Sq micro-roughness values (μm) were in the range of 2.81–16.68. It was the highest for control specimens ($p < 0.05$), and smoothest surfaces were achieved after following combined protocol of mechanical polishing and acid

treatment; or after electro-chemical treatment ($p < 0.05$). Sq values were reduced by at least a factor of 5 and were 3.27 and 2.81 μm respectively. Surfaces' kurtoses (SKu) were in acceptable range of 1.87–3.03.

Conclusions: Different finishing treatments have variable effect on cranioplasty titanium surface micro-roughness. Electro-chemical treatment produced the smoothest surface; hence it might be a good protocol to finish titanium implants.

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Effects of beverages on color stability of PICN material

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Purpose/Aim: Polymer infiltrated ceramic network (PICN) materials, which are also called hybrid ceramics, are new materials in dental market. The manufacturer of the PICN material used in the present study (VITA Enamic) suggests 3 different finishing procedures for this new material. It is known that dental materials subjected to beverages may have discoloration and color change. The aim of the present study was evaluation of the color stability of PICN materials that finished with different methods and immersed to the coffee and wine.

Materials and methods: 144 specimens were prepared with dimensions of 2 × 10 × 12 mm using by "high translucent and 2M2 shades" VITA Enamic hybrid ceramic blocks. The prepared specimens were divided into 4 groups ($n = 36$) randomly. Group 1: The samples that were not subjected to any polishing procedures (Control group); Group 2: Clinical Kit applied group; Group 3: Technical Kit applied group and Group 4: VITA Enamic Glaze applied group. Then, the samples were divided into subgroups ($n = 12$) according to immersion as Group A: water immersion, Group B: Coffee immersion and Group C: Red Wine immersion. Color change was measured by a clinical spectrophotometer before immersion and after 1, 7 and 28 d of immersion. Color change values were calculated using CIEDE2000 (ΔE00*). Results were statistically analyzed using Kruskal–Wallis and Mann–Whitney *U* test to represent the differences between groups, and Friedman and Wilcoxon tests were used to determine the changes in time ($p < .05$).

Results: According to the results of the present study, coffee and wine may cause color difference in time. The staining and discoloration of the material was also affected by the different polishing procedures. The highest color difference regarding ΔE00 was observed in Clinical Kit applied groups. Within 28 d, all ΔE00* values in the experimental group were significantly lower than the values of the control group ($p < 0.05$).

Conclusions: It was found that color of the PICN materials affected from coffee, wine and different polishing techniques. It may be suggested that finishing with technical kit for the least color change of the material used in the present study.

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Tooth substance removal for crown *versus* filling therapy

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Purpose/Aim: In some clinical situations it is difficult to decide whether to make a new filling or a crown. Prosthodontic treatment is an invasive, but sometimes necessary treatment to restore teeth that are severely damaged by caries or previous restorative treatment. Direct restorations are less invasive compared to crown treatment, but may not have as long time in clinical function as a crown. Previous studies on tooth substance removal for the two treatment options have, however, not taken into consideration the loss of tooth substance that is already present in these situations. Monolithic zirconia crowns have become available as a treatment that requires less tooth substance removal. The aim of this study was to assess the amount of tooth substance removed prior to treatment for monolithic zirconia crown therapy or composite fillings in teeth that are already severely damaged.

Materials and methods: Twenty-four extracted molars with damages simulating a large MOD amalgam preparation and two fractured cusps were paired and prepared for either a new composite filling or a monolithic zirconia crown. The amount of tooth substance removal was measured by weight loss in percent. The differences were assessed with paired t-test.

Results: There were statistically significant differences in the amount of tooth substance removal between the two groups ($p < 0.001$). The teeth prepared for a new filling had a substance loss of 6% (SD 1%) while the teeth prepared for crown therapy had 16% (SD 4%). The difference between the two treatments in a realistic clinical simulation is substantially less than described previously for intact teeth.

Conclusions: Crown therapy with modern materials, such as monolithic zirconia, can be considered as less invasive than previously assumed.

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pH-activatable nano-amorphous calcium phosphate to reduce dental enamel demineralization

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Purpose/Aim: Enamel demineralization is destructive, esthetically compromised, and costly complications of patients with orthodontic treatments. The use of orthodontic appliances may lead to accumulation of oral microorganisms causing enamel demineralization. To address this challenge, a novel class of orthodontic cement has been explored. Calcium phosphate (CaP)-based materials have outstanding bioactivity and biocompatibility.

Materials and methods: Here, nano-sized amorphous calcium phosphate (NACP) was prepared and hybridized with orthodontic cement to reduce enamel demineralization in a simulated caries model. NACP was synthesized by atomizing/spraying of a Ca/P suspension. NACP mass fraction was screened by shear bond strength. The enamel mineral content around the orthodontic brackets was investigated by cross-sectional microhardness.

Results: NACP was incorporated at 20 wt% into orthodontic cement. As a result, the 20% NACP-loaded ortho-cement notably exhibited favorable behavior on reducing demineralization of human enamel around brackets in an in a vitro PH-cycling model designed to simulate the carious attack. NACP incorporation markedly promoted high levels of calcium/phosphate ion release under an acidic environment (pH = 4). The 20%NACP-loaded ortho-cement reduced by twofold local (at 20 and 100 μm-distance) mineral loss near the enamel surface under conditions approximating the intra-oral environment, and decreased the carious lesion depth by approximately 1/3, compared to unmodified ortho-cement.

Conclusions: Nanoparticles of calcium phosphate could be engineered to avert enamel demineralization. The calcium phosphate nanotechnology holds high potential for a coadjuvant route in the prevention of dental caries in patients under orthodontic treatment.

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Evaluation of universal bonding agent containing MDP on zirconia bonding

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Purpose/Aim: To compare the evaluation of the universal bonding agents and their surface treatment methods, sandblasting and zirconia primer application, on Y-TZP zirconia bonding.

Materials and methods: 60 Y-TZP blocks (20 mm × 80 mm × 5 mm) were produced. Three universal bonding agents (Single Bond Universal (SBU), All-Bond Universal (ABU), GC-Premio Universa (GCU), one MDP containing self-etching agent (Clearfil SE bond (CSE)) and one total etching agent (Optibond FL (OFL)) were tested with/without sandblasting and zirconia primer (Z-Primer plus (ZP)). Bonding procedure of each group was performed according to the guidance of the manufacturer. Microshear bond test was carried out using a universal testing machine with cross-head speed of 1.0 mm/min. Peak load to fracture (N) was measured for each specimen. Data for bonding agent, z-primer, sandblasting were analyzed by 3-way ANOVA. Bonferroni Post-hoc analysis was performed for each z-primer, sandblasting, materials, materials & z-primer, and materials & sandblasting deemed statistically significant.

Results: As a result, the sandblasting and z-primer application increased the bond strength of the Y-TZP and resin composite ($p < 0.05$). There are significant differences between SBU and ABU, GCU, ZP, OFL group. Also, there is a significant difference between OFL and CSE group ($p < 0.001$).

Table 1 – Mean and standard deviation of microshear bond strength (N, mean ± SD) (n = 15 for each condition).

Bonding agent	Z-primer		No sandblast	Sandblast	Total mean of bonding agents (n = 15 or 30)
Z Primer only			28.98 ± 11.03	32.77 ± 8.52	30.88 ± 9.87^{BC}
Optibond FL	No		22.18 ± 6.66	27.19 ± 8.64	28.67 ± 8.43^{C}
	Yes		30.85 ± 6.93	34.47 ± 6.65	
Clearfil SE Bond	No		25.03 ± 8.31	30.86 ± 10.75	33.35 ± 9.42^{AB}
	Yes		38.61 ± 5.00	38.88 ± 4.61	
Single Bond Universal	No		37.95 ± 7.70	36.38 ± 5.32	37.43 ± 6.65^{A}
	Yes		38.39 ± 6.87	36.99 ± 6.99	
All-Bond Universal	No		32.84 ± 4.68	30.43 ± 5.65	32.34 ± 6.17^{BC}
	Yes		34.00 ± 7.10	32.11 ± 6.99	
G-Premio Universal	No		23.59 ± 4.40	29.26 ± 7.36	29.92 ± 7.48^{BC}
	Yes		31.68 ± 5.27	35.12 ± 7.67	
Total mean of Z-Primer	No	29.57 ± 8.58*	Total mean of sandblasting		
	Yes	34.40 ± 7.62**	31.28 ± 8.78$^{?}$	33.13 ± 7.93$^{??}$	

Conclusions: Generally sandblasting and Z-primer application can increase bond strength between Y-TZP and composite resin. However, SBU showed superior bond strength than other universal bonding agents with or without sandblasting and Z-primer, and it can simplify the clinical procedure.

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Service simulation and reliability of multilayer ceramic structures

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Purpose/Aim: The study evaluated the reliability of monolithic and multilayer ceramic structures and the critical crack produced by loading such structures bonded to a dentin analogue (G10-fiber-reinforced epoxi resin).

Materials and methods: Disc-shaped specimens were fabricated according to the manufacturer's recommendations (Ivoclar) with one of the following structural design ($n = 30$): - CAD-on (IPS e.max® ZirCAD + Crystall/Connect + IPS e.max® CAD); - YLD (IPS e.max® ZirCAD + IPS e.max® Ceram); - LDC monolithic (IPS e.max® CAD); - YZW monolithic (Zenostar Zr). The ceramic structures were bonded (Multilink, Ivoclar) to G10 supporting bases and submitted to compressive load (0.5 mm/min) under 37 °C distilled water using an universal testing machine until the sound of the initial crack that was detected using an acoustic system (Audacity Sound Editor). Maximum loads (in N) to the initial crack were recorded and statistically analyzed using Weibull distribution, Kruskal–Wallis and Student–Newman–Keuls tests ($\alpha = 0.05$). Failures were evaluated using fractography principles and transillumination.

Results: There was no significant difference between CAD-on and YZW groups ($p = 0.917$), which showed greater load values than YLD and LDC groups ($p < 0.01$). There was no statistical difference for the Weibull moduli (m) of all groups. Radial crack was the failure origin of all monolithic structures (LDC and YZW). Multilayer structures (CAD-on and YLD) showed, predominantly, cone + radial cracks as the failure origin. The study design (ceramic structures bonded to dentin analogue + testing under 37 °C distilled water + acoustic detection of initial crack + fractography using transillumination) brought service and results closer to the ones clinically reported, showing the positive potential of the used methodology.

Conclusions: Monolithic zirconia (YZW) and trilayer structure (CAD-on) showed similar loads to failure but different failure behavior, which are similar to clinical reports.

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Strength of 3Y-TZP depending on grain size and LTD

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Purpose/Aim: The sintering temperature influences the final grain size in 3Y-TZP materials, which in turn has an effect on quasi-static mechanical properties and the potential for martensitic transformation. This study aimed to evaluate the influence of the grain size and LTD on the strength of a 3Y-TZP material.

Materials and methods: Oversized bars were cut from pre-sintered 3Y-TZP blocks (e.max ZirCAD, Ivoclar-Vivadent) and sintered for 2 h at 1450 °C or 1650 °C ($n = 180$). After sintering final dimensions (height × thickness × length) were 2 × 2.5 × 25 mm. One third of the specimens from each sintering temperature were subjected to an accelerated low-temperature degradation (LTD) process via autoclave sterilization at 134 °C under 3 bar pressure for 0 h, 5 h or 20 h (hereafter designated "aged"). The specimens were dried in an oven at 150 °C for 2 h and immersed in silicon oil prior to testing. Specimens were then fractured under 4-point bending and analyzed using 2-parameter Weibull statistics (shape parameter m and scale parameter s_0). LTD was measured on the surface of specimens using Raman Spectroscopy (Gaussian-Lorenzian fits).

Results: Grain sizes scaled with sintering temperature. The amount of transformation increased with aging time and was higher for specimens with larger grains. The strength of 3Y-TZP was significantly influenced by the sintering temperature, with specimens sintered at 1450 °C showing higher strength values and lower scatter. LTD did not affect the strength or the reliability of 3Y-TZP specimens sintered at 1450 °C, but showed a significant increase in these parameters for the group 1650 °C/20 h. Weibull parameters for the non-aged groups sintered at 1450 °C and 1650 °C were, respectively: $m = 12.2$, $s_0 = 1192$ MPa and $m = 5.7$, $s_0 = 967$ MPa. For the aged groups: 5 h: $m = 14.6$, $s_0 = 1226$ MPa and $m = 6.2$, $s_0 = 1003$ MPa; 20 h: $m = 18.3$, $s_0 = 1153$ MPa and $m = 16.5$, $s_0 = 1094$ MPa.

Conclusions: High sintering temperatures decrease the strength and increase the scatter of flaw sizes in 3Y-TZP, while LTD simulated in an autoclave for 20 h seems to improve the strength and scatter of specimens having larger grain sizes.

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Hydrothermal degradation of monolithic zirconia: Guidelines for finishing treatments

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Purpose/Aim: Full-contour monolithic Y-TZP restorations gained popularity due to the decreased potential for chipping and reduced wall thickness. Clinical adjustment procedures are often necessary chairside to achieve balanced occlusal contacts. These procedures can modify the near-surface region of Y-TZP and possibly affect its resistance to hydrothermal degradation (aging). The present in-vitro study aimed at investigating the aging behavior of zirconia copings made of different commercial materials and shades, with respect to finishing procedures such as grinding, polishing and regeneration, performed according to clinical practice. The proposed guidelines could help dentists and dental technicians on how to treat zirconia surface in order to maximize the restorations lifespan.

Materials and methods: 18 copings of Y-TZP made up of 3 different materials (NobelProcera Nobel Biocare, Lava Plus 3 M ESPE, Lava Classic 3 M ESPE) were produced with the same flat occlusal surface and divided into 9 groups made up of 2 copings each. The microstructure was characterized by scanning electron microscopy (SEM). Each coping was divided into 3 regions, respectively ground (G), ground-and-polished (P) and control (C), using diamond burs and diamond silicone polishers for ceramics. Half of the samples of each group were regenerated at 1200 °C (i.e. annealing) and all the copings were subjected to accelerated artificial aging for 2, 6, 18, 54 h in autoclave (134 °C at 2 bar). At each time interval, X-ray diffraction (XRD) patterns were recorded from the three experimental regions to measure the surface monoclinic content (Vm) and evaluate the aging behavior. The polished surfaces were observed by differential interference contrast optical microscopy.

After the last aging treatment, the sub-surface regions of selected copings were investigated by focused ion beam (FIB) coupled with SEM.

Results: Significant differences were found in terms of grain size between the different tested materials. Vm at 18 h aging time was lower for smaller grain size, while the aging process was delayed in regenerated materials. Although no significant differences in Vm were recorded by XRD on the different analyzed surface regions, FIB analysis revealed significantly different subsurface damage.

Conclusions: Commercial zirconia with small grain size can be recommended to hinder the hydrothermal degradation, as well as a regeneration process after the last superficial treatment. Control, ground and polished surfaces showed similar phase characteristics and behavior.

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Biomimetic remineralizing agents influence artificial caries-affected dentin surface properties

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Purpose/Aim: Wettability is considered a prerequisite for adhesion, and the type of substrate plays an important role in this property. Therefore, biomimetic remineralizing agents seems to be an interesting approach, since it can restructure and increase physical properties of caries-affected dentin, a substrate commonly preserved in restorative procedures. This study aims to evaluate the effect of 0.2% NaF-NF, MI Paste^{TM}-MP and Curodont^{TM} Repair-CR on artificial caries-affected dentin surface, concerning wettability-WT and changes in inorganic components of hybrid layer by energy dispersive X-ray spectroscopy-EDS. The hypothesis tested is that artificial caries-affected dentin surface remineralization can improve the physical-chemical properties of hybrid layer.

Materials and methods: Fifty-five flat dentin slices were obtained from 55 sound third molars and randomly distributed into five groups according to remineralizing agents (NF, MP and CR) and control groups ($n = 8$): G1 – sound dentin-SD; G2 – demineralized dentin-DD; G3 – DD + 0.2% NF; G4 – DD + MP; and G5 – DD + CR. Forty-four slices were submitted to caries-like lesion by biological model (*S. mutans* biofilm). Then, caries-infected dentin was removed with 600-grit silicon carbide paper until get the caries-affected dentin. Remineralizing agents were applied, and surfaces were tested for wettability-(contact angles) measured using a goniometer. For the EDS test, 2 mm-high composite resin blocks (Filtek Z350XT) were bonded (Adper Single Bond 2) to the treated and non-treated surfaces. Bonded interface zones were evaluated by SEM, Calcium (Ca), Phosphorus (P) contents and the Ca/P ratio and were analyzed by EDS analyzed. Data were submitted to ANOVA/Tukey and Dunnet tests ($\alpha = 0.05$).

Results: G2 (60.30 ± 5.80) and G3 (53.68 ± 6.47) showed the highest contact angles values, differing significantly from the other groups ($p < 0.01$), while G4 (30.15 ± 6.99) presented the lowest contact angle. G5 (42.79 ± 8.28) exhibited contact angle value similar to G1 (40.60 ± 3.77) ($p > 0.05$), which were significantly higher than G4 ($p < 0.05$). The highest and lowest Ca amount was observed on G5 (51.33 ± 4.39) and G2 (16.77 ± 2.95)/G4 (21.43 ± 5.43) and intermediary values for G3 (29.13 ± 4.27) ($p < 0.05$). The highest and lowest P amount was found for G5 (21.90 ± 1.56) and G4 (9.03 ± 2.62); intermediary values were found on G2 (11.06 ± 3.31)/G3 (12.65 ± 1.58) ($p < 0.05$). For Ca/P ratio, there was no statistically significant difference between G3 (2.30 ± 0.13), G4 (2.44 ± 0.41) and G5 (2.34 ± 0.10) ($p > 0.05$). SD showed the highest amount of Ca, P, and Ca/P ratio (Dunnett test $p < 0.05$), but there was no significant difference between P amount of G5and the Ca/P ratio than G4 ($p > 0.05$).

Conclusions: MP and CR were able to increase the superficial properties of demineralized dentin.

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In vitro analysis of *Rhus coriaria* extract interaction with demineralized dentin matrix

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Purpose/Aim: Pretreatment of demineralized dentin matrices (DDM) with *Rhus coriaria* extract (RCE) results in the reduction in enzymatic degradation and improved biostability of dentin over time. This study investigated the interaction of RCE with type I collagen in the DDM by attenuated total reflectance Fourier transform-infrared spectroscopy (ATR-FTIR) and micro-Raman spectroscopy.

Materials and methods: Dentin discs (0.5 mm) were prepared from mid-coronal dentin and etched with 37% phosphoric acid (PA) for 30 s. After rinsing, dentin discs were treated with 10 wt%, 15 wt% or 20 wt% SBE solution for 30 s ($n = 5$). Non-treatment groups served as control. Dentin surfaces were analyzed using ATR-FTIR using single-beam with 4 cm^{-1} resolution with 140 scans. To assess the effect of the RCE modified PA on dentin collagen and demineralization depth, mineralized dentin discs were etched with 37% PA modified with 20 wt%, 15 wt% or 10 wt% RCE. Both ATR-FTIR and Raman spectra were collected from the etched-dentin surfaces for each sample ($n = 5$) with three replicates. The relative intensity of the peaks associated with the mineral (PO_4^{3-} at 960 cm^{-1}) was recorded to identify the depth of demineralization by using a computer-controlled laser Raman microprobe (Renishaw InVia; Renishaw PLC) connected to an optical microscope with a 100× objective. Data were analyzed with ANOVA at $\alpha = 0.05$ followed by Tukey's comparison.

Results: The FTIR spectroscopy revealed high interaction between dentin collagen matrix and RCE both as a pre-treatment or in combination with PA with all experimental concentrations compared to untreated samples ($p < 0.05$). The presence of RCE significantly decreased the ratio of amide III/CH_2 bending (A1235/A1450) peak area compared non-RCE control group ($p < 0.05$). Similarly, RCE-modified PA showed significantly lower A1235/A1450 ratio compared to RCE pre-treatment only. Micro-Raman analysis demonstrated that the etching depth of dentin slabs was similar for experimental groups and PA ($p > 0.05$). The presence of RCE did not affect the demineralization depth on dentin.

Conclusions: This study confirmed that RCE treatment of dentin matrix can increase the intra and interfibrillar crosslinking density of collagen matrix. The use of RCE both as a pre-treatment agent or incorporation in etching agent is effective for clinical use.

A1235/A1450	Control	S10	S15	S20
SBE Treatment	0.972 ± 0.19^{a}	0.176 ± 0.05^{c}	0.164 ± 0.04^{c}	0.161 ± 0.05^{c}
SBE in 37% PA		0.463 ± 0.01^{b}	0.522 ± 0.1^{b}	0.526 ± 0.05^{b}

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Fractographic analysis of fractured lava ultimate crowns over zirconia abutments

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Purpose/Aim: The one-year outcome of a clinical trial of adhesively fixed Lava Ultimate (3 M ESPE) CAD/CAM crowns onto zirconia implant abutments (ZirDesign/Atlantis, Dentsply) has shown an unacceptable high failure rate due to debondings (80%) or fractures (6%). The aim of this study was to fractographically examine the retrieved crown fragments (three crowns) and to conclude on reasons for failure.

Materials and methods: The complete history and processing parameters were previously documented in a prospective clinical trial (Schepke et al., Clin Implant Dent Relat Res, 2015). Patients in the three cases reported that a debonding happened prior to the fracture. Fractographic techniques using light and scanning electron microscopy were used to trace back failure origins and to describe the respective failure patterns on three fractured premolar Lava Ultimate crowns (labeled K09, K10, and K17). Profilometry, Raman spectroscopy, and EDX analysis were performed on the zirconia abutments.

Results: All three crowns showed a brittle fracture plane in mesio-distal direction. The failure origins were identified on the disto-occlusal side (K09), mesio-marginal side (K10) and for K17 most probably on the disto-occlusal side, all diagonally located to the compression curl, indicative for an off-axis loading. The adhesive and cement remnants were completely detached either from the crown surfaces but to a significantly greater amount from the zirconia abutments.

Conclusions: The fracture of the crowns was most probably a secondary event subsequently happing after delamination, lift-off/twisting from the abutment and out-of-axis loading from mastication. The actual reasons for failure are identified as an interfacial bonding problem between the adhesive and the zirconia abutment.

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WITHDRAWN

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Unbound monomers do diffuse through the dentin barrier

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Purpose/Aim: The aim of this study was to assess the role of dentinal fluid proteins in trans-dentinal diffusion of free monomers.

Materials and methods: A .25 μL artificial pulp chamber (APC) topped with .5 mm thick human dentin disks was used. A simplified two-step etch-and-rinse adhesive was formulated with 2-hydroethyl-methacrylate (HEMA 30%m), bisphenol-A-diglycidyl-methacrylate (BisGMA 70%m), using Camphorquinone/tertiary amine as initiators. Two extraction media were used: buffered saline (control $n=10$), buffered saline with 1% bovine serum albumin (BSA $n=10$). Samples were etched for (37% H3PO4, 15 s), rinsed (water, 20 s), air dried (<5 s), then used to close the APC. Simplified primer was used (absolute ethanol, 60 s), adhesive applied, brushed (15 s), air thinned and dried (15 s) then light cured (20 s) with a LED curing unit (irradiance > 1000 mW cm^{-2}). Monomer diffusion was assessed by reverse phase HPLC (C18 column, particle size 2.4 μm, length 150 mm, diameter 4.6 mm), 70% acetonitrile/30% water isocratic mobile phase, UV detection at 205 nm. Monomer concentration in extraction media was calculated against calibration curves and statistically analyzed (Mann–Whitney, $p<0.05$).

Results: Quantifiable amounts of HEMA were detected in both extraction media (control: 663 μmol/L, BSA: 1625 μmol/L) while BisGMA was present in quantifiable amounts in BSA medium only (75 μmol/L). Diffused monomers concentrations were significantly different for both monomers ($p<0.001$ BisGMA, $p<0.01$ HEMA)

Conclusions: Albumin is sometimes referred to as taxi protein for its ability to bind and transport hydrophobic ligands. From our results, it can be hypothesized that albumin can also transport unbound monomers released from dental adhesive through the dentin barrier. Elution is dependent upon chemical characteristics of leachable species (hydrophobicity) and extraction media. However, dentinal fluid proteins like albumins could have significant effect on monomer diffusion through dentin to the dental pulp transporting highly hydrophobic molecules like BisGMA alongside passively transported more hydrophilic ones like HEMA.

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Oral environment simulation affects ceramic failure behavior

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Purpose/Aim: To investigate the failure behavior of a leucite-reinforced glass-ceramic subjected to a monotonic compressive test and a fatigue test, both simulating oral environment conditions.

Materials and methods: Fifty-three ceramic slices (1.5 × 8.3 × 8.4 mm) were produced by cutting CAD-CAM blocks and cemented with resin cement over dentin analogue substrate (fiber-reinforced epoxi resin). For the monotonic test ($n=23$), a gradual compressive load (0.5 mm/min) was applied by a piston to the center of the specimen immersed in 37 °C water, using a universal testing machine. The initial crack was detected using an acoustic device. The fatigue test was performed in a mechanical cycling machine (37 °C water, 2 Hz) using the boundary technique ($n=30$). Two lifetimes were evaluated (1 million and 2 million cycles). Failure analysis was performed with transillumination. Monotonic fracture load data were evaluated with Weibull analysis. A cumulative damage model with an inverse power law (IPL) lifetime-stress relation was used to fit the fatigue data.

Results: Characteristic failure load of 1615 N and Weibull modulus of 5 were obtained with the monotonic test. The estimated probability of failure (Pf) for 1 million cycles at 100 N, 150 N and 200 N were 31%, 55% and 75%, respectively. For 2 million cycles, the Pf increased 20%, which was not significant. The most frequent failure mode was radial crack. For fatigue, combined failure modes were also found (radial crack combined with cone crack or chipping).

Conclusions: Fatigue affects the fracture load and failure mode of leucite-reinforced glass-ceramic. The methodology used successfully reproduced clinical failure modes.

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Effect of pine bark proanthocyanidins on dentin and resin–dentin interfaces

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Purpose/Aim: This study tested the hypothesis that oligomeric proanthocyanidins (OPACs) from *Pinus massoniana* can induce stable cross-links in the dentin matrix and also function as a bioadhesive for reparative and restorative dentistry.

Materials and methods: Two fractions were prepared, polymer-knockout (poly-KO) and monomers and polymers knockout (mono-poly-KO) from crude source of pine park from

Pinus massoniana. Twenty sound molars were cut into dentin beams, which were fully demineralized with 10% phosphoric acid and divided into 4 groups according to the biomodification solution: pine, poly-KO, mono-poly-KO and control. The modulus of elasticity was obtained on a 3-point bending test at baseline, immediately and after 6 months storage in simulated body fluid (SBF). Twenty-eight teeth were ground flat to expose mid-coronal dentin, etched for 15 s, rinsed, the respective priming solution applied for 1 min and restored with an HEMA-free adhesive. Specimens were cut into 0.8 × 0.8 mm thick resin–dentin beams and tested using standard microtensile bond strength (TBS). Two beams from each specimen were embedded in epoxy resin, polished, infiltrated with 0.1% Rhodamine-B for 1 hour, rinsed and evaluated on a confocal laser scanning microscope (CLSM). Data were statistically analyzed by two-way ANOVA and Tukey's post hoc test ($\alpha = 0.05$).

Results: No differences in E were observed between pine crude, poly-KO and mono-poly-KO ($p > 0.05$); all groups were statistically higher than control ($p < 0.001$). The experimental fractions resulted in statistically similar TBS ($p > 0.05$). E and TBS results were stable over time for all groups ($p > 0.05$). Treatment with pine and fractions significantly decreases the permeability at the resin–dentin interface ($p < 0.001$).

Conclusions: The enriched mixtures of pine presented similar increase in E when compared to the crude extract. Remarkable and stable increase in the bond strength was observed for the enriched mixtures and pine crude extract. Collagen fluorescence was significantly higher in the OPACs treated dentin, whereas permeability of the interface was significantly reduced.

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Experimental ZnO cement containing bioactive niobium biophosphate fillers

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Purpose/Aim: To develop a cement based on zinc oxide, ethoxybenzoic acid and niobium phosphate glass (NbG) with bioactive properties for dentistry use.

Materials and methods: Zinc oxide (ZnO), zinc phosphate monohydrate (Zn_2SO_4) and ethoxy-benzoic acid (C9H10O3) were used to obtaining the matrix, where the NbG was added in different concentration. The groups were: Group 1 – without NBG; Group 2 – 5% NBG; Group 3 – 10% NBG; Group 4 – 20% NBG; Group 5 – 30% NBG; Group 6 – 40% NBG. Elemental analysis was assessed through energy dispersive spectroscopy (EDS) attached to the scanning electron microscope. Characterization of interaction between the components was performed by ATR-FTIR, in reflectance mode with a 4 cm^{-1} resolution in the 1800–500 cm^{-1}. The crystalline phases resulting after reagents mixed was performed by XRD, operated with Cu Kα radiation ($\lambda = 0.15418$ nm). The diffraction patterns were obtained in the 2θ range from 10° to 90° in continuous mode at 0.02°/min.

Results: EDS showed calcium, phosphorous and niobium elements, except for control group and 5%. These elements were observed from the 10% niobium biophosphate group (Nb K 3.56%; P K 2.84% and Ca K 1.19%), the highest concentrations founded in 20% NBG samples (Nb K 7.69%; P K 5.31% and Ca K 3.57%) (Fig. 1). The FTIR spectra demonstrate the 1673, 1110, 754 cm^{-1} peaks: zinc-oxygen bond (matrix). The 921, 851, 641 cm^{-1} peaks express the niobium and oxygen bonds. According to the X-ray diffractograms the crystal Zincite was present in all experimental groups, tetracalcium dipotassium decaphosphate was observed in 5%, 10% and 20% niobium biophosphate groups, sodium potassium niobate was verified at 30% and 40% NBG groups, and Calcite in 40%.

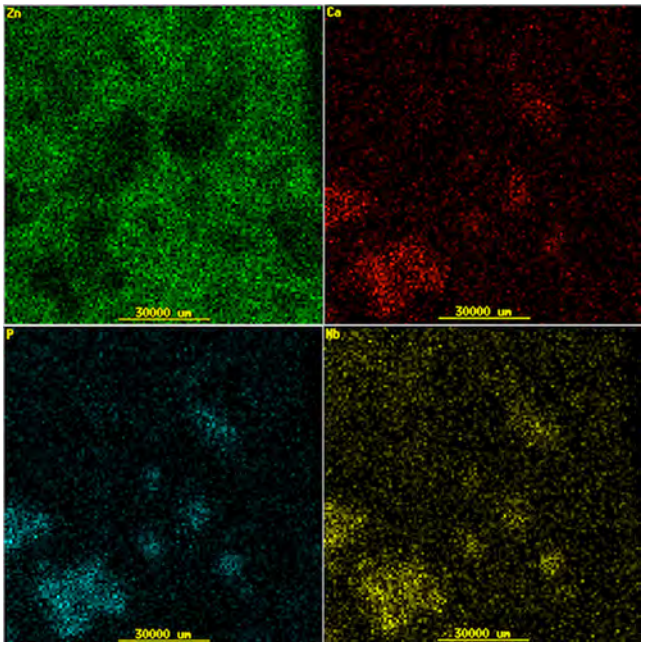

Fig. 1

Conclusions: The addition of NbG does not interfere with the chemical structure of a zinc oxide matrix. However, only at 40% concentration was observed Calcium carbonate.

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Influence of acid saliva on wear of human dentin

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Purpose/Aim: The literature reports tooth-brushing and acid association which an important etiologic factor for cervical dentin hypersensitivity. The hypothesis of this study is that acid saliva improves the mechanical-chemical degradation during brushing. The purpose of this study was to evaluate the effect of 05 types of saliva (S) with different pH on human cervical dentin by Tribology test.

Materials and methods: Twenty-four sound extracted human third molars were selected. The teeth were thawed, cleaned of adhering soft tissues and the approximately 3 × 3 mm buccal cervical dentin of each molars were sectioned mesio-distally with a slow speed diamond blade (Buehler-Series 15LC Diamond, USA) under constant water irrigation. The samples were immediately involved by Epoxic resin (Buehler, USA) and divided into 6 groups ($n = 4$) according to PH of artificial saliva: S1.5 (pH = 1.5); S3 (pH = 3.0); S4.5 (pH = 4.5); S6 (pH = 6.0); S7.5 (pH = 7.5) and DDW (distilled and deionized water, control group). All samples were gridded with 320 and 600 grit silicon carbide paper (Buehler) exactly before the Tribology test. A 3/8″nylon6-6 ball applying a static 8.5N-normal load was used. This test was conducted 4000 cycles with 1 Hz frequency and 4.0 mm of displacement, simulating the contact of toothbrush with exposed buccal cervical dentin. The scars were created and coefficient to friction (CF) was obtained. After test all samples were analyzed by white light interferometry microscopy (Zygo New View 6300, USA) to measurement the roughness (Ra), volume loss (VL) and pattern of scars. All data were analyzed by ANOVA test one-way and Tukey test ($\alpha = 0.05$).

Results: There was a significant influence of acid pH on CF, Ra and VL values. CF: S1.5(0.18 ± 0.05)A; S3(0.22 ± 0.08)A; S4.5(0.37 ± 0.11)B; S6(0.41 ± 0.17)B; S7.5(0.40 ± 0.09)B; DDW(0.45 ± 0.12)B. Ra(μm): S1.5(29.8 ± 7.46)A; S3(16.6 ± 2.91)A; S4.5(2.65 ± 0.99)B; DDW(1.12 ± 0.16)C; S6(0.96 ± 0.12)C; S7.5(0.53 ± 0.15)C. VL(?m3 × 107): S1.5(10.1 ± 2.70)A; S3(6.3 ± 2.27)A; S4.5(1.27 ± 0.48)B; DDW(0.74 ± 0.11)C; S6(0.54 ± 0.26)C; S7.5(0.52 ± 0.11)C. The analysis of images demonstrated high difference on scars pattern between groups. This study improved the tribology test because employed sphere with same nylon type of toothbrush and showed the acid saliva-friction association to dental degradation.

Conclusions: On limits of this study, we concluded that the association of acid saliva promotes chemical degradation of dentin improving the mechanical wear by nylon-dentin friction.

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Glass ionomer cements: Liquid dynamics in confinement

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Purpose/Aim: During setting of conventional glass ionomer cements, hydrogen ions become bound to the cement structure as the inorganic network develops. The aim of this study was to follow the liquid dynamics in confinement within restorative glass ionomer cements.

Materials and methods: Thermogravimetric analysis (TGA) combined with Fourier transformed infrared spectrometry (FTIR) from encapsulated restorative glass ionomer cements (GC Equia Forte, GC Fuji IX GP, VOCO Ionofil Molar AC, 3 M ESPE Ketac Molar Aplicap) and their respective powders were obtained. Differential scanning calorimetry (DSC) measurements from the cements as well as their respective powders and liquids were acquired. Cement samples were prepared in triplicates and stored air-tight for 5 or 28 days before the measurements.

Results: From the TGA-FTIR, it was noted that the liquid loss from cement samples occurred at two stages: (1) around 100 °C (from the unbound water) and (2) between 300 and 550 °C (from the bound liquid). The amount of unbound water differed, whilst losses resulting from bound liquid were less obvious among the investigated cements. DCS measurements in heating showed decomposition of the acid from cement samples to occur at distinct temperatures. During cooling, differences in the liquids were evidenced by distinct crystallization temperatures, being the liquid of Ketac Molar with the most distinctive behavior. This is in agreement with the decomposition curve from the respective cement samples on heating.

Conclusions: Marked differences in the liquid dynamics were observed by DSC analysis, while TGA-FTIR indicated varied content of unbound water in the investigated cements.

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Fatigue limit of monolithic zirconia FDP: Damage and glass infiltration

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Purpose/Aim: The aim of this study was to evaluate the effect of damage with diamond bur and glass infiltration by two different methods on the fatigue limit of 3-unit fixed dental prosthesis (FDP) in monolithic zirconia.

Materials and methods: 92 FDP were milled from pre sintered blocs in a CAD CAM system, based on the scanning of simplified NG10 abutments. Groups tested were: (1) control; (2) abraded with diamond bur on the gingival area of the connector; (3) glass infiltrated by sol-gel method; (4) graded material (glass-zirconia-glass). Three samples from each group were tested in load-to-fracture test (30° inclination), and 20 samples from each group were tested in fatigue (stair case method, 100K cycles). Results were evaluated by Collins' method.

Results: The fatigue limit was similar to control and abraded groups (1335.36 N and 1448.56 N, respectively), and both were lower than fatigue limit of glass infiltrated groups: 1833.42 N for the sol-gel method and 2264.85 for the graded material. Samples infiltrated by sol-gel method presented values with lower standard deviation. Fractographic analysis showed failure origin located at the vestibular region of the connector, and at the load-application point (occlusal of the

pontic). A finite element analysis confirmed that the higher stress concentration was at the vestibular region of the connector.

Conclusions: Fatigue limit of FDP in monolithic zirconia was not affected by abrasion with diamond bur and were enhanced by glass infiltration

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Does zirconia translucency affect bond strength of resin-based cements?

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Purpose/Aim: To evaluate if yttria-stabilized tetragonal zirconia polycrystals of different translucency and thickness affect the bond strength of two resin-based cements irradiated with different curing protocols. The null hypothesis was that the bond strength to Y-TZP was not influenced by translucency, thickness, and energy density during light-curing.

Materials and methods: 4 types of Y-TZP of different translucency were selected (Noritake Katana UTML, Noritake Katana STML, Noritake Katana ML, Amann Ghirrbach Ceramill Zi White), cut in order to obtain 1 mm and 2 mm disks ($n = 36$ per thickness) and sintered. The disks were air-abraded with 46 μ Al_2O_3 and divided in two groups according to the luting cement (Rely X Ultimate or Panavia V5) employed to lute 2 mm diameter composite cylinders to Y-TZP surface according to the manufacturer instructions. Resin-based cements were light-cured with 2 different lamps (Led and Halogen) and with different energy densities (0 J (no light), 14 J and 42 J) by placing the light-curing tip in contact to the opposite surface of each disk. Bond strength was tested with shear-bond strength test before and after 10,000 thermocycles and 30 days of water storage. The fracture pattern was evaluated with an optical microscope. Results were statistically analyzed with a 3-way ANOVA test ($p < 0.05$).

Results: 3-way ANOVA test showed that the bond strength was not influenced by the translucency of the Y-TZP, but was significantly affected by its thickness ($p < 0.01$) and by the curing procedure ($p < 0.001$). No differences were highlighted between curing lights employed.

Conclusions: The null hypothesis was partially accepted since the bond strength to zirconia mainly depended to thickness and curing procedure. Different cements seem to not influence the bond strength.

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Surface finishing does not affect catastrophic failure of glass-ceramic crowns

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Purpose/Aim: Ceramic restorations often require adjustments using diamond burs and abrasive points, which damage the glazed surface. These adjustments are necessary when the restoration presents heavy occlusal contacts or inadequate contours. The aim of this study was to evaluate the influence of induced surface defects on the fracture load and the mode of failure of lithium disilicate based (LDS) glass-ceramic restorations.

Materials and methods: Pre-molar crowns were obtained from LDS CAD/CAM blocks ($n = 60$) and were glazed. The crowns were cemented on dentin analogue dyes (fiber-reinforced epoxi resin), and divided into 5 groups for occlusal surface finishing ($n = 12$): G – Glaze. D – abrasion (diamond bur 2135); DG – abrasion and reglaze; DP – abrasion and polishing (diamond bur 2135F, 2135 FF and abrasive points); P – Polishing. Crowns topography was qualitatively observed by SEM images and quantitatively evaluated using roughness parameters (Ra, Rz and Rq). A gradual compressive load (0.5 mm/min) was applied by a piston to the center of the palatal cusp until fracture. The fracture load was recorded and the groups were compared by ANOVA and Student–Newman–Keuls. Fractured crowns were examined under light microscopy (×40) and SEM to determine the fracture origin.

Results: Surface polishing and/or reglazing resulted in lower roughness compared to group D ($p < 0.05$), but did not influenced the fracture loads ($p = 0.696$). Catastrophic failure with origin at the interface between ceramic and resin cement was the mode of failure for all groups.

Conclusions: Reglazing or polishing were effective in reducing surface defects, promoting a smoother surface and higher fracture loads. Therefore, these surface treatments are recommended after adjustments using diamond burs. The catastrophic failure of LDS crows was not influenced by the surface finishing in the clinically meaningful in-vitro scenario.

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Bond strength evaluation of a novel hydrophilic adhesive system

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Purpose/Aim: The aim of this in vitro study was to compare conventional adhesive systems and universal adhesives with a novel hydrophilic adhesive system. The null hypothesis is that there is no difference between adhesive systems tested.

Materials and methods: Sixty-four freshly extracted, non-carious, human third molars were collected for this study and divided into five groups ($n = 16$ per group) according to adhesive system employed. Universal adhesives were employed both with a etch&rinse (8 teeth) and etch&dry (8 teeth) techniques. Group 1: experimental hydrophilic adhesive (Kuraray, Japan). Group 2: Adhese Universal (Ivoclar Vivadent, Luxembourg). Group 3: Scotchbond Universal (3 M, USA). Group 4 ($n = 16$) was divided into 2 sub-groups ($n = 8$): OptiBond FL (Kerr, USA) and Clearfil SE BOND 2 (Kuraray Noritake Dental, Japan). Every adhesive was applied on dentin according to manufacturer's instructions. After light curing, the restoration was completed with nano-hybrid resin composite (Clearfil ES-2; Kuraray), which was placed in 2 mm horizontal layers and light-cured individually for 20 s. After 7 days, samples were submitted to microtensile bond strength test. In sixteen additional teeth ($n = 4$/group) the restoration was performed with flowable composite (ES-2 Flow, Kuraray) to perform interfacial nanoleakage evaluation. Data were statistically analyzed with 2-way ANOVA test and significance was set for $p < 0.05$.

Results: ANOVA test showed any significant statistical difference among the groups in terms of adhesive system employed. However, etch&rinse technique showed significantly higher bond strength than etch&dry technique on dentin.

Conclusions: The null hypothesis is partially accepted since the adhesive tested showed similar bond strength, even if etch&rinse techniques were better than etch&dry.

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Curing effectiveness of multiple resin composites with LED curing units

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Purpose/Aim: To evaluate the curing effectiveness of 4 mm-composite layers cured by monowave (MW) or polywave (PW) LED light-curing units (LCU).

Materials and methods: Cylindrical, 4 mm height samples were produced from 5 resin composites using silicone molds. They were light-cured for 10 s or 20 s using a monowave (ACTEON) or a polywave (Ivoclar-Vivadent) LCUs. The Degree of Conversion (DC) at the light-exposed surface (top) and opposite surface (bottom) was determined by FTIR (Shimadzu) using the DC formula. Data were normalized assuming the DC at the top being 100% and the DC at the bottom expressed as a percent of the top. Effective curing assumes bottom values being >80% of the top. Data were analyzed by two-way ANOVA and Tukey tests ($\alpha = 5\%$).

Results: Except for Filtek Bulk-Fill cured for 20 s, no other resin was effectively cured at 4 mm regardless of the LCU and curing time. Less effective curing was observed for 10 s curing time. Curing ability of the LCUs appears to be dependent on the resin and curing time, but, in general, PW resulted in improved curing for both curing times. Four-mm increment appears to be too thick of a layer to permit effective curing with LCUs used in this study for a 10 s or 20 s curing time.

Conclusions: Curing effectiveness was poor at 4 mm for most of the resin composites regardless of the LCU and curing time.

Composite	10 s		20 s	
	Monowave	Polywave	Monowave	Polywave
Percentage of curing at the bottom vs. top ($n = 15$)				
HerculiteXRV	70.0(21.1)^{A,a}	58.0(19.0)^{B,b}	75.5(15.4)^{A,a}	79.1(11.7)^{A,ab}
EsthetX	37.9(11.8)^{A,b}	41.1(9.0)^{A,c}	47.2(17.4)^{A,b}	39.7(20.8)^{A,c}
TetricEvoCeram	58.8(16.2)^{A,a}	55.3(10.7)^{A,bc}	68.4(18.8)^{A,a}	71.5(19.23)^{A,ab}
TetricEvoCeram-BulkFill	56.7(20.8)^{B,a}	70.5(11.8)^{A,ab}	63.7(23.1)^{A,ab}	65.4(14.6)^{A,b}
Filtek Bulk-Fill	70.7(15.7)^{A,a}	76.0(15.1)^{A,a}	80.6(13.3)^{A,a}	85.9(11.5)^{A,a}

Upper case compare columns and lower case compare rows.

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Interaction between a bisphosphonate, zoledronate, and biomimetic hydroxyapatite

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Purpose/Aim: Bisphosphonates (BPs) are well established as antiresorptive agents for the prevention and treatment of bone diseases. BPs can be administered either orally or intravenously. Studies suggest that patients using BPs may be prone to develop a type of maxillary osteonecrosis, although a direct causal relationship has yet to be determined. Conversely, once released locally from the surface of coated dental implants, bisphosphonates have shown to improve bone fixation of such implants. The aims of the present study were to dope hydroxyapatite (HA) with a bisphosphonate of 3rd generation, zoledronate (ZL), and to characterize their interaction by physicochemical and morphological analyses.

Materials and methods: Well-crystallized hydroxyapatite (HA) was synthesized via a sol-gel process using calcium nitrate and phosphoric acid as precursors solutions in concentrations that were set to achieve a stoichiometric Ca:P ratio = 1.67, for the final synthesis of 5 g of HA. The adsorption of zoledronate (ZL) to HA nanocrystals was performed by dispersing 50 mg of HA powder in 5 mL of an aqueous solution of zoledronate (0.2342 g/L diluted in a 1 mM KCl solution). The pH was monitored to assure it was neutral. The suspensions were sonicated and incubated at 37 °C without agitation. Then, centrifugation was performed and the supernatants were filtered through Millipore membranes (0.22 μM pore size). Physicochemical and morphological characterizations of the HA and HA doped with ZL (HA + ZL) were performed by X-ray diffraction (XRD), vibrational spectroscopy by Fourier transform infrared (FTIR), and scanning electron microscopy (SEM).

Results: The XDR diffractograms for HA + ZL showed that ZL adsorption did not cause formation of new crystalline phases

since all the observed peaks and planes were exclusively in accordance with that of the crystallographic record (74-0565) of pure HA. Conversely, adsorption of ZL was found to decrease the relative intensity of all diffraction peaks of HA, then suggesting an interaction between HA and ZL. All chemical bonds observed in the vibrational spectra for HA were identified for samples of HA + ZL, but, for these, it was noticed a reduced intensity of all bands, then suggesting an effective interaction between HA and ZL. SEM analyses did not show a consistent morphological difference between particles of HA and HA + ZL.

Conclusions: This preliminar study shows that ZL can potentially interact with HA. This result is driving studies that seek for understanding how the adsorption and release mechanisms can provide fundamental tools for the development of drug delivery systems using hydroxyapatites and bisphosphonates.

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Fatigue resistance of cubic/tetragonal translucent zirconia crowns

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Purpose/Aim: Fracture of veneering ceramic of zirconia-based FPDs is reported as the most common clinical failure. Monolithic zirconia restorations have been proposed to improve strength and survival-rate. However, due to their low translucency, the esthetics of partially stabilized tetragonal zirconia (Y-TZP) restorations is mostly unacceptable. Translucent zirconias, containING tetragonal and cubic grains, have been recently introduced (Katana Super-Translucent STML, and Ultra-Translucent UTML; Kuraray-Noritake), to enhance their esthetic properties. The aim of this study was to compare the fatigue resistance of crowns made of different ceramic materials. The null hypothesis was that the survival-rate among the different ceramic crowns did not vary ($\alpha = 0.05$).

Materials and methods: A digital impression of a lower molar was taken using TDS oral-scanner (3 M) and digitally reduced (3D-Materialise-Magic software) to receive all-ceramic crowns. One-hundred monolithic crowns having the anatomy of the original natural tooth were replicated using CAD/CAM technology in different thicknesses: 20 Y-TZP (Katana) 1.0 mm crowns; 20 STML 1.0 mm crowns; 40 UTML 1.0 and 1.5 mm crowns (20 specimen each), and 20 lithium-disilicate (IPS e.max CAD, LT, Ivoclar-Vivadent) 1.5 mm crowns. The crowns were cemented with Panavia V5 cement (Kuraray-Noritake) on 100 CAD/CAM abutment replicas milled from a dentin analogue fiber-composite material. The initial weight of each specimen was measured. The crowns were subjected to fatigue cycling. The fatigue-testing device (ball-mill) is a rotating 120 mm ID-stainless steel, water-tight drum which contains stainless steel and zirconia balls with the test specimens. During rotation at 84.5 rpm in 37 °C distilled water, the balls generate randomly distributed impact stresses of known maximum energy (Steel: 0.0510 J; ZrO2 = 0.0197 J) on the occlusal surface of the restorations. The ball impacts may induce cracks growth and fracture propagation in brittle materials. Every 60 min the ball-mill was opened for specimen's weight losses measurements and failures examination. Kaplan–Meier survival curves and Log-rank comparison test have been performed ($\alpha = 0.05$) considering early and catastrophic failure events.

Results: The Y-TZP 1.0 mm crowns showed the highest fatigue resistance, not statistically different from UTML cubic/tetragonal 1.5 mm zirconia and lithium-disilicate 1.5 mm crowns. 1.0 mm STML and UTML crowns were significantly less resistant to fatigue cycling ($p < 0.001$). UTML showed the highest failure rate.

Conclusions: The greatly improved optical properties of the newest translucent zirconia ceramics require a low content (<50%) of tetragonal phase; as a consequence, their fatigue resistance seems to approach the lithium-disilicate levels. This study suggests that the fatigue survival of translucent zirconia crowns reflects the tetragonal/cubic phase ratio (T/C), being STML (higher T/C) more resistant than UTML (lower T/C).

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Mini-interfacial fracture toughness of adhesive-dentin interfaces after plasma pre-treatment

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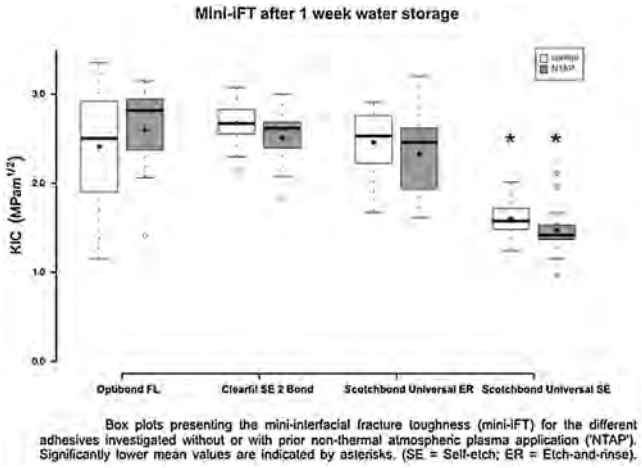
Purpose/Aim: This study aimed to determine the mini-interfacial fracture toughness (mini-iFT) of adhesive-dentin interfaces prepared following different adhesive strategies with/without beforehand non-thermal atmospheric plasma pre-treatment (NTAP). Water contact-angles of pre-treated dentin were also measured to assess possible alterations in surface hydrophilicity upon NTAP.

Materials and methods: Forty human molars ($n = 10$) were used after partial crown removal to expose flat dentin surfaces. One half of each dentin surface received NTAP for 15 s, while the other half was covered with metallic paper during the plasma application and served as intra-tooth control. The adhesives tested were the 3-step etch-and-rinse (ER) adhesive Optibond FL (O-FL, Kerr), the 2-step self-etch (SE) adhesive Clearfil SE Bond 2 (C-SE2, Kuraray Noritake) and the multi-mode adhesive Scotchbond Universal (SBU, 3 M ESPE), the latter applied following both a 2-step ER and 1-step SE mode. Upon application, the adhesives were light-cured



Box plots presenting the mini-interfacial fracture toughness (mini-iFT) for the different adhesives investigated without or with prior non-thermal atmospheric plasma application ('NTAP'). Significantly lower mean values are indicated by asterisks. (SE = Self-etch; ER = Etch-and-rinse).

Fig. 1

using a polywave LED curing light (Bluephase 20i, Ivoclar Vivadent: "high mode") for 10 s. A composite (Filtek Z100, 3 M ESPE) build-up was made in layers. After 1-week water storage at 37 °C, the specimens were sectioned perpendicular to the interface to obtain 6 rectangular mini-iFT specimens (1.5 × 2.0 × 16–18 mm) per tooth. Next, a single notch was cut at the adhesive-dentin interface. Half of the specimens were loaded until failure in a 4-point bending test to determine the 'immediate KIC', while the other halves were stored in distilled water to determine the 'aged KIC' in the future. Water contact-angle analysis was conducted using CAM200 goniometer (KSV Nima, Finland). Droplet images of dentin surfaces with or without 15-s plasma treatment were captured at different water-deposition times (5–55 s) (Fig. 1).

Results: Two-way ANOVA revealed significant differences in mini-iFT among the different adhesives tested. However, no difference in mini-iFT was recorded with or without NTAP pre-treatment. The lowest mini-iFT was recorded for SBU applied in SE mode, while no statistically significant difference was found among the other adhesives. Plasma-treated dentin surfaces revealed lower contact-angle values for all water-deposition times.

Conclusions: The mini-iFT appeared to depend on both the adhesive and application mode. SBU applied in ER mode resulted in a similar bonding performance as C-SE2 and O-FL, but performed less effectively in SE mode. At short time, dentin pre-treatment with plasma did not influence the mini-iFT of adhesives bonded to dentin, despite the dentin-surface hydrophilicity was considerably increased. The long-term mini-iFT will be measured in due time.

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Marginal adaptation of composites on dentin with different imaging techniques

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Purpose/Aim: The aim of this in vitro study was the comparison of different imaging techniques, the optical coherence tomography (OCT), the Confocal Laser Scanning Microscopy (CLSM) and the micro CT, for the evaluation of the marginal sealing to dentin cervical margin of second class cavities restored with different composite materials after thermal aging. The null hypothesis is that there is no difference in marginal sealing between composites of different viscosity and that the three imaging techniques used are valid methods to evaluate marginal interfaces.

Materials and methods: 16 intact upper molars, extracted for periodontal reasons, were selected. A mesio-occluso-distal cavity with cervical margins placed 1 mm below the CEJ was performed on each tooth. Samples were then all treated with Scotchbond Universal in etch-and-dry technique. Restorations were performed as follow: a 1 mm horizontal layer of bulk-fill composite was placed over the mesial box cavity floor and cured for 20 s with LED lamp. Then, a composite restoration was performed with oblique layering of nanohybrid resin composite. OCT, micro CT and CLMS images were performed after 24 h and 10,000 thermocycles. Images were analyzed with the software ImageJ to assess the percentage of marginal gap between composite and cervical margin. Data were statistically analyzed ANOVA test and significance was set for $p < 0.05$.

Results: ANOVA test showed that the interfacial adaptation was statistically influenced by thermocycling, which worsened the interfacial adaptation of composite materials to dentin. CLSM closely confirmed OCT and micro CT findings in all samples, because the interfacial adaptation analyzed by OCT and micro CT corresponded to the interfacial adaptation analyzed by CLSM.

Conclusions: Thermal aging negatively affected interfacial adaptation of all tested materials. The three imaging methods employed in this in vitro study showed comparable results and can be considered valid non-destructive method to assess marginal adaptation.

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Marginal and internal space of metallic copings

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Purpose/Aim: Marginal and internal fit of prosthetic crowns are necessary characteristics to a successful rehabilitating treatment. It is expected that a CAD/CAM system provides an adaptation with either equivalent or superior fit when compared to those produced by the conventional technique. Yet, many studies in the area have shown varied results. Based on that, the aim of this study was to evaluate the marginal fit and internal space of cobalt-chromium copings produced by conventional casting ($n = 10$) and laser sintering ($n = 10$).

Materials and methods: The preparation for a total crown was executed on an acrylic master die-model, replicated in plaster and digitalized. After injection and sintering, copings were filled with light-body addition silicone and positioned on the die-model, obtaining a replica of the cementing agent's space. The replicas were included in heavy-body addition silicone and sectioned in the vestibular, lingual, mesial and distal regions. Posteriorly, silicone thickness relative to marginal, axial and occlusal spaces of each side were analyzed in a stereomicroscope (Leica® MZ6), 10× magnification. Data were submitted to ANOVA 2 factors and Tukey test.

Results: Considering all sides, the conventional group showed a narrower cementing space when compared to the sintering group ($p = 0.041$). The values statistically differed regarding the regions ($p < 0.001$) and there was interaction between the variables group and region. The Tukey test showed significant statistical difference between the occlusal region and the other regions ($p < 0.001$) for both groups and showed that there was not significant statistical difference between the cementing space of marginal and axial regions among the analyzed groups ($p = 0.238$).

Conclusions: Thus, it is possible to conclude that both the conventional manufactured copings and the sintered copings showed a comparable cementing agent's space in the marginal region. However, when all regions were considered, the conventional group showed a superior fit.

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Fluoride varnishes: *In vitro* assessment of fluorine diffusion on enamel

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Purpose/Aim: Fluoride varnish is widely used in North America and Europe to reduce caries risk and remineralize enamel tooth structure. The purpose of this study was to determine the effectiveness of four fluoride varnishes, aiming quantify the amount of fluoride absorbed by the enamel surface in a given period of time by determining the outermost layer chemical composition.

Materials and methods: Four brands of fluoride varnish were used in this study: FluoroDose (FD) [Centrix], 3 M Vanish (3MV) [3 M], Enamel Pro Varnish (EPV) [Premier Dental] and Fluor Protector (FP) [Ivoclar Vivodent]. Human enamel samples ($n = 3$) were prepared by carefully sectioning each permanent human molar into four equal parts for the application of fluoride varnish on three parts representing one week, one month, and six months, leaving the fourth part to serve as the control. Each varnish was mixed as per the manufacturer's instructions and applied to a piece of enamel. In three different spots for each sample, energy-dispersive X-ray (EDX) was used to analyze the enamel chemical composition and the fluoride amount. After application of fluoride varnish, each sample was stored in 100% relative humidity condition and kept at 37 °C.

Results: The table below brings the fluorine atomic percentage on enamel after the four fluoride varnishes application according to the time. After one month, no statistically significant intergroup difference in surface fluoride was noticed on the prepared sample. After six months, FP had the largest release of fluoride of exposure on prepared human enamel samples. 3MV had a steady release of fluoride for one week up to one month, with little fluoride found on the enamel surface after 6 months. Enamel Pro Varnish showed a large increase in enamel surface fluoride after one week, but this dwindled dramatically after one month, and more so after six months. Fluor Protector had a steady presence of fluoride from one week to one month and showed a dramatic increase after six months. FluoroDose showed a steady increase over the six-month period, increasing in surface fluoride after each time interval.

Conclusions: The EDX spectroscopy reveals a different trend for each fluoride varnish. If we extrapolate this data to in vivo situations, there appears to be an effect related to the presence of moisture and the activity of fluoride varnish.

	1 week	1 month	6 months
F (%) Average (SD)			
3 M varnish	0.695 (±0.6)	1.445 (±0.8)	0.2 (±0.07)
EPV	3.8 (±0.4)	1.1 (±0.7)	0.88 (±1.1)
Fluor protector	0.6 (±0.4)	0.7 (±0.5)	5.05 (±0.7)
Fluor dose	1.14 (±0.29)	0.74 (±0.09)	3.82 (±0.33)

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Effects of bioactive suspensions on resin–dentin interface

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Purpose/Aim: To evaluate the effect of rewetting suspensions of niobiophosphate (NbG) and 45S5 glass on the microtensile bond strength (μTBS), ultra-microhardness, and hardness modulus of resin–dentin interfaces after 3 months of storage. Antibacterial properties of the suspensions were also investigated.

Materials and methods: The specimens were assigned into 5 groups of 4 different concentrations (5%, 10%, 15% and 20% w/v) of either NbG or 45S5 suspensions, and 1 control group (distilled water). Dentin surfaces of twenty-five human third molars were etched with phosphoric acid (35%), water rinsed and air-dried (30 s). Dentin surface was rewetted according to their respective groups and two consecutive layers of a simplified etch-and-rise adhesive system (One Step) were applied. Resin–dentin bond strength testing was carried out after 24 h and 3 months of storage in PBS. Hardness and modulus testing across the interface were processes using a Berkovich diamond indenter at both 24 h and 3 months storage time. Antibacterial activity (*Streptococcus mutans* ATCC 159) of each group was evaluated. The suspensions were serially diluted and inoculated into BHI agar plates and incubated for 48 h at 37 °C, and CFU were counted under stereomicroscope and expressed as log CFU/ml. The data were analyzed by 2-Way ANOVA and Holm–Sidak tests at $\alpha = 5\%$. Hardness properties were analyzed by paired t test for each group (24 h × 3 months).

Results: There were significant interactions between groups in μTBS ($p = 0.033$). Significant reductions in μTBS were observed after 3 months PBS storage for control and NbG 5% groups. Suspensions with 5% and 20% of 45S5 and 20% of NbG resulted in stable μTBS and increased hardness after 3 months storage. Both 45S5 and NbG 20% solutions increased modulus after 3 months. A significant reduction in bacterial growth was observed with 20% 45S5 when compared with the other groups.

Conclusions: Rewetting demineralized dentin with a suspension of 20% 45S5 prevented bond strength reduction, increased hardness and modulus at the interface and showed antibacterial activity against *Streptococcus mutans*.

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Effect of resin cement and aging on bonding to ceramic

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Purpose/Aim: The purpose of this study was to evaluate the effect of resin cement types, and thermocycling and fatigue procedures on the microtensile bond strength (μTBS) of ceramic IPS e.max Press/resin cement/composite after 24 h and 1 year.

Materials and methods: Seventy two ceramic square blocks (8 mm × 8 mm × 3 mm thickness) were made, and divided into 12 groups ($n = 6$): Groups 1 to 6 were bonded with a self-adhesive (RelyX U200, 3 M ESPE) resin cement; and Groups 7 to 12 bonded with a dual-cured (Variolink II, Ivoclar Vivadent) resin cement. All the specimens were etched with 10% hydrofluoric acid for 20 s, rinsed for 30 s, and dried for 30 s. Silane material was applied on treated ceramic surfaces and the blocks were bonded to a block of composite resin (Z250, 3 M ESPE) with resin cement RelyX U200 or Variolink II, and light cured for 40 s. The specimens were stored in deionized water at 37 °C for 24 h. Groups 2, 5, 8 and 11 were submitted to 3000 thermal cycles between 5 °C and 55 °C while Groups 3, 6, 9 and 12 to a fatigue test with 250,000 cycles at 2 Hz and 80 N. Specimens were sectioned perpendicularly to bonding area to obtain sticks with a cross-sectional area of 1mm2 (15 sticks per group), and submitted to the μTBS at a crosshead speed of 0.5 mm/min. Data were submitted to ANOVA and Tukey's post-hoc test ($\alpha = 0.05$).

Results: Bond strength values (MPa – mean ± standard deviation) for the specimens stored 24 hours and 1 year were: Control group (30.9 ± 1.7 and 25.3 ± 1.4) were significantly higher than both thermocycled (25.2 ± 1.5 and 21.3 ± 1.6) and fatigue (22.3 ± 2.8 and 19.5 ± 1.2) groups, regardless of resin cement and storage time procedures ($p < 0.05$). The thermocycled groups showed significantly stronger values than fatigue groups ($p < 0.05$). The storage for 24 hours showed significant higher value than 1 year for all treatments ($p < 0.05$). Value for Variolink II (25.2 ± 3.2) was significantly higher than RelyX U200 (23.1 ± 3.7), regardless of storage time and treatment procedures ($p < 0.05$).

Conclusions: The thermocycling and fatigue procedures significantly decreased the μTBS for both storage times in relation to control group. Variolink II had significantly higher performance than RelyX U200.

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Fluoride uptake measurements from sealants formulated with microencapsulated remineralizing agents

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Purpose/Aim: Primary caries at the interface of sealants and mineralized tooth structure continues to be an issue due to marginal defects and gaps. The objective of this study was to demonstrate that sealants with microencapsulated remineralizing agents with sustained release of fluoride, calcium and phosphate ions promotes enamel fluoride uptake.

Materials and methods: Sealants that contained 5% w/w microcapsules with aqueous solutions of 5 M $Ca(NO_3)^2$ or 0.8 M NaF or 6.0 M K2HPO4 or a mixture of all three were prepared. Ion release profiles were measured as a function of time. Enamel fluoride uptake by demineralized tooth structure was determined by a modified version of FDA Method 40. In the Method 40 experiment, the enamel surface is soaked in acid before and after treatment with a fluoride releasing material. The amount of fluoride in the acid is measured using ion specific electrodes. The amount of enamel was derived from the amount of calcium in the acid by AA. The amount of fluoride associated with the enamel before and after treatment is therefore reported as microgram of fluoride per gram of enamel. All sets of data was $n = 12$.

Results: Sustained release of fluoride, calcium and phosphate ions from a sealant was demonstrated. Fluoride uptake by demineralized enamel was significantly increased compared to a control sealant manufactured without microcapsules. Bovine enamel that contained 2.2 ± 2.1 μg F/g of enamel prior to exposure to a sealant without microcapsules had 2.3 ± 0.5 after 90 days. Enamel exposed to sealant with 5% w/w NaF microcapsules went from 3.5 ± 3.5 μg F/g of enamel of enamel prior to exposure to 148 ± 76 after 90 days. Enamel exposed to sealant with 2% w/w NaF, 2% w/w $Ca(NO_3)^2$ and 1% w/w K_2HPO_4 microcapsules went from 1.7 ± 0.7 μg F/g of enamel prior to exposure to 190 ± 137 after 90 days.

Conclusions: Sealants with encapsulated remineralizing agents are capable of releasing biologically available fluoride, calcium, and phosphate ions. Incorporation of these microcapsules in pit and fissure sealants is a promising method for remineralization based on enamel fluoride uptake measurements.

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Immediate effect of different hydrophobic-resin-coating approaches on dentin with universal-adhesive

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Purpose/Aim: The purpose of this study was evaluating the immediate effect of photoactivation moment and application mode of an additional hydrophobic resin coating (HB) on dentin adhesion associated with a universal adhesive system used in etch-and-rinse (ER) and self-etch (SE) approach, by the resin–dentin microtensile bond strengths (μTBS) and nanoleakage (NL) tests.

Materials and methods: For this, sixty caries-free extracted third molars were divided into 10 groups according to the combination of the factors: (1) photoactivation moment (1200 mW/cm^2), of the HB (adhesive system and HB were photoactivated together [PhTg]; or the adhesive system and HB were photoactivated separately [PhSp], and (2) application mode of HB (active or passive), into two levels corresponding to the adhesive approach (ER and SE). As controls, ER and SE were applied according to the manufacturer's instructions. After adhesive procedure as described above for each group, resin restorations were constructed (2 layers of 2 mm each/photoactivated 1200 mW/cm^2), the teeth were stored in water (37 °C/24 h) and sectioned according to "x" and "y" axis, into resin–dentin beams (cross-sectional area 0.8 mm^2) to be tested under tension (0.5 mm/min) for μTBS. Selected beams from each tooth and group were used for NL evaluation by SEM. Data from each experimental groups were analyzed with two-way ANOVA and Tukey's test ($\alpha = 0.05$).

Results: For μTBS test, the application modes influence the results. In PhSp moment, the active application of HB resulted in higher μTBS values for ER and SE approach ($p < 0.01$). For PhTg moment, the active application decreases the μTBS values only for SE ($p < 0.01$). For NL evaluation, lower values of infiltration rate were observed when the HB was applied independent of adhesive approach ($p < 0.01$) and photoactivation moment ($p < 0.01$).

Conclusions: In resume, the use of HB is useful strategy to improve the immediate μTBS values of adhesive interface in dentin when universal systems its used, but their optimum effectiveness is dependent of the application mode, photoactivation moment and adhesive approach.

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Survival of lithium-disilicate table-tops as a function of thickness

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Purpose/aim: Thin lithium-disilicate adhesive restorations (table-tops) have been proposed to rehabilitate worn-out posterior teeth, increasing the vertical dimension of occlusion. However, doubts still remains about the minimum ceramic thickness to be used when making this type of occlusal coverage. The purpose of this study was to determine the survival rate of simplified lithium-disilicate restorations as a function of their thickness.

Materials and methods: Sixty sound human molars were extracted and stored in 0.02% thymol solution then severed in a plane parallel to the occlusal surface in order to create a flat dentin surface surrounded by enamel edges. Teeth with pulp horns exposed during cutting were discarded. Three groups ($n = 20$) were randomly created and 60 lithium-disilicate (IPS e.max Press, Ivoclar-Vivadent) flat table-tops were made using the lost wax technique. The ceramic restorations were applied to each teeth using Multi-Link cement (Ivoclar-Vivadent) spaced to obtain a 100 μm film. The restoration thicknesses were distributed as follows: group A: 0.5 mm, Group B: 0.8 mm, and group C: 1.2 mm. The restored teeth were placed in a ball-mill fatigue machine containing 10 zirconia and 10 stainless steel spheres 20 and 25 mm in diameter, respectively, along with 500 ml distilled water at 37 °C. The ball-mill created randomly distributed impacts of a known maximum energy on a target area of about 6 mm in diameter on the occlusal ceramic surface of the restored teeth. Crack growth and damages occurring in the lithium-disilicate table-tops were evaluated under a stereomicroscope by opening the ball-mill every 60 min. Survival curves (Kaplan–Meier) were obtained and compared using the Log-rank test (alpha = 0.05).

Results: As expected, group A (0.5 mm) showed the lowest survival rate among the three groups tested, the difference was statistically significant when compared to both group B and C ($p < 0.05$). Group B and C showed similar survival rate ($p > 0.05$), slightly higher for specimens belonging to group C. The most common failures observed were conchoidal and full-thickness parting-type fractures.

Conclusions: Fatigue survival of lithium-disilicate restorations is strongly related to the thickness of the ceramic material. This study suggests that simplified table-top restorations could be safely made adopting a thickness of 0.8 mm or more. Very thin restorations (0.5 mm) are not recommended as far as survival to fatigue testing is concerned.

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Bisphenol A and other monomers release from four orthodontic adhesives

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Purpose/aim: Bisphenol A (BPA) is an endocrine disruptor with potential toxicity. Dental adhesives do not contain pure BPA, but its derivatives are widely used in their compositions, as well as other monomers, which could have adverse effects. The aim of this study was to analyze, by high performance liquid chromatography (HPLC), the release of BPA, Bis-GMA, TEGDMA, UDMA and HEMA, from four orthodontic adhesives, in two in vitro conditions and to extrapolate the data to the clinical situation.

Materials and methods: 24 cylinders were prepared, 6 per adhesive, which were divided in 2 subgroups ($n = 3$). In the first subgroup, the cylinders were light-cured 2s, then the three cylinders of each adhesive were respectively placed in acetonitrile; in the second subgroup, the cylinders were light-cured 20s, then the three cylinders of each adhesive were respectively placed in artificial saliva. For each subgroup, the monomers release was determined with HPLC at 28 days. Besides, 120 brackets (10 of each type) were bonded over metal teeth. Monomers release from adhesive residues was calculated from the data obtained with the cylinders.

Results: Whatever the adhesive and the extraction solution, no trace of BPA was detected. Other monomers were detected, in varying amount according to the adhesive. Some discrepancies were found between these results and the manufacturer data. Whatever the adhesive, the amount of monomers released was significantly higher in acetonitrile than in saliva.

Conclusions: The risk of BPA release after orthodontic bonding would be 4000 times lower than the TDI for a 30-kg child. However, other monomers are released. Light-curing condition and storage solution impact on the amount of released monomers. The precautionary principle should be applied and preventive measures implemented to reduce the monomer exposure. Manufacturers should be required to report the exact composition of their products.

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Polymeric proanthocyanidins effects on dentin and adhesive interfaces

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Purpose/aim: Proanthocyanidins (PACs) are plant derived compounds with ability to modify the dentin matrix by mediating collagen cross-linking. This study aimed to determine the effect of high molecular weight proanthocyanidins on the modulus of elasticity and the dentin-resin bond strength.

Materials and methods: The source of PACs was grape seed extract (GSE) from Vitis vinifera. A fraction (LP-GSE) of high molecular weight PACs (degree of polymerization 10 and up) was obtained by 2 phase solvent partitioning method. Dentin beams ($n=15$) (0.5 mm × 1.7 mm × 6.5 mm), were demineralized in 10% phosphoric acid and treated with the respective solutions for 1 h and tested immediately, after 3, 6 and 12 months: LP-GSE 6.5%, GSE 6.5% (crude) and HEPES (control). The apparent modulus of elasticity (E) was assessed using three-point bending.

For the dentin-resin bond strength, an experimental HEMA-free adhesive system was used. Human molars ($n=7$) were ground flat to expose the middle coronal dentin, etched with 35% phosphoric acid, followed by the surface treatments (15% LP-GSE, 15% crude and control). The adhesive system was applied and restorations were incrementally built using Filtek Supreme. The teeth were sectioned to obtain 0.8 mm × 0.8 mm specimens and tested on a microtensile tester after 24 h and after being aged for a year. Data were statistically analyzed by ANOVA and Tukey, with $\alpha=0.05$.

Results: In E, there were no differences among immediate, 3, 6 and 12 months for LP-GSE ($p=0.122$). However, significant decrease in E occurred after 3 months for crude ($p<0.001$) and after 12 months for control ($p<0.001$). Higher bond strength was observed in crude (35.05 ± 3.54; 30.99 ± 14.40 MPa) and LP-GSE (37.49 ± 4.18; 24.59 ± 12.14 MPa) for 24 h and 1 year respectively, as compared to control (4.06 ± 5.27; 1.43 ± 2.56 MPa) ($p<0.001$). Significant decrease in TBS was observed for all groups after 1 year ($p=0.016$).

Conclusions: High molecular weight PACs exhibited less, however stable increase in dentin matrix modulus of elasticity, likely due to the size of the polymers. The decrease in bond strength observed herein after 1 year reinforces the need for more enriched versions of PACs-rich agents.

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Effect of bioactive-glass primer on dentin adhesion with a universal-adhesive

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Purpose/aim: The aims of this study were evaluated the immediate effect of a primer with Bioactive Glass [BG-primer], on biomechanics and ultra-structural properties of universal adhesive system on dentin adhesion in two different strategies. As the null hypothesis, it suggests that the BG-primer not influence the immediate biomechanical and ultra-structural properties of the interface formed.

Materials and methods: To evaluated this, ten third molars extracted caries-free, donated under informed consent, were divided into 4 groups ($n=5$) according to the: (1) use of the BG-primer before the adhesive strategy and (2) adhesive strategy selected to dentin hybridization (self-etch [SE] and etch-and-rinse [ER]), according to the manufacturer's instructions without use of BG-primer. These groups were implemented as a control. After application of the adhesive strategies described, the teeth were restored with composite (2 mm × 2 mm increments separately photoactivated 1200 mW/cm^2) and stored in water at 37 °C for 24 h. Before that, the teeth were sectioned according to "X", "Y" and "Z" axis, to obtain resin-dentin specimens (0.8 mm^2 cross-sectional area). In the sequence, the specimens obtained were tested under tension (0.5 mm/min) to evaluate the microtensile bond strength of dentin-resin interface (μTBS). Selected specimens from each tooth and group were used for nanoleakage pattern evaluation (NL) by scanning electron microscopy. Data from each group and test were analyzed with two-way ANOVA and Tukey's test ($\alpha=0.05$).

Results: For adhesive strategy factor, the ER strategy shows better bond strength values when compared to SE ($p<0.05$), independent of BG-primer use. In other hand, no significant differences were observed between the different groups for immediate μTBS ($p>0.05$) values when the use or not of BG-primer was compared. Meanwhile NL patterns show no significant differences in the degree of infiltration between groups ($p>0.05$), despite the trend of higher degree of infiltration observed in the SE strategy.

Conclusions: Stable immediate values for biomechanical and ultra-structural evaluation were observed to universal system tested, independent of the addition of BG-primer step, showing that this strategy did not affect the immediate performance of the adhesive. Accordingly, the null hypothesis is accepted. This initial study allows future evaluations of longevity, in function of observed the BG-primer capacity to prevent the degradation.

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In vitro antimicrobial activity of chitosan/propolis gels for intracanal medication

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Purpose/aim: Chitosan based gels might be carriers for antimicrobial drugs acting in the infected root canal. In addition, chitosan has showed antimicrobial properties, and it might be formulated as a gel with a physical consistency ad hoc for application as intracanal medication. Propolis has been largely investigated for its antimicrobial activity. Hence, the formulation of a chitosan/propolis gel is of great interest for possible applications in endodontics. In this study, we aimed the formulation of chitosan/propolis gels and the evaluation of its antimicrobial activity.

Materials and methods: Chitosan (Sigma–Aldrich MW 50,000–190,000) was dissolved in 1% acetic acid (4 mL). Then, we added a solution of 30% propolis (1 mL), and the mix was stirred (500 rpm for 10 min) to get a homogenous gel (CH-30%P). Another gel was formulated, but for that one we added propolis as a powder (CH-Pp). The antimicrobial activity of the gels was assayed with a modified Kirby–Bauer method. We used an *Enterococcus faecalis* strain for the assay. The gels were dropped in a 6-mm hole in the Mueller–Hinton agar, and the bacteria and gels were incubated for 24 h. A chitosan gel (blank) (CHb), a 30% propolis solution (30%-P), a PEG 400-calcium hydroxide paste ($Ca(OH)_2$), a positive control (ampicillin disc) (PC), and a negative control (NC) (sterile water) were also tested under the same conditions. The inhibition zones were imaged and measured. A One-way ANOVA and Tukey's HSD test were performed to identify possible statistical differences between treatments.

Results: The chitosan/propolis gels showed an antimicrobial activity stronger than the antimicrobial activity induced by the chitosan gel (blank), the 30% propolis solution, the PEG 400-calcium hydroxide. The zones of inhibition caused by the chitosan/propolis gels were almost 2 times larger than the inhibition zones caused by the other treatments. However, no statistical significance was noticed between the gels and the treatments. The positive control produced the largest zone of inhibition, and all groups showed significant statistical differences with the ampicillin disc.

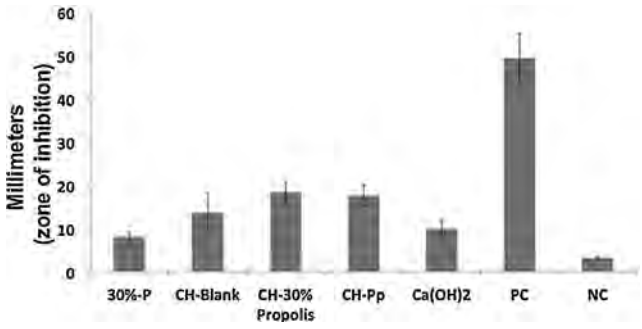

Fig. 1

Conclusions: The chitosan/propolis gels showed in vitro antimicrobial activity agains *E. faecalis*, and although it was stronger than the activity observed for the other treatments, the antimicrobial action of the gels was no superiors to the positive control (Fig. 1).

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WITHDRAWN

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Dentin treatments effect on the bond strength of universal adhesives

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Purpose/aim: To evaluate the effect of different conditioning protocols on the resin-dentin bond strengths of two multimode dental adhesives.

Materials and methods: 60 flat dentin surfaces from sound extracted human molars were polished with 320-grit SiC paper and randomly divided into two groups according to the adhesive applied: ScotchBond Universal™ (3M/ESPE) or Adhese Universal (Ivoclar/Vivadent). Five subgroups were formed according to the dentin conditioning strategy: 35% Phosphoric acid for 15 s 35%; Phosphoric acid for 5 s; Self-etching mode; 17% Ethylenediaminetetraacetic acid for 30 s; 25% Polyacrylic acid for 10 s. The adhesives were applied for 20 s and 4-mm resin composite build-ups (Aelite, Bisco) were incrementally added and light-cured (Bluephase G2, Ivoclar Vivadent) at 1100 mW/cm^2. The specimens were stored in PBS at 37 °C and sectioned after 24 h into beams (1 mm^2) to be tested under microtensile (μTBS) in a Universal Testing Machine (Shimazdu) at 0.5 mm/min until failure. Bond strength data were analyzed using two-way ANOVA and Tukey's test ($\alpha = 0.05$). Failure mode was examined under light microscopy.

Results: The interaction between factors was statistically significant ($P = 0.002$). The treatments with Phosphoric acid with reduced etching time (5 s) presented the highest μTBS with Scotchbond Universal, not different statistically from the Self-etching application and Ethylenediaminetetraacetic acid treatment, while with Adhese Universal the highest μTBS was obtained with Ethylenediaminetetraacetic acid and Self-etching treatments. The lowest μTBT was obtained when the Polyacrylic acid treatment was used with Adhese Universal (Table 1).

Table 1 – Microtensile bond strength values (MPa ± SD). Capital letters compare treatments. Lowercase compare adhesives.

Treatments	Scotchbond Universal	Adhese Universal
Phosphoric Acid (15 s)	39.90 (20.1)^{B,a}	34.25 (11.3)^{C,D,a}
Phosphoric Acid (5 s)	57.58 (21.8)^{A,a}	39.07 (17.1),^{BC,b}
Self-etching Mode (20 s)	50.78 (19.4)^{A,a}	49.80 (14.4)^{A,B,a}
Ethylenediaminetetraacetic acid (30 s)	47.83 (20.2)^{A,a}	56.31 (20.8)^{A,a}
Polyacrylic Acid (10 s)	40.90 (17.1)^{B,a}	22.86 (11.7)^{D,b}

Conclusions: Different conditioning protocols significantly affected the bond strength of Scotchbond Universal and Adhese Universal to dentin and the effects were material-dependent.

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Fractal analysis of anodized titanium surfaces

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Purpose/aim: The success of dental implants is often associated with a positive integration of bone tissue with the implant surface. Titanium implants are anodized to enhance the surface oxide layer for improved osseointegration and antimicrobial properties. Surface roughness is often used to describe the morphology of anodized coatings, but fractal dimension may help explain the complex mechanisms occurring at the implant tissue interface. The purpose of this research was to compare the surface roughness and fractal dimension values of anodized titanium implant material.

Materials and methods: Commercially pure titanium grade 4 (CPTi-4) specimens were anodized to six forming voltages using a potentiostatic waveform in four mixed acid electrolytes A–D. Surface pore morphology of each specimen was examined using scanning electron microscopy (SEM). Surface roughness was measured on four 50 μm × 50 μm areas with an AFM using ScanAssyst mode. Gwyddion software was utilized to calculate roughness average (Ra) values. Fractal dimensional increment values (D^*) were determined by exporting height data from the AFM into a MathCAD script to convert it into the format used by FRACTALS software. A custom MathCAD script was used for leveling of 3D surface coordinates. FRACTALS software was utilized for leveled and unleveled fractal dimensional analysis using Minkowski Cover (MC) and MC D^* values were corrected for bias using the method of McMurphy et al.

Results: The surface morphologies showed formation of numerous small pores initially, then growth of these pores with increasing voltage, and initiation of new small pores with continued growth in the surrounding areas. Electrolyte A showed the highest Ra value at 144 V while the highest D^* was shown at 108 V. The Ra values peaked at 204 V for electrolytes B–D while the D^* values were highest at 120 V, 144 V, and 108 V, respectively.

Conclusions: Surface roughness values showed that in general, the surfaces became rougher with increasing forming voltage up to 204 V in each electrolyte. Fractal analysis showed that D^* reached a peak value at lower forming voltages (108–144 V) and leveled off at higher forming voltages near 204 V. In the present study the highest surface roughness did not correlate with the highest D^* value in any of the electrolytes. Future studies will be completed to determine the relationship between Ra, D^*, and surface energy in each of the electrolytes.

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Surface roughness and gloss of lithium-based glass ceramics after finishing

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Purpose/aim: To assess the efficacy of a dedicated manual finishing and polishing system on roughness and gloss of VITA Suprinity(1) Lithium-Silicate Zirconia-reinforced and e.max CAD(2) lithium disilicate blocks for CAD/CAM systems.

Materials and methods: Twenty-four blocks of VITA Suprinity and 24 blocks of e.max CAD for CAD/CAM systems were cut in a wedge shape using a .cdt file for 3.88 InLab software(3) and an InLab MC-XL milling unit(3). After crystallization, the 24 VITA Suprinity wedges (GROUP A) were randomly divided into 4 subgroups according to the finishing procedure: Group A.1 = VITA Suprinity Polishing Set Clinical used for 30 s(1). Group A.2 = VITA Suprinity Polishing Set Clinical used for 60 s. Group A.3 = VITA AKZENT Plus PASTE(1); Group A.4 = VITA AKZENT Plus SPRAY(1). Similarly to VITA Suprinity, the 24 e.max CAD wedges (Group B) were randomly divided into 4 subgroups according to the finishing procedure Group B.1 = Optrafine Ceramic Polishing System(2) used for 30 s. Group B.2 = Optrafine Ceramic Polishing System used for 60 s. Group B.3 = IPS e.max CAD Crystall./Glaze paste(2); Group B.4 = IPS e.max CAD Crystall./Glaze(2). For each group, 5 wedges were used for roughness and gloss measurements. As both sides of wedges underwent the test, a total of 10 surface for group were treated ($n = 10$). After milling, Gloss was assessed by mean of a Gloss Meter in Gloss Units (Gu) while Roughness was assessed by mean of a Prophilometer (Ra). Gloss and Roughness measurement were then repeated after the finishing/polishing procedure (AF) and compared with the after-milling (AM) data to calculate the differences. Results (Gu and Ra) were analyzed by applying two separate 2-Way ANOVA for gloss and roughness measurement respectively, followed by Tukey's *t*-tests ($\alpha \leq 0.05$) (1. VITA Zahnfabrik, Bad Sackingen, Germany; 2. Ivoclar Vivadent AG, Schaan, Liechtenstein; 3. Sirona Dental, Bernsheim, Germany).

Results: The calculated ΔGu and ΔRa and the statistical significance are reported in the table.

Conclusions: Manual finishing and polishing for 60 s and glazing paste are the most effective procedures in lowering

the roughness of CAD-CAM silica-based glass ceramics. Manual finishing and polishing for 60 s allows milled silica-based glass ceramics to yield the higher gloss. VITA Suprinity displayed higher polishability than IPS e.max CAD. Silica-based glass ceramics restorations can achieve clinically acceptable roughness and gloss comparable to that of enamel by either manual finishing and polishing or glazing, even though some systems proved to be more efficient.

	Gloss (GU)						Sign.	Roughness (μm)						Sign.
	VITA Suprinity			IPS e.max CAD				VITA Suprinity			IPS e.max CAD			
	Mean	SD	Sign.	Mean	SD	Sign.		Mean	SD	Sign	Mean	SD	Sign	
30 s Polishing	49.0 b	6.2	c	63.1 a	12.1	a	B	0.69 a	0,15	b	0,62 a	0,21	a	BC
60 s Polishing	85.0 a	12.9	a	65.8 b	12.4	a	A	0.37 a	0.08	a	0.53 b	0.13	a	A
Glazing paste	72.2 a	10.6	ab	48.3 b	9.5	b	B	0.42 a	0.12	a	0.66 b	0.15	a	AB
Glazing spray	69.9 a	9.4	b	54.9 b	13.9	a	B	0.64 a	0.31	b	0.91 b	0.21	b	C
Sign.	A			B				A			B			

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Proteolytic activity profile of human dentinal fluid

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Purpose/aim: The dentinal fluid is a unique entity of the pulp-dentin complex that forms a virtual communication of the pulp with different regions of dentin, allowing both inward and outward transportation of nutrients and others molecules between different compartments the pulp-dentin complex. The present study aimed to investigate the presence of proteolytic activity in the dentinal fluid.

Materials and methods: Coronal occlusal dentin slices from freshly extracted sound human third molars ($n = 3$) were trimmed and centrifuged at 2750 × *g* for 30 min at 4 °C in order to collect a pool of dentinal fluid. The hydrolysis of selective fluorogenic substrates was monitored in spectrofluorometer (Hitachi F-2500) under time course scan. Metallopeptidase activity was verified in 100 mM Tris buffer (pH 7.4) containing 5 μM of the substrate ABZ-GPQGLAGQ-EDDnp at 37 °C (excitation wavelength: 320 nm; emission wavelength: 420 nm). For cysteine peptidases, the proteolytic activity was evaluated in 100 mM sodium acetate buffer (pH 5.5) in the presence of 10 μM of the substrate Z-FR-MCA after activation with 1 mM dithiothreitol (DTT) for 5 min at 37 °C (excitation wavelength: 380 nm; emission wavelength: 460 nm). The substrates hydrolyses were performed in the absence or presence of specific metallopeptidases and cysteine peptidases inhibitors, 1,10-Phenanthroline and E-64, respectively. The assay was performed under favorable conditions to detect the activity of these proteases. In addition, conventional zymography was performed to verify the gelatinolytic activity in the extracted dentinal fluid.

Results: Under the tested conditions, metallopeptidase and cysteine peptidases specific activity was detected using selective fluorogenic substrates and strong inhibition was demonstrated when using 1,10-Phenanthroline and E-64. The preliminary results showed the presence of proteolytic activity in the dentinal fluid, indicating that, for sound human dentin matrices, the activity of metallopeptidases seems to be preponderant over the cysteine peptidases one. In zymographic analysis, it was detected gelatinolytic activity that indicates the presence of MMP-2 pro- and active-form (72- and 66-kDa respectively) and active-form of MMP-9 (86 kDa).

Conclusions: The present study indicated that, as previously observed for dentin, different families of proteolytic enzymes seem to contribute with the enzymatic profile of the dentinal fluid.

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WITHDRAWN

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Stress reduction and toughening of dental composites with thiourethane-silanized fillers

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Purpose/aim: Thiourethane oligomers have been shown to significantly reduce stress and increase fracture toughness in dental composites. The objective of this study was to functionalize the surface of inorganic fillers to improve the distribution of the oligomer in the composite, and harness the same benefits with a lower concentration of the oligomer.

Materials and methods: Oligomers were synthesized by combining a difunctional isocyanate (1,6-hexanediol-diisocyanate – HDDI; 1,3-bis(1-isocyanato-1-methylethyl)benzene – BDI or 4,4′-Diisocyanato-methylenedicyclohexane – DESM) and (3-Isocyanatopropyl)triethoxysilane, with pentaerythritol tetra-3-mercaptopropionate (PETMP) or trimethylol-tris-3-mercaptopropionate (TMP), at 1:2 isocyanate:thiol. Oligomers were attached to 0.7 μm Ba glass via ethanol-based silanization reaction. Fillers with no silane (UNS) or silanized with conventional methacrylate silane (SIL-MA) were used as controls. Filler particles were added at 50 wt% to BisGMA-UDMA-TEGDMA (50–30–20 wt%). Irgacure 819 was used as the photoinitiator (0.1 wt%). Composites were photoactivated at 800 mW/cm^2 (320–500 nm). Near-IR was used to evaluate conversion in depth (4.5 mm) and polymerization kinetics (degree of conversion, DC, and maximum rate of polymerization, Rmax). Fracture toughness (FT) was evaluated in 2 mm × 5 mm × 25 mm notched specimens, light transmission (ΔLT) by MARC and, polymerization stress (PS) using the Bioman. Data was analyzed with one-way ANOVA and Tukey's test (95%).

Results: Results are shown in Table 1. In general, composites prepared with the thiourethane-silanized filler presented higher or at least similar DC compared to the controls, reached at similar rates. Interestingly, the polymerization stress was significantly reduced by up to 35% with all TMP-containing compositions and PETMP-HDDI. The fracture toughness significantly increased for the modified groups (with one exception). In some cases, the increase in FT was almost two-fold. Light transmission at high irradiance (not shown) was not significantly affected by the addition of thiourethane silanes, and the conversion at 4.5 mm was statistically similar for all groups, except for the SIL-MA group, which showed a lower decrease on the bottom in relation to the top of the specimen.

Conclusions: Overall composites filled with thiourethane-silanized inorganic particles showed up to 35% lower stress while improving fracture toughness by two-fold. This was obtained with no prejudice to the viscosity of the material and with similar photoactivation protocols as used by the dentist.

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Table 1

Groups	DC (%)	Rmax (% s^{-1})	PS (MPa)	FT (MPa m$^{1/2}$)	ΔDC (%)
SIL-MA	44.04 (1.07)^{b}	4.16 (0.11)^{b}	2.66 (0.06)^{a}	1.54 (0.11)^{b}	2.0^{b}
UNS	42.06 (2.68)^{b}	6.16 (1.41)^{ab}	2.67 (0.19)^{a}	0.83 (0.09)^{b}	9.0^{a}
TMP:BDI	50.38 (0.62)^{ab}	4.14 (0.39)^{b}	2.00 (0.16)^{b}	1.46 (0.33)^{b}	8.0^{a}
TMP:HDDI	48.06 (1.09)^{ab}	5.28 (0.52)^{b}	1.75 (0.21)^{b}	2.17 (0.16)^{a}	7.0^{a}
TMP:Desm	50.77 (1.08)^{a}	8.84 (1.42)^{a}	1.77 (0.18)^{b}	1.94 (0.33)^{a}	5.0^{a}
PETMP:BDI	44.42 (0.97)^{b}	5.35 (0.27)^{b}	2.06 (0.30)^{ab}	2.86 (0.75)^{a}	6.0^{a}
PETMP:HDDI	49.18 (0.96)^{ab}	6.73 (0.16)^{ab}	1.94 (0.32)^{b}	2.38 (0.56)^{a}	8.0^{a}
PETMP:Desm	48.55 (0.81)^{ab}	5.58 (1.94)^{b}	2.26 (0.08)^{ab}	2.21 (0.28)^{a}	8.0^{a}

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HF concentration and etching times on lithium disilicate glass-ceramic

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Purpose/aim: The aim of this study was to evaluate the influence of different concentrations of hydrofluoric acid (HF) associated with varied etching times on the surface morphology/microshear bond strength (μSBS) of a lithium disilicate glass-ceramic to a resin cement.

Materials and methods: Two-hundred and seventy five ceramics blocks (IPS e.max Press (EMX), Ivoclar Vivadent) of 8 mm × 8 mm × 3 mm thickness, were randomly distributed into 5 groups according to the HF concentration ($n = 50$): 1%, 2.5%, 5%, 7.5% and 10%. Further random distribution to subgroups was performed according to the etching times ($n = 10$): 20, 40, 60, 120 and 20 + 20 s. After etching, all blocks were treated with a silane-coupling agent followed by a thin layer of an unfilled resin. Three resin cement cylinders (∅ = 1 mm) were made on each EMX surface and then stored in deionized water at 37 °C for 24 h before testing. The μSBS was carried-out in a universal testing machine at a crosshead speed of 1 mm/min until failure. Data were submitted to two-way ANOVA and the Tukey's post hoc test ($\alpha = 0.05$). The failure modes of all debonded specimens were analyzed under optical microscopy at 40× magnification. One representative EMX sample was etched according to each subgroup description and evaluated using scanning electron microscopy (SEM) for characterization of the etched morphology.

Results: The values of μSBS (MPa) for 5% (27.8 ± 1.6), 7.5% (28.1 ± 1.6) and 10% (31.1 ± 1.7) HF concentrations provided significantly higher μSBS values than 1% (10.2 ± 2.1) and 2.5% (16.8 ± 2.6) ($p < 0.05$), regardless the etching times. For 1% and 2.5% HF, the etching times from 40 s to 120 s increased the μSBS values compared to 20 s ($p < 0.05$), but they did not differ within the 5%, 7.5% and 10% HF groups ($p > 0.05$). The effect of re-etching was more evident for 1% and 2.5% HF ($p < 0.05$).

Conclusions: Different etching times/HF concentrations have directly influenced on the bond strength and surface morphology of EMX.

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Mechanical, physicochemical and biological properties of experimental EGCG based adhesive

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Purpose/aim: This study evaluated the mechanical, physicochemical and biological properties of an experimental adhesive with EGCG incorporation in different concentrations.

Materials and methods: The first phase of the experiment involved the manipulation of a model adhesive (ADS) in relation BisGMA 55%/45% HEMA (mol), 0.5% camphorquinone and 0.5% DMAEMA; and incorporation of EGCG 0.5%, 1.0% and 1.5%. The conversion kinetics of the adhesive monomers was evaluated by micro-Raman Spectroscopy after 40 s of light curing LED (1100 mW/cm^2). The second phase involved the mechanical tests: flexural strength (RF) (load cell 10 kg, F 0.5 Nm/s, speed 0.5 mm/min), diametrical tensile strength (TD) (diametrical load 1000 kg); physicochemical analysis polymerization shrinkage (CP) (through AcuvolTM apparatus); and organic: cytotoxicity assay (CI) (ADS were serially diluted in DMEM and exposed to PF fibroblastos. Cell viability was measured photometrically by the SRB colorimetric assay. The degree of conversion and cytotoxicity were subjected to the Kruskal–Wallis test. The other tests were submitted to ANOVA and Tukey factor-1 ($\alpha = 5\%$).

Results: Raman: EGCG1.5% – 99,11a; EGCG1.0% – 99,02a; EGCG0.5% – 96,58ab; Control (C) – 85,47b. RF, TD and CP showed no difference between groups (RF: $p = 0.19$; TD: $p = 0.38$; CP: $p = 0.21$). CI: EGCG1.5% – 0,27ab; EGCG1.0% – 0,67a; EGCG0.5% – 0,61a; C – 0,06b. The incorporation of EGCG at concentrations of 0.5% and 1.0% reduced cellular cytotoxicity of the adhesive. The incorporation of EGCG at concentrations 0.5%, 1.0% and 1.5% increased the degree of conversion and kept the mechanical and physico-chemical properties of the experimental adhesives.

Conclusions: Since EGCG is MMP inhibitor, the experimental adhesives are promising to increase the longevity of the restorations.

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Effects of solvents on size-exclusion characteristics of collagen

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Purpose/aim: Resin infiltration through demineralized collagen is a critical step during dentin bonding. Dental monomers are not able to completely displace bound and unbound water after acid etching of dentin. The use of organic solvents may allow collagen molecules to come close enough and modify size-exclusion characteristics of type I collagen. This study evaluated the size-exclusion characteristics of dentin collagen, using a gel permeation-like column chromatography technique, filled with dentin powder as the stationary phase.

Materials and methods: Dentin powder was produced from 600 bovine teeth and inserted in a glass chromatography column. Blue Dextran, BisGMA, TEGDMA and HEMA were individually tested using mineralized powder. Tris–HCl buffer was continuously pumped into the column and 1 mL samples collected every 5 min and read in UV spectrophotometer to obtain the elution peaks. Then, the powder was demineralized in 10% phosphoric acid and the tracers were tested again in buffer. Subsequently, the powder was saturated with 100% ethanol, 85% ethanol, 75% ethanol or 100% acetone and new elution volumes were established. The elution volumes for the same solution (buffer, ethanol or acetone) and powder condition (mineralized or demineralized) were submitted to multiple ANOVA test, complemented by Tukey's test. Data for the same tracer and mobile phase (solution) were evaluated by *t*-test, at a significance level of 5%.

Results: For all the tracers, mineralized dentin presented the lowest elution volumes. When demineralized powder was used, Blue Dextran eluted faster than HEMA, TEGDMA and BisGMA. HEMA infiltration was not affected by the mobile phase. TEGDMA showed higher elution volumes when collagen was saturated with buffer, 100% ethanol and 75% ethanol. BisGMA infiltration was favored by 100% ethanol and 100% acetone.

Conclusions: The elution volumes of different sized test molecules studied in both buffer-saturated dentin, and again in ethanol or acetone-dehydrated dentin powder, showed that resin monomers can diffuse into both hydrated and dehydrated collagen molecules. The results confirm that adhesive

monomers can permeate tightly-bound water in ethanol or acetone-saturated collagen molecules during infiltration. Generally, 100% ethanol favored monomer elution through demineralized collagen.

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Role of proteoglycans on dentin biochemical and biomechanical properties

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Purpose/aim: Proteoglycans (PGs)/glycosaminoglycans (GAGs) are crucial components of organic matrices of collagen-based tissues, such as dentin. As other organic components, PGs/GAGs may significantly influence the restorative adhesive therapies in dentin. This study compared different protocols for selective PGs/GAGs removal from dentin and the influence of PGs/GAGs on tissue's mechanical behavior and biostability.

Materials and methods: PGs/GAGs from demineralized dentin matrix (DDM) blocks (1.5 mm × 1.5 mm × 0.5 mm) were removed by incubation with chondroitinase ABC from Proteus vulgaris (c-ABC) or trypsin from bovine pancreas TPCK treated (Try) for 48 h at 37 °C. Control group was incubated with distilled water under same conditions. GAGs release was quantified by 1,9-dimethylmethylene blue (DMMB) assay in enzymes supernatant. Remaining GAGs in dentin after enzymatic treatments were determined by DMMB assay after complete digestion with 50 μg/ml proteinase K. Similarly, after PGs/GAGs removal, DDM was incubated with collagenase (100 μg/ml of collagenase from *Clostridium hystoliticum* for 24 h at 37 °C) to determine biostability by quantification of hydroxyproline release. Differences in dry weight of DDM before and after PGs/GAGs digestion were also assessed. Removal of GAGs/PGs in cABC- and TRY-treated DDM was assessed by cupromeronic blue staining and observed under TEM. To evaluate the influence of PGs/GAGs on mechanical properties, hour-glass shape slabs obtained from mid-coronal dentin were demineralized and digested with the same enzymatic methods. Changes in the ultimate tensile strength (UTS) and energy to fracture at different tubule orientations [parallel (PL) × perpendicular (PP)] were investigated. Data were statistically analyzed by two-way and one-way ANOVA followed by the appropriate post-hoc tests ($\alpha = 0.05$).

Results: C-ABC and Try released similar amounts of GAGs from DDM ($p = 0.276$), while remaining GAGs were quantified only for c-ABC. Complete removal of GAGs was observed in TEM for both enzymatic methods. Try caused significant reduction in percentage of weight and faster degradation of DDM than control ($p < 0.001$), but no differences were observed between c-ABC and control. Try digestion significantly decreased UTS when test was performed PP to tubule orientations ($p < 0.002$). Under the same condition, PGs/GAGs removal caused pronounced modifications in energy to fracture.

Conclusions: PGs/GAGs play an essential role in the dentin mechanical properties, significantly affected by the dentin tubule orientation. Modifications in PGs/GAGs and on their interaction with collagen fibrils may influence the dentin collagen biodegradation. The role of such proteins on dentin fracture and proteolysis is of great clinical relevance to both physiological and pathological conditions.

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Functionalized pink manganese-doped alumina ceramic pigments applied in prosthetic dentistry

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Purpose/aim: This study was designed to synthesize, functionalize and characterize pink manganese-doped alumina ceramic pigments. The hypothesis that functionalized ceramic pigments would render pink coloration to a translucent acrylic resin and not jeopardize its mechanical properties was tested.

Materials and methods: Pink powders of 1% and 2% manganese-doped alumina (Al_2O_3:Mn) were prepared by means of a polymeric precursor method. The chemical and microstructural characteristics of the pigments were evaluated by using XRD, and SEM analyses. Pigment particles were functionalized with a silica coating method, followed by silanation prior preparation of acrylic resin-based composites (5 wt.%). The composites (Pig. 1% and Pig. 2%) and acrylic resin controls (Pink and Translucent) were evaluated with regards to their optical properties (CIEL*a*b* color coordinates) and mechanically tested in three-point-bending mode.

Results: Results of the chemical and microstructural analyses confirmed the successful synthesis of the pink manganese-doped alumina ceramic pigments. The microstructure of the pigment particles showed nanocrystals (~55 nm) of manganese-doped α-alumina clustered into irregular porous micrometric particles (max. 60 μm). The composites formulated with the functionalized pigment powders showed no difference in flexural strength, flexural modulus, and work-of-fracture compared to control acrylic resins (pink and translucent). The tested hypothesis was confirmed as functionalized pigment powders yielded adequate pink coloration to a translucent acrylic resin and did not jeopardize its mechanical properties.

Conclusions: The method to prepare pink manganese-doped alumina ceramic pigments presented here is a promising novel strategy for the development of colored polymeric composites.

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Cytotoxic effects of silver tungstate microcrystals on fibroblast human cells

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Purpose/aim: The oral cavity is a complex ecosystem of over 700 microbial organisms including bacteria and fungi. Microorganism grows on natural or artificial surfaces as biofilm, but when the delicate balance of the existing microbial ecosystem is disrupted, these surfaces become the base for colonization and invasion of pathogens. Researchers have studied and modified the dental materials surfaces with silver nanoparticles (AgNPs), which although provide excellent antimicrobial properties, it is toxic to mammalian cells in high concentration. Thus, to improve the antimicrobial properties and reduce the amount of silver available, our team has synthetized silver tungstate (Ag_2WO_4) as micro crystals. Our preliminaries data showed promising results in relation to antimicrobial activity, however, little is known about the cytotoxicity of these compounds. In this study, the focus was to develop a better understanding of the pertinent properties of this new component, specifically if this microcrystal influences the viability and proliferation of human fibroblasts (FGH) cells cultured in monolayer and in 3-D collagen matrices.

Materials and methods: To carry out the experiments, lowest concentration of Ag_2WO_4 that prevents visible growth of fungi planktonic cells was defined as our test concentration ranging from 0.781 (C1), 7.81 (C2) to 78.1 (C3) μg/mL. Complete medium (C4) was used as a negative control and lysis buffer (LB) as a positive control (C5), equating to 100% cell death. The effect of the microcrystal concentration on cell morphology, remodeling and proliferation of human FGH was evaluated.

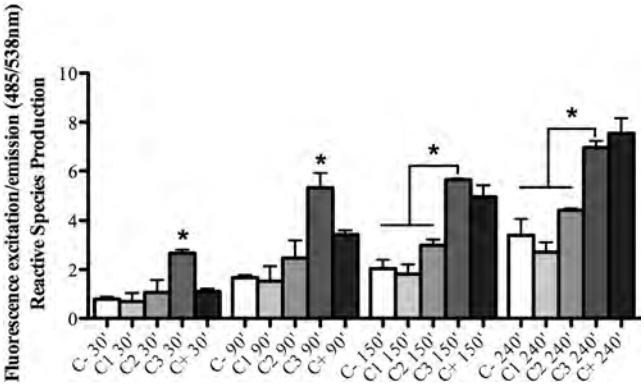

Fig. 1 – 3-D collagen cell culture system. Data are shown as the mean ± SD, *$p < 0.05$ was considered statistically significant.

Results: One way analysis of variance (ANOVA) was employed with a Tukey's posthoc test using Graph-Pad Prism version 5.0c. The quantitative and qualitative results showed that Ag_2WO_4 did not affect mitochondrial enzymatic activity of FGH cells cultured in monolayer and in 3-D collagen matrices (Fig. 1). Furthermore, generation of reactive oxygen species was not detected in the minimum fungicidal concentration.

Conclusions: The creation of new materials has always been attractive to researchers, mainly by the possibility of treat a disease. From our point of view, we believe that the Ag_2WO_4 not only has outstanding potential in biological applications, but is also extended to other medical devices outside the field of dentistry where the impact of altering tissue attachment is paramount to restore normal healthy function.

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Polymerization stress, gap formation and bacterial infiltration in bulk-fill restorations

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Purpose/aim: The aim of this study was to evaluate polymerization stress, internal gap formation, bacterial infiltration and secondary caries development in bulk-fill composites restorations.

Materials and methods: Six resin-based composites were investigated: two conventional materials (Filtek Supreme Ultra (FI) and Beautifil II (BI)) applied in increments, two flowable bulk-fill composites (Filtek Bulk Fill Flowable (FBF) and Beautifil Bulk Flowable (BBF)), and two high-viscosity bulk-fill composites (Filtek Bulk Fill Restorative (FBR) and Beautifil Bulk Restorative (BBR)). Polymerization stress was determined using composite ($n = 5$) bonded to acrylic rods and an extensometer attached to a universal testing machine, monitored for 10 min from beginning of light-activation. Standardized class I preparations (4-mm-depth) were made in decontaminated molars and restored with each material ($n = 4$). During restorative procedures selective enamel etching and dentin self-etching was applied with respective adhesive systems: Single Bond Universal for 3M composites, and FL-Bond II for Shofu products. Restored teeth were sterilized using UV light and left in Streptococcus mutans culture for 14 days. After this period, samples were gram positive stained. Restorations were cross-sectioned and bacterial infiltration was evaluated using laser confocal microscopy. Secondary caries development was observed using optical microscopy. Gap formation was analyzed through epoxy resin replicas of restorations using scanning electron microscopy. For statistical analyses, polymerization stress and gap formation data were compared using one-way ANOVA and Tukey tests ($\alpha = 0.05$).

Results: Polymerization stress and percentages of discontinuous interfaces (total gap formation length as a function of total length of the interface) are presented in Table 1.

Table 1 – Mean (standard deviation) of maximum polymerization stress (MPa) and gap formation (% length of entire internal interface).

Material	Polymerization stress (MPa)	Gap (% total interface length)
FI	3.29 (0.28) c	49.63 (4.11) ab
FBF	3.52 (0.15) bc	50.47 (5.06) ab
FBR	3.25 (0.21) c	38.78 (18.35) b
BI	3.90 (0.27) ab	67.51 (6.74) a
BBF	4.08 (0.10) a	69.51 (7.85) a
BBR	3.08 (0.29) c	41.67 (14.08) b

Statistically significant differences among the materials were identified. Composites with higher polymerization stress demonstrated more internal gap formation, independent of restoration technique. Gaps were mostly located at line angle areas and pulpal wall for all materials. Also, flowable bulk-fill composites presented more gaps in enamel margins of surrounding surfaces. Bacteria were able to infiltrate in marginal gaps. No difference was observed in secondary caries development among investigated groups.

Conclusions: Internal gap formation relies on the composite polymerization stress, independent of restoration technique. All materials presented bacteria infiltration through the gaps with no difference on secondary caries development.

Within a column, means followed by the same letter are not statistically different ($p > 0.05$). $N = 4$ specimens/group.

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Effect of doxycycline incorporated within dental adhesives on tooth-restoration interface

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Purpose/aim: The purpose of this study was examined the effect of the incorporation of different dosages of doxycycline in an experimental dental adhesives on nanomechanical properties of adhesive interface.

Materials and methods: Five etch-and-rinse experimental adhesive systems were formulated according to the doxycycline weight percentage (wt%): 0.05% (EXP1), 0.1% (EXP2), 0.5% (EXP3) and 1% (EXP4). Fifteen caries-free human third molar teeth were used in this study. Acid etching was applied on dentin for 15 s; rinse with water for 30 s; excess moisture was removed with absorbent paper, two consecutive coats of the adhesive was applied, gentle air stream was applied for 5 s and the adhesive was light-activated for 10 s using a Bluephase unit (~1200 mW/cm^2). The composite resin (Filtek Z350, shade A2; 3M ESPE) was placed in 2 mm-thickness increments, and each composite layer was light-activated for 20 s. Teeth were stored in Hanks' balanced salt solution at 37 °C for 24 h. After the storage teeth were longitudinally sectioned in the mesio-distal direction through the bonded interface to obtain 1.5-mm-thick bonded slices. Two slices from the middle the specimens were selected for the nanoindentation test. The dentin-bonded slices were embedded in resin epoxy, the surface was polishing first using Silicon Carbide with decreasing abrasiveness (600, 1000, 1200, 1500 and 2000). The samples were also polished using soft discs with diamond suspensions of 9, 6, 3, 1 and 0.5 μm. The nanoindenter Hysitron was used in Load of 1000 μN and a standard trapezoidal load function of 5–2–5 s. 5 indentations was performed for the dentin, hybrid layer and adhesive and the nanohardness and Young's modulus of each area were computed according to the Oliver and Pharr's method.

Results: There were no differences between groups for Young's modulus values for adhesive. Control group and experimental groups showed higher values for hybrid layer nanohardness. Experimental groups showed increase of the Young's modulus of hybrid layer. Nanohardness and Young's modulus of dentin was not affect by incorporation of MMP inhibitor.

Conclusions: The incorporation of doxycycline at subantimicrobial dosagen might display a valuable therapeutic role of MMPs based in the increase of the Young's modulus of hybrid layer.

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Laser phototherapy on rat excisional wound model: Biomechanical analysis

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Purpose/aim: The last two phases of wound healing consist of tissue formation and remodeling. In these phases the injured skin tissue increases its tensile strength. However, some authors reports that, even at the third week, the tissue tensile strength reaches only 20% of that to be achieved at the end of the wound healing. The aim of this study is to evaluate the load at initial tear of the wounded skin, as a measure of the tensile strength, of rats treated with Low Level Laser Therapy (LLLT), compared with controls. In addition, clinical, histological and immunohistochemical analyzes were performed.

Materials and methods: Wistar rats, weighing 250–350 g each, were divided into 2 groups: C (Control group, which did not receive any treatment) and L (which received 5 sessions of LLLT at 660 nm/5 points of 10 J/cm^2/40 mW/0.04 cm^2 of the spot area). Standardized 6 mm diameter wounds were created on the dorsal skin of rats and biopsied at 7 (only for clinical, histological and immunohistochemical analysis) and 10 and 14 days (for clinical, histological, immunohistochemical and biomechanical analysis) post-wounding.

Results: The *t* test showed difference ($p \leq 0.05$) in tensile strength between the wounds of LLLT (6.0 ± 0.6 N) and control (4.8 ± 0.7 N) groups at the 10th day of the experiment, however, no statistical difference was found at the 14th day. In addition, differences also were found in clinical, histological and immunohistochemical analyzes in favor of the laser group, supporting the results of biomechanical analysis. At the 7th day, clinical analysis (% of the remaining wound) showed difference ($p \leq 0.05$) between LLLT (6.9 ± 5.7%) and

control (22.5 ± 13.8%) groups. Ten days after initial wounds are placed; the laser group still has significantly better results than the control group, for clinical analysis. However, at 14th day of the experiment, all animals did not show any remaining wound. Histological examination demonstrated that, at 10th day, the LLLT group (47.2 ± 9.2%) showed a significant increase of collagen formation ($p \leq 0.05$) when compared with the control group (25.7 ± 9.0%). The immunohistochemical analysis showed a significant increase in % of cytokeratin 10, for the LLLT group, compared to the control group, on days 7 (44.9 ± 4.2 vs. 21.2 ± 3.8%), 10 (59.4 ± 6.4 vs. 44.2 ± 4.3%) and 14 (64.8 ± 9.8 vs. 52.2 ± 14.0%) ($p \leq 0.05$).

Conclusions: The Low Level Laser Therapy improved the tensile strength of the wounded tissue, as well as, decreased lesion area, increased collagen formation and showed an advanced wound healing process by histological and immunohistochemical analysis.

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Comparison of different titanium alloys for biomineralization and biological response

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Purpose/aim: A challenge nowadays in implantology research is to find a new titanium alloy without cytotoxicity that will be able to improve the biological tissue response by changes on the surface topography that has the same efficiency than conventional alloy. The aim of this study was to evaluate the behavior of osteogenic cells on porous disks with different alloys made by powder metallurgy technique.

Materials and methods: The disks were divided in three groups, the control group with Ti-6Al-4V (Group 1) disks and the two tests groups with Ti-35Nb (Group 2) or Ti-35Nb-7Zr-5Ta (Group 3) disks. Human mesenchymal stem cells and mouse mesenchymal stem cells were used.

Results: Scanning electron microscopy images of the groups with and without cells were obtained. The alkaline phosphatase (ALP) activity was evaluated at 3, 7 and 14 days; the formation of nodules of mineralization at 14 and 28 days and quantitative real-time PCR was used to measure the mRNA levels from osteogenic markers at 7 and 14 days. ALP activity was significantly greater at 14 days in Group 2 and 3. At 28 days the calcium content (mineralization) was improve in Group 3. At seven and 14 days there was an 8-fold and a 2-fold increase for ALP gene compared to control in Group 1. In Group 2 there was a 4-fold increase compared to control at day 7 and in Group 3 there was a 4-fold at 7 and 14 days for ALP gene.

Conclusions: Different alloys disks showed to have the same biological response as the conventional in in vitro studies. The use of different metals with lower cytotoxicity can improve the use of dental implants and reduce future complications that have been related to the conventional alloy.

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Effect of titanium nanotopography on mobilization of mesenchymal stem cells

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Purpose/aim: The characterization of the dental implant surface treatment is one of the factors that may affect and/or increase the degree of osseointegration. The proliferation and differentiation of cells, and modified tissue, eventually results in bone formation in direct contact with the surface of the implant. The nanotopography has a great influence on the differentiation of mesenchymal stem cells (MSCs) into osteoblasts. The aim of this study was to investigate effect of nanotopography of titanium that affect the osteogenic potential in vivo.

Materials and methods: Titanium disks were treated with a combination of H_2SO_4/H_2O_2 or HCl/H_2O_2 at varying concentrations and treatment times. After harvesting human mesenchymal stem cells, on the different surfaces, the alkaline phosphatase (ALP) activity and gene expression was measured. For in vivo tests the Tg(Sp7/mCherry)2Pmay/J mouse was used in this experiment. Implants were placed in the distal femur, accessed through a medial parapatellar arthrotomy. After locating the femoral intercondylar notch, the femoral intramedullary canal was manually reamed with a 25 gauge needle. A cpTi grade IV wire (diameter 0.9 mm) prepared with smooth or nanoscale topography was placed. Each femur received a different surface. For mobilization of MSCs by flow cytometry and mRNA evaluation, tissues were collected at 1, 3, 5 and 7 days following surgery. For NanoCT and histomorphometric analysis samples were collected at 7, 14 and 21 days.

Results: Flow Cytometry showed an increase on MSCs committed to osteoblast differentiation on the Nano surface with 3, 5 and 7 days. These were also confirmed by mRNA expression of inflammatory and osteogenic genes that showed a pattern on nanoscale surface compared to control. At day 1 the Il1b, ARG1 and iNOS2 genes showed a 3.5-, 2.5- and 2.5-fold increase, respectively, on the nano surface. At later stages

The Nano surface showed at day 7 a 2.5-, 11.5-, 4.5-, 2.5-, 2.5- and 4-fold increase, respectively, on ALP, OCN, Col1a1, Satb2, RUNX2, BSP and OSX genes.

Conclusions: In vitro and in vivo results demonstrated increased osteogenic differentiation that could lead to osseointegration with an early inflammatory response when using an implant surface with nanotopography treatment.

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Systematic review of dental pulp capping materials

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Purpose/aim: The aim of this systematic review was to analyze the current trends and future perspectives of dental pulp capping materials through an analysis of scientific and technological data.

Materials and methods: This study is reported in accordance with the PRISMA Statement. Eight papers databases were screened: PubMed (MedLine), Lilacs, IBECS, BBO, Web of Science, Scopus, SciELO and The Cochrane Library. Additionally, the search of patent applications was conducted in Questel Orbit (Paris, France), USPTO, EPO, JPO, INPI and Patentscope. A total of 716 papers and 83 patents were included.

Results: Calcium hydroxide was the main type of material studied, especially for direct pulp capping, followed by MTA. Animal experiments were predominant in direct pulp capping analysis, while clinical studies were the main study design to indirect pulp capping and pulpotomy. Patents related to adhesives or resins increased from 1998 to 2008, while in the last years it was observed a major increase in bioactive materials (containing bioactive proteins), MTA and MTA derived materials (calcium silicate, calcium phosphate and calcium aluminate based cements).

Conclusions: It was possible to obtain a scientific and technological overview of pulp capping materials. MTA have shown favorable results in vital pulp therapy that seems to surpass the disadvantages of calcium hydroxide. Recent advances in bioactive and MTA derived materials have shown promising results that could improve biomaterials used in vital pulp treatments (Fig. 1).

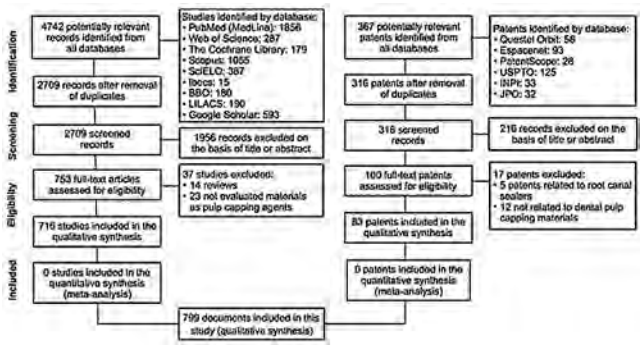

Fig. 1

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Transparency of highly viscous conventional glass ionomers after long-term immersion

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Purpose/aim: The objective of this study was to examine the transparency of highly viscous conventional glass ionomers after long-term immersion in water.

Materials and methods: Five commercially available glass ionomers (GlasIonomer FX ULTRA, Shofu, A; Fuji IX GP EXTRA, GC, B; Riva self cure, SDI, C; ChemFil Superior, DENTSPLY, D and Ketac Molar Easymix, 3M ESPE, E) were used in this study. Disc specimens (12 mm in diameter, 1 mm thick) were prepared according to each manufacturer's recommended procedure (Cement A: P/L = 2.8/1.0, Cement B: P/L = 3.4/1.0, Cement C: P/L = 2.8/1.0, Cement D: P/L = 3.2/1.0 and Cement E: P/L = 4.5/1.0). Ten minutes after the start of mixing, L^*, a^* and b^* of each cement in the black or white background were measured using a spectrocolorimeter as the 'Baseline'. After that, the specimens were stored in distilled water at $37 \pm 2\,^{\circ}C$, or placed in container maintained at $37 \pm 2\,^{\circ}C$ and $95 \pm 5\%$ r. h. up to 3 months. The TP values of each glass ionomer were calculated from L^*, a^* and b^* values and were determined at 24 h, 1 week, 1 month and 3 months after the start of mixing ($n = 5$). The results were statistically compared using a two-way ANOVA followed by Scheffè's test at $\alpha = 0.05$.

Results: Table 1 shows the measured TP values (mean ± sd, $n = 5$) for the cements stored in distilled water at $37 \pm 2\,^{\circ}C$. Cement A at Baseline had significantly greater TP values compared to the other cements ($p < 0.05$) and revealed no significant changes in TP values at any storage time period ($p > 0.05$). Cements B and C showed no significant differences in TP values after 24-h immersion ($p > 0.05$). Statistically no significant differences in TP values of Cements D were observed after 1 week ($p > 0.05$). The TP values for Cement E increased significantly with time elapsed after mixing ($p < 0.05$), however no significant changes in the TP values were found after 1 month ($p > 0.05$).

Table 1

Immersion time	Cement A	Cement B	Cement C	Cement D	Cement E
Baseline (10 min)	9.6 ± 0.9	5.9 ± 0.6	3.4 ± 0.3	3.9 ± 0.6	4.3 ± 0.2
24 h	8.9 ± 0.5	8.6 ± 0.5	5.9 ± 0.5	7.1 ± 1.2	5.2 ± 0.3
1 week	9.3 ± 0.5	9.2 ± 0.4	7.3 ± 0.4	8.3 ± 0.6	6.1 ± 0.3
1 month	8.5 ± 0.3	9.3 ± 0.4	7.9 ± 0.6	8.3 ± 0.7	6.9 ± 0.4
3 months	8.5 ± 0.5	8.8 ± 0.6	7.6 ± 0.6	7.7 ± 0.5	7.1 ± 0.2

Conclusions: All highly viscous conventional glass ionomers for filling examined exhibited comparable levels of transparency after 1 month immersion in water.

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Tear energy of dentin matrices depleted of non-collagenous proteins

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Purpose/aim: This preliminary study investigated the effect of non-collagenous proteins (NCPs) on the tear energy of demineralized dentin. The energy to tear was estimated using Mode III trouser-shaped tear specimens.

Materials and methods: Ten extracted sound human molars were sectioned into 6–9 slices from coronal dentin with a thickness range of 0.43–0.45 mm and standardized length and width of 6 mm and 2 mm, respectively. Specimens were distributed into two groups randomly ($n = 30$): demineralized dentin (control) and trypsin treated demineralized dentin (NCPs-depleted). The disks were demineralized in 25% EDTA for 14 d and trypsin-treated specimens were incubated for an additional 48 h at 37 °C in 1 mg/ml of Trypsin from bovine pancreas in 0.2 M ammonium bicarbonate solution. A central notch of 4.00 mm, using a flat blade, was placed shaping the disk into a trouser-shaped tear specimen. This allowed for guided-propagation of the crack. Once glued to an apparatus, specimen was mode III loading using a 50 N load cell with a cross-head speed of 1.0 mm/min. The tear energy was calculated using the following equation: $T = 2P/t$, where P is the tearing force and t is the thickness of the specimen. Statistical analysis was performed using an unpaired two-tailed t-test at $\alpha = 0.05$.

Results: Only specimens that tore parallel to the notch were included in calculating the tear energy. The tear energy for control and NCPs-depleted groups were 2.50 ± 0.38 kJ/mm^2 and 2.00 ± 0.24 kJ/mm^2, respectively. There was a significant decrease ($p = 0.0011$) in the tear energy with the enzymatic removal of NCPs from dentin matrices by trypsin.

Conclusions: The trouser-shaped tear testing is a promising method to characterize the biomechanical behavior of dentin matrices. Preliminary findings show that NCPs have a significant role in the fracture resistance of sound dentin matrices.

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3D printed *versus* conventionally cured experimental dental composites

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Purpose/aim: In this project we compare the degree of conversion (DC), mechanical and structural properties of 3D printed versus conventionally photocured experimental composites.

Materials and methods: BisGMA and TEGDMA were combined at 70:30 and 50:50 (wt%), using 0.3 wt% Phenylbis(2,4,6-trimethylbenzoyl)phosphine oxine as photoinitiator. Sample groups were determined to compare the effect of 3D printing versus conventional curing based on standard laboratorial procedures, rather than light irradiance or time. To that end, samples measuring 2 mm × 2 mm × 15 mm were fabricated either via Stereolithography 3D printing (405 nm, 120 mW laser at 25 μm increment layers), or conventional photopolymerization (405 nm, 40 mW UV light, two min top and bottom). A FT-IR was used in Near-IR to determine the DC at every 200 μm along the 2 mm height of cross-sectioned specimens. Mechanical properties were evaluated in three-point bending using a universal testing machine to determine flexural strength/modulus and toughness (ISO-4049). Fractured sample surfaces were examined with an SEM. Results were analyzed with ANOVA/Bonferroni test ($\alpha = 0.05$).

Results: Both 70:30 and 50:50 groups had a similar average DC, regardless of the curing method. FTIR maps of the DC of cross-sectioned samples showed similar results for both the 3D printed and conventionally cured samples. The top and bottom regions of the 3D printed 70:30 samples had an average DC of 75 ± 8 and 71 ± 6% respectively, while conventionally cured specimen had a DC of 75 ± 8, and 71 ± 10%, at top and bottom respectively. Flexural strength of the 3D printed samples, for both 70:30 and 50:50 groups, was significantly lower than for conventionally cured specimens. 3D printed 70:30 and 50:50 samples had an average flexural strength of 2.9 0.45 and 2.3 ± 0.34 GPa respectively, while conventionally cured specimens had 3.8 ± 0.32 and 3.3 ± 0.2 GPa, respectively. Toughness values, on the other hand, were consistently higher for 3D printed samples, and averaged 4.85 ± 2.98 and 4.4 ± 1.82 MPa for 70:30 and 50:50 groups respectively. Conventionally photocured 70:30 and 50:50 specimens, on the other hand, had average toughness of 3.95 ± 1.57 GPa and 4.02 ± 0.92 GPa, Toughness values for 3D printed samples, however, were not significantly higher than conventionally photocured composites. SEM images revealed similar fracture patterns with no obvious effect of delamination between printed layers.

Conclusions: Despite the resulting lower flexural strength, 3D printing may be a comparable method to current con-

Table 1

Group	Modulus (GPa)	Peak load (N)	Peak stress (MPa)	Load at yield (kN)	Toughness (MPa)	Degree of convergence
3D 70:30 BisTEG	2.94	37.15	106.74	0.04	4.85	−5.24
	(0.45)	(12.43)	(36.98)	(0.02)	(2.98)	(18.53)
3D 50:50 BisTEG	2.31	34.74	92.62	0.034	4.39	29.38
	(0.35)	(5.08)	(6.98)	(0.01)	(1.82)	(6.42)
Conventional 70:30 BisTEG	3.78	28.02	128.45	0.030	3.96	40.55
	(0.32)	(4.21)	(14.30)	(0.00)	(1.57)	(9.90)
Conventional 50:50 BisTEG	3.26	24.17	119.70	0.024	4.03	55.23
	(0.20)	(3.08)	(5.40)	(0.00)	(0.93)	(5.13)

ventional methods for fabricating indirect dental restorative materials (Table 1).

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Quality of cure in depth of bulk-fill composites: Physico-mechanical properties

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Purpose/aim: Bulk-fill composites are an emerging class of resin-based composites, claimed to enable the restoration build up in thicker layers (≤4 mm) than conventional composites (≤2 mm). The work objective was to compare various physico-mechanical properties in depthbetween nine bulk-fill and two reference conventional composites, one flowable and one paste.

Materials and methods: TetricEvoCeramBulk-Fill (Ivoclar-Vivadent), VenusBulk-Fill (Heraeus-Kuzer), SDR (Dentsply), X-traFil (VOCO), X-traBase (VOCO), SonicFill (Kerr), FiltekBulk-Fill (3M-Espe), Ever-Xposterior (GC Europe), Fill-up! (Coltene-Whaledent) were compared to two conventional materials: GrandioFlow and Grandio (VOCO). First, composites were placed in a rectangular teflon mold of 5 mm × 5 mm × 10 mm and light-cured from the top for 20 s with Bluephase G2 (Ivoclar-Vivadent, irradiance = 1050 mW/cm^2). Degree of conversion (DC) and microhardness (VHNdry) were measured (after 24 h dark storage at room temperature) at 0, 2, 4 and 6 mm along the sample side using Raman Spectrometry and Vickers micro-indentation, respectively. VHN measurement was repeated after 24 h ethanol storage (VHNEtOH), to evaluate crosslinking density. Second, composites were placed into 3 superimposed rectangular teflon molds (2 mm × 2 mm × 25 mm), separated from each other by polyester films and assembled perpendicularly to the long axis by 2 screws to align them perfectly. They were then light-cured from the top of the upper mold by three overlapping irradiations of 20 s. The samples were stored in distilled water in the dark at 37 °C during a week. Each bar was then submitted to a three-point bend test to determine flexural strength (FS) and elastic modulus (E). Data were analyzed by one-way ANOVA and either Tukey's or Wilcoxon test ($p = 0.05$).

Results: For each material, no significant difference in DC was observed within the first 4 mm, except for SonicFil, Grandio and GrandioFlow ($p < 0.05$ between 2 and 4 mm). For VHNdry, no significant decreased was observed within the first 4 mm, except for TetricEvoCeramBulk-Fill, Grandio and GrandioFlow ($p < 0.05$ between 2 and 4 mm). After ethanol storage, significant differences were observed below 4 mm for two thirds of the materials, and below 2 mm for SDR and FiltekBulk-Fill. No significant difference could be observed between the first 2 layers for E, except for FiltekBulk-Fill, SDR and GrandioFlow. Flexural strength was similar between the first two layers except for SDR ($p < 0.05$).

Conclusions: Globally, despite some exceptions, a similar evolution of the quality of cure in depth was observed when considering DC, VHNdry, E and FS. This tends to confirm the capacity of most bulk-fill to be cured in thick layers (≤4 mm). However, significant differences appeared at lower depths after ethanol storage, probably highlighting differences in crosslinking densities, which is subject to caution.

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Acid concentration and etching time efficacy on lithia-based glass ceramics

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Purpose/aim: To evaluate the influence of hydrofluoric acid (HF) concentration and etching time on the microshear bond strength (μSBS) of RelyX Unicem 2 to VITA Suprinity and IPS e.max CAD.

Materials and methods: To replicate the intaglio surface, 40 bars were fabricated with CEREC InLab MC-XL for both VITA Suprinity and IPS e.max CAD. A specific device was developed for specimen's fabrication. Sixteen groups ($n = 15$) were defined based on the tested etching times of 20, 40, 60 and 120 s, and HF concentration of 4.9% and 9.5% for both materials. A silane coupling agent was used prior to building up resin cement cylinders for microshear bond strength test. After storage for 24 h in 37 °C distilled water storage, specimens were subjected to the μSBS test. Shear bond strength was measured in MPa. Mode of failure was evaluated under an optical stereomicroscope and classified as adhesive, mixed, cohesive in resin cement or cohesive in ceramic. The etched surfaces were observed using scanning electron microscopy. The Three-Way Analysis of Variance was applied with bond strength data as the dependent variable, material, etchant concentration, and etching time as factors. The Tukey test was used for post hoc comparisons as needed. In all analyses the level of significance was set at $p < 0.05$.

Results: Table 1 reports descriptive statistics of shear bond strength data and statistically significant results. Regardless of the other factors, Suprinity yielded significantly higher bond strengths than e.max ($p < 0.001$). Etchant concentration was found to be an influential factor per se, with superior strengths being measured following etching with 4.9% HF ($p = 0.009$). Etching time did not significantly affect adhesion ($p = 0.066$).

Table 1

Material**	Etchant concentration	Etching time	Mean	SD	N
Suprinity Mean 18.33 SD 6.43 N 120	4.9%Aa	20	21.07	4.34	15
		40	20.84	3.75	15
		60	19.68	6.31	15
		120	20.09	6.50	15
	9.5%B	20	16.45	7.71	15
		40	13.77	3.41	15
		60	20.60	6.41	15
		120	14.17	7.38	15
e.max Mean 15.47 SD 5.89 N 120	4.9%b	20	14.29	7.13	15
		40	16.83	7.73	15
		60	15.84	5.52	15
		120	14.52	4.58	15
	9.5%	20	11.54	5.29	15
		40	15.25	5.25	15
		60	18.19	4.63	15
		120	17.34	4.82	15
Etchant concentration*					
4.9%	Mean 17.90 (6.30) N 120				
9.5%	Mean 15.91 (6.21)N 120				

** $p < 0.001$.
* $p < 0.05$.
Capital letters label the statistically significant interaction within material, small letters label the statistically significant interaction within etchant concentration.

The material-etching interaction was found to be significant ($p = 0.004$).

Conclusions: The 4.9% HF concentration more effectively enhanced adhesion of RelyX Unicem 2 to either lithia-based glass ceramic materials. Conversely etching application time was not an influencial factor. VITA Suprinity showed higher bond strength values than e.max CAD.

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Can bioactive proteins enhance the outcome in pulp conservative therapies?

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Purpose/aim: Systematically review the literature about the efficacy of bioactive dentin proteins in pulp conservative therapies using animal experiments.

Materials and methods: This study is reported in accordance with the PRISMA Statement. Two reviewers independently conducted a literature search in eight databases: PubMed (MedLine), Lilacs, IBECS, BBO, Web of Science, Scopus, SciELO and The Cochrane Library. It was included animal experiments in which bioactive dentin proteins were applied directly or indirectly to the pulp tissue. Data regarding the characteristics of proteins evaluated, the delivery systems used and the main findings from each study were tabulated in order to assess the outcomes of interest (tertiary dentin formation, inflammatory response, intratubular mineralization).

Results: A total of 3019 studies were initially identified. After screening, 32 papers fulfilled selection criteria and were included in the qualitative analysis. Direct pulp capping was the most evaluated therapy with bioactive dentin proteins. Besides, the most studied proteins were BMP-7 (Bone morphogenetic protein-7), TGF-β1 (transforming growth factor-β1) and soluble dentin matrix proteins (EDTA-soluble). In general, bioactive proteins enhanced tertiary dentin formation in direct and indirect pulp capping, and promoted a initial lower inflammatory response. However, for pulpotomy the bioactive materials did not demonstrated differences from control in the outcomes evaluated.

Conclusions: There are potential areas to be explored for novel therapeutic approaches to dental tissue repair and regeneration with new bioactive materials. There is evidence in the literature that suggests bioactive dentin molecules could be able to improve tertiary dentin formation with less initial inflammatory response in direct and indirect pulp therapy.

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Mechanical and morphological alterations produced by radiation on bone-implant interaction

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Purpose/aim: This study aimed to evaluate the radiotherapy effect on implant retention and additionally on the biomechanical, histomorphometric and microstructural parameters of bone close and distant of the dental implant.

Materials and methods: Twenty adults male New Zealand rabbits received three implant of 3.5 mm in diameter (Titamax CM, Neodent) and were divided into 2 groups: NoIr, control group-no radiation; Ir, the animals were irradiated 2 weeks after implant installation with 30 Gy in single dose. After 4 weeks of the implant installation the animals were sacrificed and the implant/bone blocks were used for each experimental test ($n = 10$). Pull-out test using axial loading at 1.0 mm/s crosshead speed was used for measuring the implant retention. Dynamic indentation test were used to quantify Vickers Hardness (VHN) and elasticity modulus (E) of bone tissue close and distant of implant surface. MicroCT was used to analyze the bone cortical volume (CtV), cortical thickness (CtTh) and porosity (Ct.Po) close (Ir-CIm) and distant (Ir-dIm) of implant surface. Data were analyzed by using *t*-Student test and one-way ANOVA followed by Tukey test ($P < .05$).

Results: The implant shear retention values for Ir group was significant lower than NIr group. The bone of Ir-cIm group had significantly lower E, VHN, CtTh and CtV values and higher CtPo for than for Ir-dIm. The Ir-dm group had significantly lower E, VHN, CtTh values and higher CtPo than NIr group.

Conclusions: Ionizing radiation over osseointegrated implant had negative effect on mechanical; on bone implant shear retention and micro-architecture parameters of bone mainly close to implant surface.

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Effect of pulp-capping and filling-technique on pulp-chamber heating and deformation

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Purpose/aim: To analyze the degree of cure, post-gel shrinkage of pulp capping materials and the effect of pulp capping and incremental filling restoration on the interface denin/material integrity at the pulpal floor, and also on the dentin strain, the temperature rise in the pulp chamber in large class II cavities.

Materials and methods: Eighty Extracted molars received large MOD cavities with 1.0 mm of dentin remaining at the pulp floor. Three pulp protection materials: self-etching adhesive system, Cle, Clearfil SE Bond (Kuraray), and two light-curing calcium hydroxides: Bio, Biocal (Biodinâmica); Ult, Ultrablend plus (Ultradent); Vit, Vitrebond (3M-ESPE) were applied on the pulpal floor. The cavities were restored with resin composite (Filtek Z350 XT, 3MEspe) ($n = 10$). Thermocouple and strain gauge were placed inside the pulp chamber in contact with the pulpal floor to detect temperature changes and polymerization shrinkage during the light curing of the pulp protection materials (Pp) and resin restoration (Rr). The interface integrity at the pulpal floor was investigated with microCT (Sky Scan 1272). The degree of cure was measured using FTIR. The post-gel shrinkage (Shr) was measured using strain gauge test. Shrinkage stresses were analyzed using non-linear finite element analysis. Data were analyzed using one-way ANOVA and Tukey test ($P < .05$).

Results: The temperature inside the pulp chamber increased 3.5 °C after light-curing the pulp protection materials and 2.1 °C after final restoration. The temperature change was significantly higher at light activation of pulp capping than at incremental filling technique. Material type had no influence on temperature increase. During light activation of capping material, CLE resulted in higher pulp-dentin strain than other materials. Cle and Bio showed higher Shr than other materials. Bio showed lower DC than other materials. The MicroCT showed perfect interface integrity after restoration for CLE, Vit and ULT. Gaps were found between BIO and pulpal floor in all specimens.

Conclusions: Light curing of pulp protection materials caused pulp-dentin deformation and increased pulpal temperature. Shrinkage of the composite restoration caused debonding of Biocal from the pulpal floor. Vitrebond caused lower and Biocal higher shrinkage stress than other materials.

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Sources of proanthocyanidins and long-term biological effects on dentin matrix

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Purpose/aim: Mechanical and chemical biomodification of dentin by plant derived proanthocyanidins (PACs) was shown to be dependent of the source and content/type of PACs. This study evaluated over a period of 18 months the effects of various crude sources of PACs on the mechanical properties and biodegradation of dentin matrix.

Materials and methods: The chemical profiling of selected PAC-rich crude extracts from four plants extracts. The barks of Pinus massoniana (PM), Cinnamomum verum (CV), Hamamelis virginiana (HV) and seeds of Vitis vinifera (VV) were obtained by HPLC analysis using Diol column after solvent partitioning. Dentin matrices ($n = 15$) were prepared from 52 extracted human molars to determine the apparent of modulus of elasticity over 18-month storage in simulate body fluid. Biodegradation of collagen by endogenous MMPs was determined by standard hydroxyproline assay of the storage media. Additional dentin matrix specimens were prepared to assess susceptibility to degradation by exogenous proteases (type VI bacterial collagenase) at baseline ($n = 10$) and 18-month storage ($n = 15$). Data analyses were performed using ANOVA and Games–Howell post hoc tests ($\alpha = 0.05$).

Results: The UHPLC analysis showed abundance of A-type trimers PACs on CV; abundance of dimeric procyanidin A1 and A-type interflavanyl linkages in PM. The HV extract contains both A and B type PACs, notably as their gallate esters as well as hydrolysable tannins; while VV mainly contains B-type PACs and galloylated PAC derivatives. After 18 months of evaluation, crude extracts of PM (42.1 ± 2.4 MPa) followed by CV, (34.4 ± 1.5 MPa) remained the greatest in modulus of elasticity ($p < 0.05$). While samples treated with CV exhibited the lowest degradation by bacterial collagenase at baseline and 18 months ($p < 0.05$); specimens treated with PM showed the least mass loss and the least collagen solubilization ($p < 0.05$) by endogenous proteases after 18 months aging.

Conclusions: A remarkable resistance against enzymatic degradation was observed for all groups treated with extracts. The most potent biomodification capacity including mechanical strength was observed with PM and CV extracts. The chemical profiles of the extracts provide clues to targeting PACs for the development of intervention materials.

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Influence of benzalkonium chloride on dentin μTbs and MMPs activity

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Purpose/aim: Benzalkonium chloride (BAC) is a nitrogenous cationic surface-acting agent containing a quaternary ammonium group claimed to inhibit dentin matrix metalloproteinases (MMPs) in addition to its disinfectant ability. The aim of the study was to investigate the adhesive bond strength and the dentin enzymatic activity of a multi-mode universal adhesive system (All-Bond Universal; Bisco Inc., ABU) and an experimental adhesive with BAC blended within its formulation (SBAC, Bisco Inc.,) employed in etch-and rinse (E&R) or self-etch (SE) mode.

Materials and methods: A standardized smear layer was created using a 180-grit silicon-carbide paper on 32 middle/deep human dentin surfaces. Specimens were assigned to the following groups ($n=8$) according to the adhesive procedure: G1: SBAC employed in E&R mode after dentin etching with 35% phosphoric acid for 15 s. G2: SBAC employed in SE mode on untreated dentin. G3: ABU employed in E&R mode after dentin etching with 35% phosphoric acid for 15 s. G4: application of ABU in SE mode on untreated dentin. All adhesives were applied according to manufacturer's instructions and cured for 20 s. Composite buildups were created on the bonded surfaces, then specimens were cut for microtensile bond strength test (μTBS) and stressed to failure at a crosshead speed of 1 mm/min after 24-h storage in artificial saliva at 37 °C. μTBS data were analyzed using two-way ANOVA and Tukey's multiple comparison tests. Additionally, enzymatic activity was evaluated using a zymographic assay on protein extracts obtained from adhesive-treated dentin powder of each tested group.

Results: Results of μTBS showed no significant differences after 24 h storage in artificial saliva within the four groups. Furthermore, zymographic analysis revealed increased expression of dentin endogenous MMP-2 and -9 after application of All Bond Universal in SE mode, while in the E&R mode the expression of the MMP-2 decreased and MMP-9 was inhibited. SBAC employed in SE mode increased the expression of MMP-2, while inactivating the MMP-9. The application of SBAC in the E&R mode also inactivated MMP-9, while MMP-2 activity was significantly decreased.

Conclusions: Further studies and longer aging are needed to clarify the influence of BAC blended within the adhesive formulation in improving bond longevity and dentin MMPs inhibition.

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5-Year randomized clinical evaluation of posterior bulk-fill restorations

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Purpose/aim: To evaluate in a randomized controlled study the 5-year clinical durability of a flowable resin composite bulk-fill technique in Class I and Class II restorations.

Materials and methods: 38 pairs Class I and 62 pairs Class II restorations were placed in 44 male and 42 female (mean age 52.4 years). Each patient received at least two, as similar as possible, extended Class I or Class II restorations. In all cavities, a 1-step self-etch adhesive (Xeno V+) was applied. Randomized, one of the cavities of each pair received the flowable bulk-filled resin composite (SDR), in increments up to 4 mm as needed to fill the cavity 2 mm short of the occlusal cavosurface. The occlusal part was completed with the nano-hybrid resin composite (Ceram X mono+). In the other cavity, the resin composite-only (Ceram X mono+) was placed in 2 mm increments. The restorations were evaluated using slightly modified USPHS criteria at baseline and then yearly during 5 years. Caries risk and bruxing habits of the participants were estimated.

Results: No post-operative sensitivity was reported. At 5-year 183, 68 Class I and 115 Class II, restorations were evaluated. Ten restorations failed (5.5%), all Class II, 4 SDR-CeramX mono+ and 6 CeramX mono+-only restorations. The main reasons for failure were tooth fracture (6) and secondary caries (4). The annual failure rate (AFR) for all restorations (Class I and II) was for the bulk-filled-1.1% and for the resin composite-only restorations 1.3%. For the Class II restorations, the AFR was 1.4% and 2.1%, respectively.

Conclusions: The stress decreasing flowable bulk-fill resin composite technique showed good durability during the 5-year follow-up.

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Non-destructive assessment of biofilm metabolism on resin composite

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Purpose/aim: Several assays are available for the quantification of oral biofilm growth, but each has critical limitations and are typically not performed on relevant substrates. Resin composites and adhesive resins release unpolymerized monomers and by-products that influence biofilm growth and confound measurement of metabolic activity. New tools are therefore needed to quantify oral biofilm growth on resin-based dental materials. The objective of this study was to determine the utility of a firefly luciferase assay for

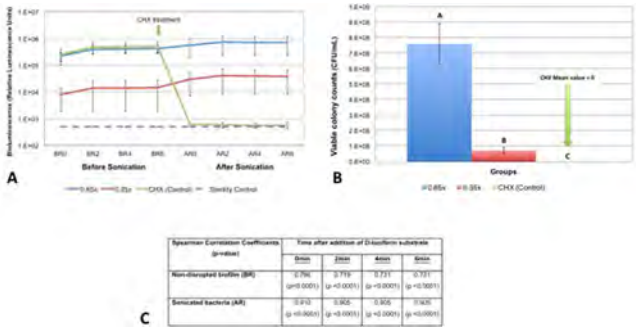

Fig. 1

quantification of *Streptococcus mutans* biofilm viability on a resin composite substrate, and correlate the results with a standard method (viable colony counts).

Materials and methods: Disk-shaped specimens ($d = 6.0$ mm, $h = 1.1$ mm; $n = 15$/group) of Point Four resin composite (Kerr Dental Products) were fabricated, wet-polished (final polish: 0.5 μm-diamond), UV-sterilized, and stored in water (72 h, 37 °C). *Streptococcus mutans* biofilms (strain UA159 modified by insertion of constitutively expressed firefly luc gene) were statically grown (1:500 dilution; anaerobic conditions, 24 h, 37 °C) in two media concentrations (0.35× and 0.65× THY medium supplemented with 0.1% sucrose). An additional group of specimens with biofilms grown in 0.65× growth media was treated with chlorhexidine gluconate (CHX) for 2 min to serve as the control group. Biofilm luminescence measurements (Relative Luminescence Units/RLU) of intact biofilms were obtained after addition of d-luciferin substrate. The adherent biofilms were removed by sonication, and bioluminescence of sonicated bacteria was then measured. Colony counts (CFU/mL) on THY agar plates supplemented with 800 μg of Spectinomycin were determined after sonication. Bioluminescence values and colony counts were correlated using Spearman Correlation tests ($\alpha = 0.05$).

Results: The results are shown in Fig. 1A–C.

Conclusions: Strong positive correlations between viable colony counts and bioluminescence values, both before- and after-sonication, validate the use of this novel non-disruptive, real-time bioluminescence assay to quantify intact *S. mutans* biofilms grown on a resin composite, and potentially on antibacterial materials and other types of dental materials.

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Surface modification of yttria-stabilized tetragonal zirconia with different glass based liners

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Purpose/aim: Yttrium oxide partially stabilized tetragonal zirconia (Y-TZP) is one of the most versatile materials used in dentistry because of its biocompatibility and distinctive fracture toughness. The modification of the surface of Y-TZP to create a roughness.

It's necessary to enhance the bonding to ceramics. Several surface treatments have been proposed, some of them promoting the phase transformation of the Y-TZP. Accordingly, the aim in this study was to modify the surface of Y-TZP by applying three different glass-based liners to increase roughness.

Materials and methods: Three glass-based liners (GBL-1, GBL-2, GBL-3; based on $Ca(NO_3)$, $NaNO_3$, and KNO_3were formulated as a sol gel solution and applied on the surface of zirconia discs. The discs then were let to dry for 2 h, and sintered to 900 °C for 2 h. The discs with the different liners were characterized with an atomic force microscope (NTegra NT-MDT Prima) with tapping mode. Total roughness (Ra) was measured on the samples treated with each glass-based liner and the zirconia without treatment in to compare them.

Results: Ra (nm) values of the Y-TZP without treatment showed a total roughness of 86.82 nm, the Y-TZP with the GBL-1 showed 189.56 nm, Y-TZP with GBL-2 showed 189.96 and the last group Y-TZP with GBL-3 showed 87.47 nm. Significant statistical differences were noticed between the control and GBL-1 and GBL-2 ($p < 0.05$).

Conclusions: The surface modification of Y-TZP by applying the three different glass based liners enhanced the total roughness of the surface. The different formulation affected on the roughness (Fig. 1).

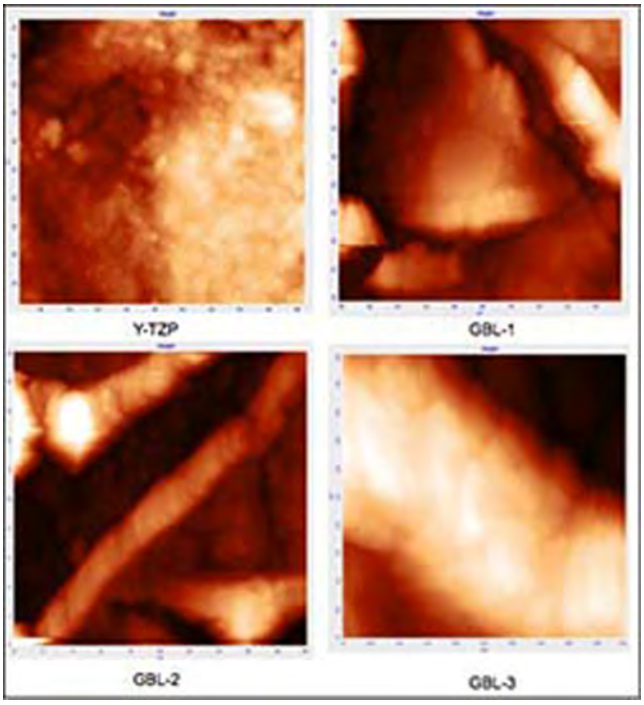

Fig. 1

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Mineral trioxide aggregate attenuates proangiogenic activity of endothelial cells

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Purpose/aim: Angiogenesis is defined as the formation of new blood vessels from preexisting capillaries, which has great importance in pulp regeneration and homeostasis. Dental pulp regeneration is a part of regenerative endodontics, which includes isolation, propagation, and transplantation of stem cells inside the prepared root canal space. The formation of new blood vessels through angiogenesis is mandatory to increase the survival rate of transplanted tissues. Current study investigated the influence of mineral trioxide aggregate (MTA) on proangiogenic properties of primary tooth pulp endothelial cells.

Materials and methods: Primary pulp endothelial cells were prepared from mice molars. The impact of MTA on proangiogenic properties of these cells was determined by examining rate proliferation, migration, and capillary morphogenesis. Cells incubated without the cement were used as control. The impact of MTA on angiogenesis was also assessed in an in vivo model of neovascularization. The *t*-test was used to evaluate the significance of the differences between the mean values.

Results: Mineral trioxide aggregate elicited a significant decrease in proliferation of tooth pulp endothelial cells compared with the control. Incubation with the MTA also attenuated the migration and capillary morphogenesis of these cells. These results were consistent with attenuation of neovascularization *in vivo*.

Conclusions: Tissue regeneration requires precise spatial and temporal coordination of proliferation and differentiation of endothelial cells, and tissue vascularization. Mineral trioxide aggregate inhibited the proliferation and capillary morphogenesis of tooth pulp endothelial cells in culture and neovascularization in vivo. These findings may help to predict the suitability of various dental materials on surrounding tissue regeneration.

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Development of PLGA/layered double hydroxide microsphere-sintered scaffolds for bone regeneration

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Purpose/aim: The purpose of this study was to fabricate and evaluate the novel three dimensional scaffolds made of poly(lactic-co-glycolic acid) (PLGA)/layered double hydroxide (LDH) composite microspheres for potential bone substitute applications.

Materials and methods: We employed microsphere sintering technique to produce the three dimensional PLGA microspheres/LDH composite scaffolds. Then, the structural, mechanical and biological characteristics of these scaffolds were evaluated.

Results: The mechanical properties of scaffolds were regulated by altering various parameters, such as sintering temperature, sintering time, and PLGA/LDH ratio. The obtained results demonstrated that the PLGA/LDH (4:1) scaffold sintered at 90 °C for 2 h had the highest mechanical properties and also had an appropriate pore structure for bone regeneration. The MTT assay results displayed that the cell proliferation on the composite scaffolds (PLGA/LDH (4:1) scaffold sintered at 90 °C for 2 h) was higher than pure PLGA scaffold and control group. Also, osteoblasts cultured on the composite scaffolds, had higher alkaline phosphatase activity (ALP activity) than those cultured on the pure PLGA scaffold and control group.

Conclusions: The novel PLGA/LDH composite may have future implications as a scaffold for bone regeneration such as the treatment of alveolar bone loss.

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Longitudinal clinical study: 3 in-office bleaching protocols, one year follow-up

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Purpose/aim: Longitudinal clinical studies, which follow patients over a long period of time, are not very common in the dental literature, especially when associated with large samples. This study evaluated the efficacy and color stability of three in-office bleaching systems over a period of time of 365 d.

Materials and methods: A randomized clinical trial was conducted with 90 volunteers divided into 3 Groups (Table 1) ($n = 30$); subjects for Groups 1 and 3 underwent a bleaching treatment session according to manufacturer's instructions while an authors' variation of the technique was used for Group 2. Color changes were measured using a spectrophotometer (Vita-Easyshade) and the shade guides Vita-Classical and Vita-Bleachedguide 3D-Master. Color assessments were made on superior central incisives and canines before, immediately after and at 7, 14, 30, 180 and 365 d post treatment. The data obtained was submitted to statistical analyses of variance (ANOVA) with the use of the software Statistica 8.0 (StatSoft-Dell) and comparisons were made using contrasts (Tukey test).

Results: The highest whitening result was obtained by Group 1, at immediate post treatment and for all evaluations thereafter. Results for Groups 2 and 3 were statistically similar among themselves. All treatments promoted tooth whitening and maintained the bleaching effect for 365 days; none of the participants in this study had fadeback to the same shade color levels that were registered before the bleaching treatments. Vita-Easyshade spectrophotometer detected statistically significant differences at the immediately post treatment assessment, while for the other two measurement methods it was only at the 30 d evaluation that statistically significant differences were first observed.

Table 1 – Groups clinical study.

Group	Whitening gel	Gel concentration	Light source
1	Zoom Whitespeed Bleaching Gel (Phillips Oral Healthcare)	25% HP	Whitespeed LED light (Phillips Oral Healthcare)
2	Zoom Whitespeed Bleaching Gel (Phillips Oral Healthcare)	25% HP	No light
3	Boost Opalescence Xtra Boost (Ultradent)	38% HP	No light

Conclusions: Group 1 presented the highest bleaching results and color stability was registered for all the experimental groups at the 365-day period. In addition it was observed that the visual analyzes have less capacity to detect bleaching shade differences than instrumental analysis.

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Effect of radiation and antioxidant on dentin-composite bonding

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Purpose/aim: The aim of this study was to evaluate the effect of radiotherapy and antioxidant N-acetyl cysteine (NAC) on the microtensile bond strength (μTBS) of the dentin-composite interface.

Materials and methods: Thirty human thirds molars were sectioned to expose middle dentin surface and distributed into 6 groups ($n = 5$), according to application or not of NAC for 5 s, before the adhesive Clearfil SE Bond and submission or not of 60 Gy total radiation (2 Gy daily doses, 5 days/week, for 6 weeks): G1: Control group, without radiotherapy and without application of NAC; G2: Control group, without radiotherapy and with application of NAC; G3: RestRtWt, was submitted to radiotherapy after restoration procedure without application of NAC; G4: RestRtNAC, was submitted to radiotherapy after restoration procedure with application of NAC; G5: RtRestWt was submitted to radiotherapy before restoration procedure without application of NAC; and, G6: RtRestNAC, was submitted to radiotherapy before restoration procedure with application of NAC. A block of 6 mm thickness resin composite Z250 (3M ESPE) was constructed on dentin and each 2 mm thickness was light cured for 40 s. After stored in deionized water at 37 °C for 24 h, the specimens were sectioned perpendicularly to bonding area to obtain sticks with a cross-sectional area of 1 mm^2, and submitted to the μTBS at a crosshead speed of 0.5 mm/min. Data were submitted to two ANOVA and Tukey's test ($\alpha = 0.05$).

Results: Bond strength values (MPa – mean ± standard deviation) for the specimens with or without application of NAC were: Control group (42.5 ± 6.3 and 49.2 ± 9.4) and Groups RestRt (49.9 ± 6.2 and 53.0 ± 1.7) were significantly higher than groups RtRest (37.7 ± 8.4 and 45.9 ± 7.3) ($p < 0.05$). The antioxidant NAC significantly reduced μTBS compared to groups without NAC application ($p < 0.05$).

Conclusions: The radiotherapy before composite restoration procedure decreased the μTBS. The use of antioxidant NAC reduced μTBS for all conditions.

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In vitro aging and mechanical properties of translucent monolithic zirconia

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Purpose/aim: The dental market demands for high-translucency monolithic zirconia dental crowns, which are usually placed either with or without a thin layer of glaze. Over the last years, an improved translucency was achieved with slight modifications in tetragonal zirconia microstructure. Moreover, novel "ultra-translucent" and multi-layered zirconia materials appeared recently on the market, showing superior esthetical properties and graded chroma. The microstructural features and the mechanical performance of these materials are still controversial, as well as their potential susceptibility to hydrothermal degradation. The present in vitro study aimed at evaluating: – the microstructure and mechanical properties of high-translucency zirconia crowns; – the aging behavior of such materials in the glazed/non-glazed state.

Materials and methods: Six monolithic zirconia dental crowns (GC Initial Zirconia) with different translucency (2 standard "ST", 2 high "HT" and 2 ultra-high "UHT") were produced by CAD-CAM technology. The outer surface was splitted into 2 halves, each comprising 1 supporting and 1 chewing cusp; one half was glazed (~50 μm), while the other half was left uncoated. The layer of glaze was compensated during the CAD process to ensure even occlusal contacts. The microstructure was analyzed by scanning electron microscopy (SEM). X-ray diffraction (XRD) analyses were performed at level of the outer surface (both glazed and non-glazed) at time 0 and after 2, 6, 18, 54 h of artificial aging in autoclave (134 °C at 2 bars). The mechanical properties were investigated by piston-on-three-balls biaxial strength, indentation fracture toughness and hardness tests performed on polished 12 mm diameter discs.

Results: XRD patterns showed clear differences between ST, HT and UHT materials. The UHT crowns exhibited bigger grain size and XRD patterns corresponding mostly to the cubic phase. No aging was observed on cubic crowns, while the tetragonal ones showed typical aging behavior of 3Y-TZP materials, where the presence of glaze had a protective effect. The mechanical properties of cubic crowns were definitely lower than for tetragonal ones.

Conclusions: In tetragonal zirconia monolithic crowns, hydrothermal degradation can be observed also for short aging times, while the glaze acted as a protective layer. The presence of the cubic phase in UHT crowns entails two main advantages: a sensible increase in translucency and the complete absence of hydrothermal degradation. On the other hand, the lack of transformation toughening for cubic zirconia and the coarser microstructure cause a severe drop in mechanical properties, which can represent a limitation for their application in conditions where high mechanical stresses are applied.

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Paffenbarger Award finalists

P1

The complexity of proanthocyanidins on the resin-dentin bond strength

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Purpose/aim: Oligomeric proanthocyanidins (OPACs) can reinforce the anchoring dentin matrix by mediating collagen cross-links. We hypothesize that the OPACs inherent bioadhesive properties can be affected by the degree of oligomerization and the presence of galloyl groups.

Materials and methods: Two fractions, one containing galloylated dimers (G) and the other non-galloylated dimers (NG) we produced from an enriched grape seed extract (e-GSE) containing OPACs with degree of polymerization predominantly between 2 and 5. The adhesive properties were assessed on dentin surfaces using standard microtensile bond strength test (TBS) on flat mid-coronal dentin of sound human molars and an experimental adhesive without HEMA. The priming solutions (e-GSE, G and NG) were prepared at 15% w/v (pH 7.2). Dentin surfaces were demineralized (35% phosphoric acid), rinsed, blotted-dry and the respective priming solutions were applied for 1 min. HEPES buffer was used control. After 24 h teeth were cut to obtain resin-dentin beams of 0.8 mm × 0.8 mm, TBS was assessed immediately, 6 and 12 months after storage in simulated body fluid (SBF).

Results: TBS of the group treated with G was significantly lower than the group treated with e-GSE ($p < 0.001$). No statistically significant differences were observed in TBS between groups G and NG ($p > 0.05$); and NG and e-GSE ($p > 0.05$). Aging in SBF for one year did not affect the microtensile bond strength ($p > 0.05$). No specimens were retrieved from control group (TBS = 0).

Conclusions: Adhesion of an essentially hydrophobic resin to a wet substrate is possible through modifications induced by the application of a OPACs primer to the dentin matrix. e-GSE priming led to the strongest adhesion followed by the fraction containing non-galloylated dimers.

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P2

Compounds incorporation that modify *Streptococcus mutans* virulence in restorative materials

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Purpose/aim: The objectives of this in vitro study were to evaluate the effect of the addition of two natural antimicrobial compounds derived from Brazilian propolis (Apigenin (A) and tt-Farnesol (T)) on the chemical/physical properties of resin composites (CO) and resin cements (CE), and on the virulence of *Streptococcus mutans* (SM) and secondary caries formation.

Materials and methods: A and F were added separately, in combination and combined with fluoride (F) to a resin composite and a resin cement. Dry weight (DW), bacterial viability (BV), protein (P), water-soluble (WSP), alkali soluble (ASP) and intracellular (IPS) polysaccharides were determined from SM biofilms grown for five days on resin composites and resin cements disks. The A and T effects on the physical/chemical properties were analyzed by flexural strength (FS), flexural modulus (FM), curing kinetics and overall degree of conversion (DC). SEM images were made from the biofilm and confocal images from the secondary caries development (in presence of gap for 14 days of growing biofilm).

Results: The DW and IPS decreased when A, AT and ATF was added for both materials, compared to the control group. A and AT addition decreased the P for CO and A and T into CE. BV decreased with addition of AT and ATF and ASP with addition of A and ATF into CE. No statistically difference was observed for BV, WSP and ASP for CO and WSP for CE. No difference was observed for curing kinetics, FS and FM for both materials. A lower DC was obtained when A and AF was added to CO, and a higher DC when A, T and ATF was added to CE. For both materials a less dense biofilm is observed under SEM images when A, AT and ATF was added. From confocal images no difference is observed when dentin is exposed, however, when remaining enamel is found the demineralization seen to be lower for all additions tested compared to the control group.

Conclusions: A, alone and combined showed better results reducing the expression of virulence of SM. No addition was able to completely avoid secondary caries formation or bacterial penetration, however, the additions into restorative materials seen to be promising when remaining enamel is present.

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P3

Y-TZP low temperature degradation: A sigmoidal or a linear behavior?

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Purpose/aim: The aim of this study was to evaluate the in vitro aging behavior of a dental Y-TZP submitted to an accelerated hydrothermal aging. Specifically: (a) determine the kinetic curve of tetragonal to monoclinic phase transformation (t-m); (b) calculate the speed of the front of phase transformation zone into the bulk during aging; (c) compare the relationship between monoclinic percentage and depth of phase transformation with biaxial flexural strength.

Materials and methods: Fully sintered (1530 °C/2 h) discs of dental Y-TZP (VITA YZ) were aged in ionized water using a hydrothermal pressurized reactor to follow the kinetics of phase transformation. Four samples per aging time were submitted to X-ray diffraction analysis (XRD), Cu-Kα, 20° to 80°, 2θ. The data was refined using the Rietveld method (GSAS). Discs were divided according to the aging time ($n = 10$): 0, 5, 25, 70 and 140 h (150 °C/3.01 bar). One specimen of each group was sectioned at the transversal section, polished and submitted to backscatter SEM to calculate the phase transformation depth (Image J). The speed of the front transformation zone was determined plotting the phase transformation depth versus aging time. Each group was submitted to biaxial flexural strength test (ISO 6872) and one way Anova ($\alpha = 0.05$) was used to compare the results.

Results: XRD results indicated that Y-TZP presented a sigmoidal phase transformation behavior, and a monoclinic phase plateau (65%) was reached at 15 h (150 °C). However, the depth of XRD penetration was limited to ~6.1 μm. SEM analysis showed that the degradation process did not follow the sigmoidal behavior indicated by XRD. Although the percentage of monoclinic phase was constant at the maximum penetration depth (~6.1 μm), SEM analysis showed a transformation front constituted of a porous microstructure (due to the polishing process) that increased linearly ($R = 0.9997$) from the surface to the bulk with a speed of 1.24.10–10 m/s at 150 °C and 3.01 bar. One way ANOVA showed that hydrothermal aging for 70 h (958 MPa, 65% of monoclinic phase, 30 μm depth) did not significantly decrease the biaxial flexural strength compared to the control group (1032 MPa). However, 140 h (849 MPa, 65% of monoclinic phase, 62 μm depth) is deleterious to the flexural resistance of the Y-TZP.

Conclusions: The results indicate that Y-TZP submitted to hydrothermal aging presents a linear front of phase transformation zone according to the aging time. The front depth of 62 μm decreased the biaxial flexural strength after 140 h of aging (150 °C).

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P4

Quality of cure on depth of bulk-fill composites: Cytotoxicity

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Purpose/aim: Bulk-fill composites as replacement of dentin in a single 4 mm-increment became rapidly popular with clinicians despite the limited data and lack of clinical studies. One point of concern is the potential toxicity of these materials due to the leaching of their components, especially in deeper layers. Hence, the work objective was to compare the cytotoxicity in depth between bulk fill composites and two reference conventional composites, one flowable and one paste.

Materials and methods: TetricEvoCeramBulk-Fill (Ivoclar-Vivadent), VenusBulk-Fill (Heraeus-Kuzer), SDR (Dentsply), X-traFil (VOCO), X-traBase (VOCO), SonicFill (Kerr), FiltekBulk-Fill (3M-Espe), Ever-Xposterior (GC europe), Fill-up! (Coltene-Whaledent) were compared to two conventional materials: GrandioFlow (VOCO) and Grandio (VOCO). Composites were placed into 3 superimposed Teflon molds (2 mm high, 5 mm diameter), separated from each other by polyester films. The molds were stacked to align the three layers of 2 mm of thickness and the composite was light-cured from the top for 20 s with Bluephase G2 (Ivoclar-Vivadent, irradiance = 1050 mW/cm^2) ($n = 4$). Each separate 2 mm-layer was then immersed into 1 ml of growth medium (Dubelcco's modified eagle's medium with 10% fetal bovine serum, 4 mmol/L L-Glu and 100 units/ml penicillin and 100 μg/ml streptomycin) in 48 well plates for 7 days at 95% humidity, 5% CO_2 and 37 °C to produce conditioned medium.

Results: Two-way ANOVA revealed significant effects for both "layer" and "composite" factors ($p < 0.0001$). Globally, there was no significant difference between upper and intermediate layers, but well with the lower layer, which is therefore associated with increased cytotoxicity. Even within the upper and intermediate layers (0–2 and 2–4 mm), some materials were significantly different from the control (cells only), showing increased cytotoxicity. Regardless of the layer, Grandio, Fill-up! and FiltekBulk-Fill resulted in the most absorbance reduction, while GrandioFlow, SonicFill, Ever-Xposterior and X-traBase in the least.

Conclusions: There are intrinsic materials differences as regards material cytotoxicity. For a given material, there is a clear increase in cytotoxicity beyond 4 mm thickness.

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P5

Modern resin-based composite filler characteristics and related physico-mechanical properties

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Purpose/aim: The mechanical properties of dental resin-based composites (RBCs) are highly dependent on filler characteristics (size, content, geometry, composition). Ideally, the classification should be predictive of one or several important material properties, as opposed to the purely descriptive current approach. Currently, most commercial materials are marketed as "nanohybrids" (i.e. filler size <1 μm) or more generally "hybrids". In the present study, filler characteristics of a selection of RBCs were described, aiming at identifying correlations with physico-mechanical properties and testing the relevance of the current classification.

Materials and methods: Micron and sub-micron particles (> or <500 nm) were isolated from 17 commercial RBCs and analyzed by laser diffractrometry and/or electron microscopy. Filler and silane content were evaluated by thermogravimetric analysis and a sedimentation technique. The flexural modulus (E flex) and flexural strength (σ flex) and micro-hardness were determined by three-point bending or with a Vickers indenter, respectively. All experiments were carried out after one week of incubation in water or 75/25 ethanol/water, to investigate solvent sensibility and matrix hydrophobicity. Further, water and ethanol/water sorption were also determined.

Results: Average size for micron-sized fillers was almost always higher than 1 μm. Both monomodal and bimodal regimes were observed for micro particles. Sub-micron fillers were observed as aggregated nanoparticles and ultra-fine glass particles. Ranges for mechanical properties were: 3.7 < E flex water < 16.3 GPa, 86 < σ flex water < 161 MPa and 23.7 < hardness water < 108.3 HV0.2/30. Values generally decreased after storage in ethanol/water (Δ max = 86%). High filler contents (>75 wt%) were associated with the highest mechanical properties (E flex and σ flex > 12 GPa and 130 MPa, respectively) and lowest solvent sorption (~0.3%). The lowest E flex was associated with lowest filler content (52.1%) and highest sorption (2.31%).

Conclusions: Mechanical properties and filler characteristics significantly vary amongst modern RBCs and the current classification does not accurately describe either of these variables. Further, significant differences in chemical stability of RBCs were observed, highlighting differences in resin and silane composition. Since E flex and sorption were well correlated to the filler content, a simple and unambiguous classification based on such characteristic can be suggested, with three levels (ultra-low fill, low-fill and compact resin composites).

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P6

Mica glass ceramics with yttria stabilized zirconia for dental restorations

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Purpose/aim: Glass ceramics have been well investigated for bio-medical and dental applications due to their bio-compatibility and process-ability. In such applications, mechanical properties also an important role in material selection. Currently, IPS Empress (Lithium aluminium silicate) are well known for their use in restorative dental applications. However, these materials have a moderate flexural strength (300–400 MPa) and fracture toughness (2.5–3 MPa $m^{1/2}$) making them susceptible to cracking and subsequently failure under loading during the course of their use. Recently, mica based glass ceramics that are highly machinable with better mechanical properties are being studied extensively. The present paper will be focusing on the development of novel mica based glass ceramics reinforced with yttria stabilized zirconia (YSZ) to improve its mechanical properties such as hardness, flexural strength and fracture toughness along with the evaluation of "in vitro" cytocompatibility.

Materials and methods: The selected glass composition contains silica (47.2 wt.%), alumina (16.7 wt.%), MgO (14.5 wt.%), K_2O (9.5 wt.%), B_2O_3 (8.5 wt.%) along with flouride additive (6.3 wt%). The glass mixture is melted at 1500 °C and quenched to obtain base glass. The base glass is then ball milled to varying amounts of YSZ (5, 10, 15, 20 wt.%) to obtain different compositions of Glass-YSZ composite. The compositions are sintered at 1080 °C for different time periods (12 h and 48 h) to achieve crystallisation of glass matrix. Phase analysis of sintered pellets of all compositions is carried out using X-ray Diffraction and microstructural characterisation using Scanning Electron Microscopy to determine major phases and morphology. The essential mechanical properties such as hardness, elastic modulus, fracture toughness and flexural strength are obtained using Vickers Hardness testing and 3-point flexure (bending) test. The in vitro cytocompatibility of the composites is evaluated using mouse fibroblast cell lines.

Results: The analysis of diffraction patterns and morphological features indicate the desired flourophlogopite phase has formed during sintering. The mechanical properties evaluated for the sintered samples were found to be much better compared to previously studied bioglasses, especially in hardness and toughness properties. The cell viability studies with mouse fibroblast cell line show no toxicity and fluorescence imaging shows good cell spreading on Glass-YSZ composites.

Conclusions: Yttria stabilized zirconia addition to base glass has been demonstrated to enhance the mechanical properties of mica based glass ceramics with potential for dental restorative applications.

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P7

Microscale spatial variability of monomer conversion in filled dental resin-based-composites

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Purpose/aim: Monomer conversion in filled dental resin-based-composites (RBCs) is routinely quantified using Fourier transform infra-red (FTIR) spectroscopy. The majority of reported FTIR measurements on RBCs have provided 'bulk' averages and micro-spectroscopic mapping approaches have been limited by insufficient resolution to discern any spatial variation in polymerisation existing at inter-particulate distances. This novel study aimed to characterise the distribution in degree of monomer to polymer conversion in filled photo-polymerised experimental RBCs and to understand how conversion varies between filler particles according to RBC composition and the photo-polymerisation variables employed.

Materials and methods: Different weight percentage (wt%) blends of Bis-GMA and TEGDMA were prepared with either 1 wt% Lucirin TPO or Camphorquinone as the photo-initiator. 8 μm (diameter) monodisperse silica microspheres were mixed with resin blends in either 50/50 or 40/60 wt% ratios to form composites. 10 mm diameter and 0.1 mm thick samples were photo-polymerised on CaF windows with a light intensity of 300 mW cm^{-2} for 60 s and then polished to a thickness of ~10 μm. FTIR micro-spectroscopy mapping was performed with a spatial resolution of 0.54 μm^2 over 500–4000 cm^{-1} using a synchrotron infra-red microscope coupled with spatial oversampling (IRENI-SRC, WI, USA). Conversion was calculated from the ratio of integrated peak intensities of aliphatic (1637 cm^{-1}) and aromatic (1608 cm^{-1}) absorption bands.

Results: 2D FTIR maps demonstrated least conversion around filler particles increasing by 15–20% ($p < 0.01$) to maximum within the inter-particulate resin matrix (Fig. 1). Most variability in degree of conversion was observed in TPO initiated RBCs. Increasing the system viscosity either due to increased Bis-GMA wt% or increased filler fraction resulted in increased variability in conversion. Principle component analyses identified photo-initiator to be the most significant factor influencing conversion variability. Azimuthally integrating conversion over the area of each filler particle produced an inverted Gaussian profile, showing reduced conversion at the 'centre' of the particle (in 2D), which increased towards the particle edge. This is indicative of less converted polymer encapsulating each filler particle. In this region, TPO initiated RBCs demonstrated a shift in the 1608 cm^{-1} aromatic

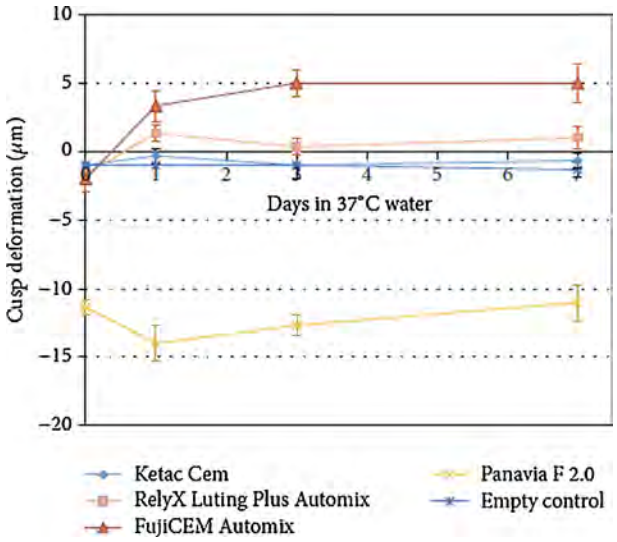

Fig. 1

IR absorption peak (found only in the Bis-GMA monomer aromatic core) to a higher vibrational frequency. This may suggest molecular bond extension storing residual strain in this layer.

Conclusions: We demonstrate that conversion can vary significantly around and between filler particles in dental RBCs and that compositional and polymerisation variables can impact on inter-particle conversion and residual strains. Microscale spatial variability of monomer conversion should now be considered in mechanical performance modelling of dental RBC materials.

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P8

Comparison of different dentifrices in organic matrix after erosion/abrasion

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Purpose/aim: Demineralized organic matrix (DOM) is an important barrier in the diffusion of acids minimizing the mineral loss of dentine submitted to erosion. Demineralization processes makes the DOM more susceptible to degradation by enzymes called matrix metalloproteinases (MMPs), which associated to a brushing may increase the tooth wear. Many agents, such as proanthocyanidin (PA) and fluoride, are considered capables to prevent degradation by MMPs. Thus, the aim of this study was to evaluate the effect of different dentifrices over dentine submitted erosion and abrasion.

Materials and methods: 60 bovine dentin blocks (4 mm × 4 mm) were randomly and equally divided into five groups: G1 – placebo dentifrice; G2 – 0.012% chlorhexidine dentifrice; G3 – NaF 1110 ppm fluoride dentifrice; G4 – 10% purified PA dentifrice; G5 – fluoride + PA dentifrice. Erosive challenge was performed by the immersion of the blocks in Cola soft drink (Coca-Cola®) for 5 min followed by immersion in artificial saliva for 120 min four times a day for 5 days (4 daily cycles). Brushing was simulated after the second and forth erosive cycles by using a tooth brushing machine with the application of slurry of each studied dentifrices and tooth brushing (200 g) during 15 s, two times a day for 30 days. The response variable was depth of dentin loss (μm) measured by profilometry. Data were analyzed by ANOVA followed by Fisher test (LSD) ($p < 0.05$).

Results: Data showed that groups G3 (1.7 ± 0.5a, b), G4 (1.8 ± 0.3a, b) and G5 (1.7 ± 0.4a) did not have significantly difference among them. G5 presented less dentine loss when compared to G1 (2.4 ± 0.5c) and G2 (2.1 ± 0.3b, c). Groups G1 and G2 did not significantly differ from each other, because both groups presented the highest dentin tissue loss.

Conclusions: PA and fluoride dentifrices could separately minimize the wear of eroded dentin submitted to tooth brushing, but the combination of these two agents presented the best results, showing that this type of dentifrice presented promising perspectives for patients who suffer with tooth erosion.

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P9

Piperonylic methacrylate: New copolymerizable coinitiator with high biocompatibility

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Purpose/Aim: The objective of this study was the synthesis of a new molecule, the piperonylic methacrylate, yet not described in literature and with potential actuation like an alternative co-initiator copolymerizable, to the composition of radical photoinitiation systems with high performance and biocompatibility.

Materials and methods: To synthesize methacrylate piperonylic, two different synthetic routes were evaluated. The obtained product was purified by fractional distillation and chromatographic column, posteriorly being passing by proton nuclear magnetic resonance and carbon to the confirmation of the structure. The adhesive was formulated with model adhesive resin, composed by BISGMA, TEGDMA and HEMA. Camforquinone (CQ) in 0.5% molar concentration was used as polymerization photoinitiator of the resin model, which was added to the different polymerization co-initiators in the composition. Experimental groups with different co-initiators,

were formulated, using: piperonylic methacrylate (MP), constituting binary photoactivation systems. Additionally, a group with tertiary amine (EDAB), was formulated, as a control. The physico-chemical and mechanical characteristics and polymer properties applied in the experimental adhesive were evaluated.

Results: The synthesis was confirmed and the methacrylate piperonylic obtained with success. The selected experimental groups: CQ + MP, CQ + EDAB were analyzed. The degree of conversion (20s photoactivation time) using Radii Cal was 60% (±1.4) to CQ + EDAB and 82% (±5.8) to CQ + MP.

Conclusions: The piperonylic methacrylate showed similar or superior performance to the tertiary amine, with the advantage its potential for copolymerization, showing an alternative reagent for photopolymerizable compositions.

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P10

Esthetic properties of multi shade vs. single shade composites

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Purpose/Aim: The aim of this study was to compare the esthetic properties of composite restorations made with a multi shade (Admira Fusion, Voco) X single shade (Admira Fusion X-tra, Voco) materials.

Materials and methods: Twenty-three intact extracted human molars were used. The teeth were cleaned and immersed in ultrapure water for one week for complete hydration. An occlusal matrix was prepared with transparent silicone material (Registrado Clear – Voco), in order to copy the original anatomy. Standardized class I preparation were performed. The same tooth was restored two times, using the different materials. For the multi shade restoration, the tooth shade was evaluated using the composite guide. After that, the universal adhesive system Futurabond U was applied into the cavity in the self-etching mode, according to the manufacturer's instructions. The composite was applied in two layers; first an opaque shade was applied and light-cured and then a second layer of enamel shade was applied. The transparent occlusal matrix was applied and pressed over the uncured composite. The light curing was performed through the matrix, it was removed and additional light curing was performed. The restorations were polished and immersed in ultrapure water for seven days for rehydration. Standardized pictures were obtained and transferred to a computer. The composite restoration was removed without change the shape and the depth of the preparation. The same adhesive system was applied and a new restoration was made using the bulk fill. A single layer of the material was applied and the occlusal matrix positioned and pressed. The composite was light cured through the matrix and additionally cured after the matrix removal. Polishing and rehydration were made exactly as done before and new standardized pictures were obtained. The images were evaluated by ten trained dentists who were blinded about the used material. First they gave scores to them according to FDI parameters, and then the evaluators were asked to indicate which restoration was more esthetic between the both materials. For the first evaluation the data was statistically analyzed using t-test for independent samples. For the second, the data were statistically analyzed using Wilcoxon test.

Results: Non-significant differences between the composites were observed for all scores ($p > 0.05$). Non-significant differences were observed for the numbers of restorations chosen as more esthetic between the two composites ($p > 0.05$).

Conclusions: The evaluators were not able to statistically differentiate the esthetical results produced by the different materials. Therefore, both materials produce the same esthetic results.

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