

# dental materials

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## Abstracts of the Academy of Dental Materials Annual Meeting, 7–10 October 2015 – Hawaii, USA

1

### A 3D-printed TCP/HA osteoconductive scaffold for vertical bone augmentation

S. Durual<sup>1,\*</sup>, J.P. Carrel<sup>2</sup>, M. Moussa<sup>1</sup>,  
P. Rieder<sup>1</sup>, S. Scherrer<sup>1</sup>, A. Wiskott<sup>1</sup><sup>1</sup> University of Geneva, Switzerland<sup>2</sup> University Hospital of Geneva, Geneva,  
Switzerland

**Purpose:** OsteoFlux® (OF) is a 3D printed porous block of layered strands of tricalcium phosphate (TCP) and hydroxyapatite. Its porosity and interconnectivity are defined and it can be readily shaped to conform to the bone bed's morphology. We investigated the performance of OF as a scaffold to promote the vertical growth of cortical bone in a sheep calvarial model.

**Methods and materials:** Six titanium hemispheres were filled with OF, OF+ bmp2 (100 µg), Bio-Oss (particulate bovine bone, BO) or Ceros (particulate TCP, CO) and placed onto the calvaria of 12 adult sheep (6 hemispheres/sheep). Histomorphometric analyses were performed after 8 and 16 weeks.

**Results:** OF led to substantial vertical bone growth by 8 weeks and outperformed BO and CO by a factor of 2 yielding OF 22% ± 2.1; BO 11.5% ± 1.9; CO 12.9% ± 2.1 total new bone. 3 mm away from the bony bed, OF led to a 4-fold increase in new bone relative to BO and CO ( $n=8$ ,  $p < 0.002$ ). At 16 weeks, OF, BO and CO behaved similarly and showed marked new bone synthesis. A moderate degradation was observed at 16 weeks for all bone substitutes. Addition of bmp2 in OF scaffolds led to a dramatic improvement in local bone metabolism and material resorption that was 3 times higher as compared to OF alone.

**Conclusion:** When compared to existing bone substitutes, OF enhances vertical bone growth during the first 2 months after implantation in a sheep calvarial model. The controlled porous structure translated in a high osteoconductivity and resulted in a bone mass 3 mm above the bony bed that was 4-times greater than that obtained with standard substitutes.



Acceleration of bone development kinetics by addition of bmp2 showed that the material has the ability to resorb in large proportions. These results are promising but must be confirmed in clinical tests.

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<http://dx.doi.org/10.1016/j.dental.2015.08.003>

3

### Facial analysis by 3D-stereophotogrammetric imaging: Reproducibility and clinical application

R. Ceinos\*, M.-F. Bertrand, E. Medioni, L.  
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Odontologie Nice, France

**Purpose:** The goal of this study was to assess the reproducibility of an innovative method for facial analysis with 3D-stereophotogrammetry (3D-spg). This tool is then put into practice in a clinical case for aesthetic purposes.

**Methods and materials:** Twelve subjects with no obvious malocclusion participated in this study. For each of them, four photographs were acquired using the LifeViz™, an absolute calibration 3D-spg system mounted on a tripod. The optical system was composed of a double-lens beamsplitter coupled to an inverted polarization double flash. Then, the three-dimensional facial reconstruction was obtained. The images were analyzed with the DermaPix™ image management software. Distance and image centring between each shot were standardized thanks to ground markings and a laser pointer. The landmarks used in this study were selected according to the definitions given by Farkas, the pioneer of modern anthropometry. Distances between landmarks were recorded by two different operators and each operator repeated the



surfaces which might require longer irradiation-time to compensate.

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### Ultra-fast photopolymerization of experimental composites: DEA and FT-NIRS measurement comparison



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<sup>2</sup> University of Birmingham, UK

<sup>3</sup> Bonn-Rhine-Sieg University of Applied Sciences, Rheinbach, Germany

**Purpose:** Polymerization kinetics of ultra-fast photopolymerizations in model resin composites using a monoacyl phosphine oxide (Lucirin-TPO, MAPO) as a photoinitiator have been shown to proceed substantially faster compared with conventional Camphorquinone/Amine systems (CQ). Monitoring such reactions requires many data points to be collected per second. In the present work we investigated the relevance of combining dielectric analysis (DEA) and near-infrared spectroscopy (FT-NIRS) to monitor polymerizations in MAPO and CQ-based composites, under clinically relevant conditions.

**Methods and materials:** Four experimental resin composites were prepared based on 20/80, 40/60, 60/40 or 80/20 mol% BisGMA/TegDMA resins, using either CQ or MAPO in equimolar concentrations (CQ/DMAEMA (0.20/0.80 wt%) or TPO (0.42 wt%). The resins were filled to 75 wt%. 2 mm thick layers of material were photo-polymerized with spectral outputs specific to each photoinitiator (395–415 nm for MAPO or 455–485 nm for CQ). DEA measured changes in ionic viscosity (Nion) and was measured through the variations in the electric field emitted from a comb-like electrode placed beneath the material (data collection rate, 20 s<sup>-1</sup>). FT-NIRS was performed in transmission mode, by measuring the decrease in absorption peak related to C=C bonds (6165 cm<sup>-1</sup>, data collection rate = 3 s<sup>-1</sup>). In both cases, measurements were carried out in triplicates.

**Results:** The monitoring of kinetics in MAPO 20/80 by FT-NIRS could not be carried out due to an insufficient collection rate, while DEA was not limited. Initial viscosity (N0ion) increased exponentially with BisGMA content (R<sup>2</sup> = 0.99), impacting the maximum rate of change dNmaxion/dt (R<sup>2</sup> = 0.95), in MAPO-composites. The comparatively slower kinetics of CQ-composites (0.04 < R<sub>pmax</sub> < 0.27 s<sup>-1</sup> compared to 0.37–0.51 s<sup>-1</sup>) could be monitored using FT-NIRS, but proved difficult with DEA due to the extensive conversion occurring post gelation where high noise was observed, in contrast with that of MAPO. Final conversion in MAPO-composites was either equal to (20/80) or higher than their CQ counterparts. If there is space, general comparison of DC values for both methods would be useful for the Abstract.

**Conclusion:** FT-NIR spectroscopy and DEA are complementary methods in the measurement of ultra-fast

photo-polymerization kinetics of highly filled resin-composite systems cured in thick layers. The complementary use of DEA and FT-NIRS allowed for a more comprehensive characterization of their curing kinetics, DEA being more adapted to initial stages, while FT-NIRS is more suited after gelation. DEA indirectly informed on the system viscosity and FT-NIRS allowed for the determination of functional group conversion.

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### 3D metrological information from fatigue fractured composite surfaces



U. Lohbauer\*, C. Itze, R. Belli

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**Purpose:** Fractographic examination of clinically failed restorations is extremely difficult in terms of load history interpretation. Fatigue fractures are hardly to distinguish from fast fractures and so the energy involved in the fracture event is difficult to approximate. Metrology in three dimensions might help to classify fracture surfaces as fatigued or fast fractured events. The aim of the study was to collect relevant parameters (amplitude and hybrid parameters) from differently fractured composite surfaces, to rank their explanatory power in terms of energy involved in the fracture process.

**Methods and materials:** The resin composite GrandioSO (VOCO, Cuxhaven, Germany) was used to manufacture four-point bending specimens according to ISO 4049. The specimens were fractured (FS in [MPa] (SD) at different cross-head speeds: 5 MPa/s @dry, 0.05 MPa/s @dry, 0.05 MPa/s @wet, 104 fatigue cycles at 0.5 Hz @ wet/14 d, staircase method (FFS). All specimens were stored 24 h at 37 °C prior to testing. A non-contact profilometer (CT100, CyberTechnologies, Ingolstadt, Germany) equipped with a confocal white-light spotsensor (vertical res: 20 nm) was used for mapping all fractured surfaces (stepsize x-y: 5 μm). The following parameters were collected from different regions on the fractured surfaces: Sa (Average Roughness [μm]), Sp (Max. Peak Height [μm]), Sv (Max. Valley Depth [μm]), Sku (Kurtosis), Sdp (Root Mean Square (RMS) Surface Slope [1/mm<sup>2</sup>]), Ssc (mean summit curvature [1/mm]), FD (fractal dimension, box counting). ANOVA/S-N-K statistics were applied in order to distinguish at a level of alpha = 0.05.

**Results:** Results are presented in the Table. Letters indicate statistically homogenous subsets within columns (alpha = 0.05).

**Conclusion:** FS decreased with water storage, particularly after cyclic loading (FFS). Amplitude parameters Sa,

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measurements after one week. The intra- and inter-examiner reproducibilities were assessed using the Intraclass Correlation Coefficient (ICC). Statistical analyses were performed using SPSS software 18.0. The significance level was set at 0.05. Once validated method, the aesthetic of the smile of a patient were analyzed on a 3D-spg reconstruction.

**Results:** This study showed reproducible intra and inter-examiners results for facial measurements. The application of this tool to the aesthetic analysis of a modelling of a face and smile of a patient has allowed an aesthetic and functional guide to treat a clinical case (Fig. 1).

**Conclusion:** The use of 3D-spg equipment in smile analysis before making anterior restorations thus appeared to be a simple and inexpensive method compared with 3D Computed Tomography.

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#### Microstructural characterization and mechanical evaluation of five different CAD/CAM materials



D. Sen\*, N. Sonmez Ceren, V. Turp

Istanbul University Department of Prosthodontics, Turkey

**Purpose:** The aim of this study was to investigate the properties of different CAD/CAM material by mechanical tests, microstructural analysis and SEM evaluation.

**Methods and materials:** 5 Test groups were set from the following materials: Vita Mark II (Vita Zahnfabrik), IPS Empress CAD (Ivoclar), IPS e.max CAD (Ivoclar), Vita Enamic (Vita Zahnfabrik) and Lava Ultimate (3M ESPE). For each group, 22bar samples (1.2 × 4 × 16 mm) were fabricated using Cercon CAD/CAM system (Cercon, Degudent, Switzerland). Half of the remaining samples from each group (n = 10) were thermocycled (5–55 °C, 30s, 10,000 cycles) and half (n = 10) were

tested directly. Vickers hardness, flexural strength and fracture toughness of the samples were determined. Samples were analyzed by X-Ray Diffraction Analysis (XRD), Electron Dispersive Spectroscopy (EDS) and Scanning Electron Microscope (SEM) for microstructural evaluation. Data were analyzed using Two-way ANOVA and Tukey HSD tests ( $p < 0.05$ ).

**Results:** Vita Mark II had the highest Vickers microhardness value ( $p < 0.001$ ), however it had the lowest flexural strength and fracture toughness values ( $p < 0.05$ ). IPS e.max CAD displayed the highest flexural strength and fracture toughness values ( $p < 0.001$ ). Before thermocycling Vita Enamic and Lava Ultimate had similar flexural strength and fracture toughness compared to IPS Empress and Vita Mark II ( $p > 0.05$ ). Thermocycling significantly decreased the mechanical properties of the Lava Ultimate and Vita Enamic ( $p < 0.05$ ), whereas it had no effect on other groups ( $p > 0.05$ ). XRD and EDS results indicated that only IPS e.max CAD had a regular crystalline distribution, other groups had amorphous structures with organic and inorganic phases.

**Conclusion:** Lava Ultimate and Vita Enamic showed similar mechanical properties with Vita Mark II and IPS Empress CAD. However clinicians should be aware that these new materials are significantly affected by aging compared to glass ceramics.

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#### Assessment of non-carious cervical lesion using swept-source optical coherence tomography



I. Wada<sup>1,\*</sup>, Y. Sshimada<sup>1</sup>, A. Sadr<sup>2</sup>, S. Nakashima<sup>1</sup>, J. Tagami<sup>1</sup>, Y. Sumi<sup>3</sup>

<sup>1</sup> Tokyo Medical and Dental University, Department of Cariology and Operative Dentistry, Tokyo, Japan

<sup>2</sup> University of Washington School of Dentistry, Department of Restorative Dentistry, Seattle, USA

<sup>3</sup> National Center for Geriatrics and Gerontology, Obu, Japan

**Purpose:** Non-carious cervical lesions (NCCLs) involve various forms of tooth loss with different etiologies. This study aimed to utilize swept-source optical coherence tomography (SS-OCT) at 1300-nm wavelength range in vitro to evaluate dentin demineralization in NCCLs.

**Methods and materials:** This study consists of two phases; in vitro study and in vivo clinical study. In the in vitro phase, 40 extracted human teeth with NCCLs were investigated. SS-OCT scanning was performed at NCCL parallel to the tooth axis. A dentin attenuation coefficient ( $\mu_t$ ) derived from the SS-OCT signal at NCCL was compared with mineral loss obtained from transverse microradiography (TMR) to determine a  $\mu_t$  threshold to discriminate demineralization of cervical dentin in vivo. In the clinical study, 242 buccal surfaces were investigated in 35 subjects. The incidence of demineralization of the cervical dentin was determined using a  $\mu_t$  threshold obtained from in vitro study.

**Results:** Dentin demineralization is displayed as enhanced brightness by SS-OCT. The incidence of demineralization of



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**Methods and materials:** This study consists of two phases; in vitro study and in vivo clinical study. In the in vitro phase, 40 extracted human teeth with NCCLs were investigated. SS-OCT scanning was performed at NCCL parallel to the tooth axis. A dentin attenuation coefficient ( $\mu_t$ ) derived from the SS-OCT signal at NCCL was compared with mineral loss obtained from transverse microradiography (TMR) to determine a  $\mu_t$  threshold to discriminate demineralization of cervical dentin in vivo. In the clinical study, 242 buccal surfaces were investigated in 35 subjects. The incidence of demineralization of the cervical dentin was determined using a  $\mu_t$  threshold obtained from in vitro study.

**Results:** Dentin demineralization is displayed as enhanced brightness by SS-OCT. The incidence of demineralization of

the cervical dentin was determined using a  $\mu\text{t}$  threshold of  $1.21\text{ mm}^{-1}$ , obtained from the *in vitro* study. 74.7% of *in vivo* NCCLs were accompanied with demineralization; a ratio close to the *in vitro* results from extracted teeth (70.6%).

**Conclusion:** SS-OCT showed the potential to obtain the cross-sectional images for studying the demineralization of NCCL noninvasively. SS-OCT results confirm that dentin mineral loss were associated with NCCLs, and can be considered as an etiological factor in formation and progress of these lesions.

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6

### Chemical bonding increases the immediate enamel bond strengths



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<sup>2</sup> Clinic of Operative Dentistry, Periodontology and Preventive Dentistry, Saarland University, Homburg/Saar, Germany

**Purpose:** The study investigated the contribution of chemical bonding/coating of some acidic functional monomers on the etched enamel surfaces to the immediate enamel micro-tensile bond strengths (MTBS), the surface micro-morphology and the resin-enamel interfaces when the enamel surfaces were etched, briefly primed-&-rinsed.

**Methods and materials:** The labial enamel surfaces of thirty-three bovine incisors were highly polished. After the enamel surfaces were etched for 15 s, water-sprayed, and thoroughly air-dried, they were randomly assigned into 4 groups according to different treatments. Twenty-eight enamel surfaces (7 each group) were applied without further treatment serving control or with one of three acidic functional monomers (40%MDP, 10-methacryloyloxydecyl dihydrogen-phosphate, Ivoclar Vivadent; AquaPrime A + B, DMG; Xeno V, Dentsply) for 5 s, thoroughly water-sprayed for 1 min, and totally air-dried. An adhesive Heliobond (Ivoclar Vivadent) was applied and subsequently Core Build-up resin-composite (Bisco) was placed incrementally on the etched or further prime-&-rinsed enamel surfaces. Twenty specimens were prepared into multiple beams of about  $1 \times 1 \times 8\text{ mm}$  for MTBS tests. The scanning electron microscopy (SEM) was used to analyze the resin-enamel interfaces of another 4 specimens, and the surface micro-morphologies of different treatments (another 5 incisors) that were treated as the above-mentioned, but not placed with resin-composite. The resin-enamel interfaces of another 4 specimens were also analyzed with the transmission electron microscopy (TEM). The MTBS data were analyzed by factorial design ANOVA and LSD test.

**Results:** Compared with the control group, the experimental groups could greatly increase the enamel MTBS ( $P < 0.05$ ). The primer MDP produced significantly higher enamel MTBS than the other two (AquaPrime A + B; Xeno V). The SEM finding revealed a typical etching pattern on the acid-etched enamel surface, and the etched enamel surfaces covered with

a glue-like layer, a thin layer, or some network structures respectively when they were primed with AquaPrime A + B, Xeno V, or MDP. The continuous and dense hybrid layers at the resin-dentin interfaces were detected with some or a lot of micro-porosities (AquaPrime, XenoV) or few micro-porosities (MDP, control) in SEM-micrographs. The TEM findings revealed that the enamel hydroxyapatite (HAp) crystallites at different orientations were tightly covered by a layer of the adhesive (control, Xeno V), a layer of the adhesive mixed with resin composite (AquaPrime), or a layer of the adhesive dotted with sparse, irregular HAp crystallites (MDP).

**Conclusion:** The additional chemical bonding/coating of acidic functional monomers on the etched enamel substrate could significantly increase the immediate mechanical enamel bond strengths.

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7

### Comparison of 3 different implant design systems



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**Purpose:** The aims of this study were to: 1. Compare the fracture strengths of three different Implant Design systems ITI, Unipost and ITO, and 2. Evaluate the failure mode of the implants.

**Methods and materials:** A stainless steel holding device was designed to align the implants at 30 degree angle with respect to the y-axis. For the compression testing Thirty-three specimens were used, 11 for each system. A computer-controlled testing machine (ElectroForce Bose) applied static compression loading by a unidirectional vertical piston until failure. After the compression test and knowing the UTS of the specimens, fatigue cycling was applied to new specimens by increments. Specimens were evaluated microscopically for longitudinal displacement, abutment looseness, and screw and implant fracture. Data were analyzed by analysis of variance (ANOVA).

**Results:** Strength and failure mode varied significantly between the implant systems and deep joints. The mean compressive strength for the Unipost system was 392.5 psi (SD 40.9), and for the ITI System 269.1 psi (SD 30.7). Regarding failure mode, the Unipost system consistently broke at the same site, while ITI system failed at different points of the connection, but mainly at the walls of the implant body and abutment screws. The Unipost system demonstrated excellent fracture resistance to compressive forces; this resistance may be attributed primarily to the diameter of the abutment screw and the 2.5-mm counter bore, representing the same and a unique piece of the implant. Generally, Unipost failure occurred at the first thread of the implant body. This may be attributed to the holding device, 2 mm below the bone line; part of the implant remained without support and is thus more susceptible to fracture.



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**Conclusion:** Within the limits of this *in-vitro* study, the following conclusions were drawn: The ITI and Unipost implant systems were superior to the ITO implant system in terms of failure, defined by fracture and bending to static compressive loading. Further studies of fatigue failures in implants may provide additional findings to assess the clinical significance of the findings reported here.

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8

### Conversion degree of resin cements through different ceramic veneer thicknesses



F. Pires-De-Souza<sup>1,\*</sup>, F. Silami<sup>1</sup>, L. Bachmann<sup>2</sup>, R. Tonani<sup>1</sup>, C. Alandia-Román<sup>1</sup>

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**Purpose:** The dental ceramics have different translucency levels depending on the amount of crystalline phase presented, which can interfere with the light transmission through restoration and monomer's conversion of resin cements used for fix veneers. The aim of this study was to evaluate the conversion degree of resin cements for fixation of different ceramic systems (IPS e.max Press and IPS e.max ZirPress) and thicknesses' veneers.

**Methods and materials:** The coronal portion of a bovine tooth was flattened until dentine and the ceramic veneers were obtained in two thicknesses (0.5 and 1.0 mm). Prior to cementation of veneers (6.0 mm diameter) it was determined the spectral irradiance (Spectroradiometer USB Model 2000 Ocean Optimus) of the curing unit (FlashLite 1401) and the light transmission percentage for each ceramic system. To analyze the conversion degree of resin cements, cementation of 3 laminate veneers were simulated on the tooth protected with plastic wrap, using two resin cements: photoactivated (Variolink II) and self-adhesive (Rely-X U200). After 40 s of light curing, the cements ( $n=3$ ) were analyzed by FTIR (Nicolet 380, ThermoNicolet) by transmission technology with a resolution of  $1\text{ cm}^{-1}$ . Cement samples uncured were also analyzed.

**Results:** The greater the thickness of the veneer, the lower the light transmission to the resin cement with no statistical difference ( $p>0.05$ ) compared to ceramic systems studied. Regarding conversion degree (3-way ANOVA, Bonferroni,  $p<0.05$ ), the results indicated that the self-adhesive cement showed a higher conversion degree than the photoactivated ( $p<0.05$ ) when used to ZirPress ceramics.

**Conclusion:** It was concluded that the type of ceramic system is a significant factor for the degree of conversion of the cement, but the thickness of the ceramic veneer not.

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### Shear strength and retentive force of experimental resin cements



Y. Nagasawa\*, Y. Hibino, H. Shigeta, J. Omatsu, I. Shimano, H. Nakajima

Meikai University School of Dentistry, Sakado, Japan

**Purpose:** This study examined the effect of resin monomer compositions and powder components on the shear strength and retentive force of crowns to implant abutments using experimental cements for temporary luting.

**Methods and materials:** Four experimental temporary resin cements were fabricated with cement liquids made from different resin monomers and powder compositions. Two experimental cement liquids were prepared using two resin monomers: MMA (Wako) and MMA/HEMA (Tokyo Chemical) (1:1). The two experimental cement powders were prepared with three powders: PEMA (Megami Chemical)/PMMA (Megami Chemical) (1:1) and PEMA/silicon (5- $\mu\text{m}$  particle size, Shin-Etsu Silicon) (1:1). Each cement liquid was mixed with each cement powder P/L=1.0(g). To polymerize the cement mixture, BPO and DMPT were also added to the powder and the liquid, respectively. One hour after the start of cement-mixing, the disc specimens (10 mm diam., 1 mm thick) for the shear strength test (the shear punch-out method) were immersed in water at  $37 \pm 2^\circ\text{C}$ . Cast crowns were fabricated conventionally using Ag-Pd-Au alloy (Tokuriki Honten) to fit the implant abutment (Nobel Biocare). The inner surface of each crown was sandblasted with glass beads. The crown specimens and implant abutments were then cemented using the experimental cement. The shear strengths and the retentive force values of each crown to the abutment were determined using a universal testing machine (Instron 3366) in air at 24 h after the start of cement mixing. The results were statistically compared using a two-way ANOVA followed by Scheffè's at  $\alpha=0.05$ .

**Results:** The Fig. 1 shows the shear strength and retentive force of the crowns to the implant abutments of the experimental cements ( $n=6$ , mean  $\pm$  sd). The greatest shear strength was found for the cement made with MMA liquid and PEMA/PMMA powder ( $p<0.05$ ). The lowest shear strength among the experimental cements was observed for

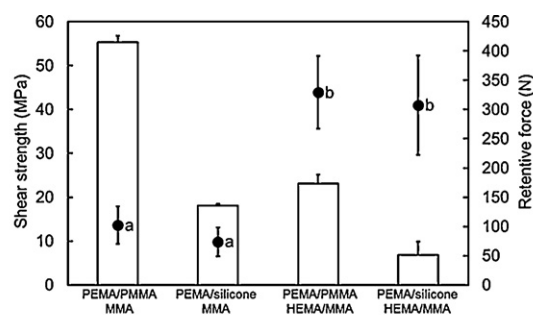


Fig. 1. Shear strength and retentive force of each experimental cement (□ Shear strength ● Retentive force). Same lower-case letters in retentive force indicate no statistical differences ( $p>0.05$ ).

Fig. 1

**Conclusion:** Within the limits of this *in-vitro* study, the following conclusions were drawn: The ITI and Unipost implant systems were superior to the ITO implant system in terms of failure, defined by fracture and bending to static compressive loading. Further studies of fatigue failures in implants may provide additional findings to assess the clinical significance of the findings reported here.

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8

### Conversion degree of resin cements through different ceramic veneer thicknesses



F. Pires-De-Souza<sup>1,\*</sup>, F. Silami<sup>1</sup>, L. Bachmann<sup>2</sup>, R. Tonani<sup>1</sup>, C. Alandia-Román<sup>1</sup>

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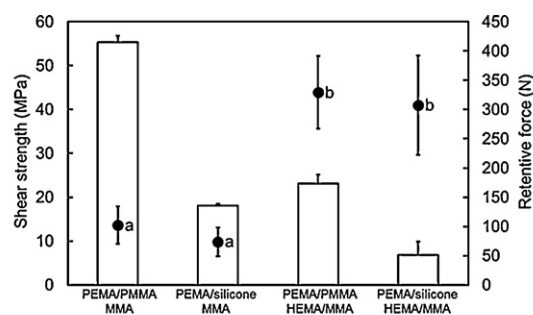


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**Purpose:** The dental ceramics have different translucency levels depending on the amount of crystalline phase presented, which can interfere with the light transmission through restoration and monomer's conversion of resin cements used for fix veneers. The aim of this study was to evaluate the conversion degree of resin cements for fixation of different ceramic systems (IPS e.max Press and IPS e.max ZirPress) and thicknesses' veneers.

**Methods and materials:** The coronal portion of a bovine tooth was flattened until dentine and the ceramic veneers were obtained in two thicknesses (0.5 and 1.0 mm). Prior to cementation of veneers (6.0 mm diameter) it was determined the spectral irradiance (Spectroradiometer USB Model 2000 Ocean Optimus) of the curing unit (FlashLite 1401) and the light transmission percentage for each ceramic system. To analyze the conversion degree of resin cements, cementation of 3 laminate veneers were simulated on the tooth protected with plastic wrap, using two resin cements: photoactivated (Variolink II) and self-adhesive (Rely-X U200). After 40 s of light curing, the cements ( $n=3$ ) were analyzed by FTIR (Nicolet 380, ThermoNicolet) by transmission technology with a resolution of  $1\text{ cm}^{-1}$ . Cement samples uncured were also analyzed.

**Results:** The greater the thickness of the veneer, the lower the light transmission to the resin cement with no statistical difference ( $p>0.05$ ) compared to ceramic systems studied. Regarding conversion degree (3-way ANOVA, Bonferroni,  $p<0.05$ ), the results indicated that the self-adhesive cement showed a higher conversion degree than the photoactivated ( $p<0.05$ ) when used to ZirPress ceramics.

**Conclusion:** It was concluded that the type of ceramic system is a significant factor for the degree of conversion of the cement, but the thickness of the ceramic veneer not.

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### Shear strength and retentive force of experimental resin cements



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**Purpose:** This study examined the effect of resin monomer compositions and powder components on the shear strength and retentive force of crowns to implant abutments using experimental cements for temporary luting.

**Methods and materials:** Four experimental temporary resin cements were fabricated with cement liquids made from different resin monomers and powder compositions. Two experimental cement liquids were prepared using two resin monomers: MMA (Wako) and MMA/HEMA (Tokyo Chemical) (1:1). The two experimental cement powders were prepared with three powders: PEMA (Megami Chemical)/PMMA (Megami Chemical) (1:1) and PEMA/silicon (5- $\mu\text{m}$  particle size, Shin-Etsu Silicon) (1:1). Each cement liquid was mixed with each cement powder P/L=1.0(g). To polymerize the cement mixture, BPO and DMPT were also added to the powder and the liquid, respectively. One hour after the start of cement-mixing, the disc specimens (10 mm diam., 1 mm thick) for the shear strength test (the shear punch-out method) were immersed in water at  $37 \pm 2^\circ\text{C}$ . Cast crowns were fabricated conventionally using Ag-Pd-Au alloy (Tokuriki Honten) to fit the implant abutment (Nobel Biocare). The inner surface of each crown was sandblasted with glass beads. The crown specimens and implant abutments were then cemented using the experimental cement. The shear strengths and the retentive force values of each crown to the abutment were determined using a universal testing machine (Instron 3366) in air at 24 h after the start of cement mixing. The results were statistically compared using a two-way ANOVA followed by Scheffé's at  $\alpha=0.05$ .

**Results:** The Fig. 1 shows the shear strength and retentive force of the crowns to the implant abutments of the experimental cements ( $n=6$ , mean  $\pm$  sd). The greatest shear strength was found for the cement made with MMA liquid and PEMA/PMMA powder ( $p<0.05$ ). The lowest shear strength among the experimental cements was observed for

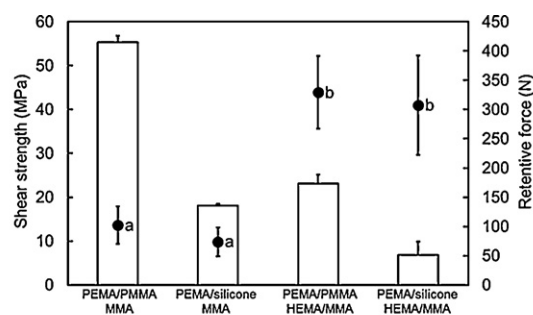


Fig. 1. Shear strength and retentive force of each experimental cement (□ Shear strength ● Retentive force). Same lower-case letters in retentive force indicate no statistical differences ( $p>0.05$ ).

Fig. 1

the cement made of MMA/HEMA liquid and PEMA/silicon powder ( $p < 0.05$ ). The experimental cement containing HEMA into MMA cement liquid had a greater retentive force when it was mixed with either PEMA/PMMA powder or PEMA/silicone powder ( $p < 0.05$ ).

**Conclusion:** Under the experimental conditions, replacing silicone with PMMA in the powder increased the shear strength of the cements and including HEMA in the liquid increased the retentive force of the cement. This study was supported by a Grant-in-Aid for Young Scientists (B) 25861864.

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### Effect of surface treatment on the roughness of all-ceramic materials



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**Purpose:** All-ceramic materials provide excellent mechanical properties and aesthetic outcomes. Amongst other factors, bonding of restorations influences the behaviour under loading conditions and the clinical performance. Different surface treatments can be performed to prepare all-ceramic surfaces for bonding. The aim of this study was to analyze the effect of surface sandblasting and etching on the roughness of hybrid ceramic, glass-ceramic and high translucent zirconia.

**Methods and materials:** Standardized specimens of hybrid ceramic, glass-ceramic and high translucent zirconia (VITA ENAMIC, VITA SUPRINITY and VITA YZ HT, all VITA Zahnfabrik, Bad Säckingen, Germany) with dimensions of  $10 \times 5 \times 17$  mm were prepared. All specimens were polished by a finishing process with 1200 grit SiC. 10 specimens were sandblasted ( $\text{Al}_2\text{O}_3$ ,  $50 \mu\text{m}$ ) or etched with 5% HF (VITA CERAMICS ETCH, VITA Zahnfabrik, Bad Säckingen, Germany) for 60 s. 10 polished specimens served as control. The specimens were observed by SEM (EVO MA10, Carl Zeiss Microscopy, Oberkochen, Germany) and the roughness was measured (Hommel-Tester T8000, ENOPTIK HOMMEL-ETAMIC, Jena, Germany).

**Results:** Polished, sandblasted and etched surfaces showed characteristic surface patterns. Highest results in arithmetic average of the surface roughness (Ra), the mean roughness depth (Rz) and the peak roughness value (Rmax) were found for hybrid ceramic and glass-ceramic with sandblasting.

**Conclusion:** Compared to a polished surface and etching with 5% HF, sandblasting with  $50 \mu\text{m}$   $\text{Al}_2\text{O}_3$ , creates the highest roughness within the hybrid ceramic and the glass-ceramic investigated in this study.

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### Comparison of LTD between Y-TZP and high translucent Y-TZP



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**Purpose:** Zirconia (primarily yttria stabilized tetragonal zirconia polycrystalline ceramics) with its excellent mechanical, biocompatible and aesthetic properties has become of major interest to the dental community. The aim of this study was to compare the kinetics of the tetragonal to monoclinic crystal transformation process of Y-TZP and high translucent Y-TZP (Y-TZP-HT).

**Methods and materials:** The materials under investigation were Y-TZP and Y-TZP-HT (VITA YZ T and VITA YZ HT, both VITA Zahnfabrik, Bad Säckingen, Germany). Standardized specimens ( $n=20$ ) with dimensions of  $14 \times 14 \times 3$  mm were prepared and sintered according to manufacturer's instructions. After autoclaving at  $134^\circ\text{C}$  (Systec DE-23, Systec, Wetztenberg, Germany) the bars were cut on one edge in a  $45^\circ$  angle with FIB (1540XB CrossBeam Focused Ion Beam FE SEM, Carl Zeiss, Jena, Germany) and analyzed at different time periods up to 200 h regarding the amount of monoclinic structures.

**Results:** Both materials showed a low temperature degradation process with different transformation kinetics. The Y-TZP-HT phase transition from tetragonal to monoclinic was twice as fast as that of the Y-TZP material.

**Conclusion:** The kinetics of the transformation process depends on the zirconia material composition. Further studies need to be done in order to investigate the clinical relevance.

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### Effects of Some Modified Coating Methods for Resin Zirconia Bonding



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**Purpose:** To investigate the effects of different surface coating methods for enhancing resin to zirconia adhesion.

**Methods and materials:** Pre-sintered zirconia discs (25 mm in diameter and 1.5 mm in thickness) were prepared and polished under running water. All the samples were randomly

**Conclusion:** Ketone based solvent has the strongest softening effect on bulk-fill composites that contained lesser filler volume fraction and more resin components, thus minimizing their resistance to fracture. Moreover, the type of the resin has an effect on the flexural properties of these composites when immersed in the corresponding solvents.

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### Effect of surface treatment with commercial silane coupling agents



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**Purpose:** The purpose of this study was to evaluate the clinical characteristics, tensile bond strength (TBS) and water resistance of eight commercial ceramic primers [seven commercial silane coupling agents (IN, MO, PL, RE, CP, TO and CS) and one alumina zirconia primer (AZ)].

**Methods and materials:** Glass plates were used as the adhered. The surfaces of the glass plates were treated according to the manufacturer's instruction using microbrushes for each group of ceramic primers. The modified glass surface and the stainless steel rod were manually held together to achieve bonding with the resin composite. Samples were then divided into two different immersion groups. One group was stored in distilled water at 37 °C for 7 days, and the other group was subjected to thermal cycling. The contact angles were obtained by dropping the resin monomer onto glass plates treated with methods described above. The TBS of resin composite and the wettability of resin monomer to glass surfaces treated with silane coupling agents was investigated, with 3-methacryloxypropyltrimethoxysilane (MPS, Shinetsu) used as a control. The values obtained from the experiments were analyzed using one-way ANOVA followed by Tukey's multiple comparison test ( $p < 0.05$ ).

**Results:** No significant difference was observed in TBS between MPS and the commercial ceramic primers except for AZ ( $p > 0.05$ ). However, after thermal stress, four products (CP, TO, CS and AZ) exhibited significantly lower TBS when compared with those stored in distilled water ( $p < 0.05$ ). Six products (IN, MO, RE, CP, TO and CS) displayed significantly higher contact angles between the treated glass and the resin monomer than MPS ( $p < 0.05$ ).

**Conclusion:** The results of the present study demonstrate that there is no significant difference in immediate bond strength among commercial silane coupling agents, but there is a significant difference in the water resistance of these bonds, showing characteristic variations depending on the products. It was suggested that the use of catalysts in silane coupling agents should be further evaluated to obtain optimum performance of these products.

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### Micro-shear bond strength of resin cement to glass ceramics



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**Purpose:** The aim of this study was to evaluate of influence of surface treatments on bond strength of resin cement (NX3, Kerr Dental) to lithium disilicate (e.Max CAD, Ivoclar-Vivadent) [LD] and lithium silicate reinforced by zirconia (Suprinity, VITA) [SZ] ceramics.

**Methods and materials:** CAD/CAM blocks were sectioned using a diamond disc under refrigerated water irrigation in a metallographic precision cutter, obtaining 104 ceramic plates. Specimens were manually grounded using a sequence of SiC abrasive papers until grit 2000, crystallized and randomly assigned to following surface treatments ( $n = 10$ ): (A) aluminum oxide sandblasting; (B) 9.5% hydrofluoric acid etching (HF) for 10 s; (C) HF for 20 s and (D) HF for 30 s. After treatments, plates were submitted to ultra-sonic bath for 5 min prior to silane and applications. A resin cement cylinder (3, 40 mm high and 1.80 diameter) was bonded to plate surface and light-cured with polywave LED curing unit. Bond strength was determined by micro-shear bond strength test ( $\mu$ SBS) and data were subjected to two-way ANOVA and Tukey's test (5%). For topography analysis after treatments, plates ( $n = 3$ ) were mounted in metallic stubs and gold sputtered for scanning electron microscopy (SEM) observation at  $\times 1000$  and  $\times 5000$  magnification.

**Results:** LD and SZ showed higher  $\mu$ SBS (MPa) values when subjected HF for 20 s: 30.6 (12.8) and 35.6 (12.2), respectively. The lowest values were observed when plates were sandblasted: 7.0 (3.4) for LD and 6.6 (2.2) for SZ. Different surface treatments resulted in different surface modifications observed in SEM. No significant differences were obtained between CAD/CAM materials.

**Conclusion:** Surface treatment of LD and SZ plates with 9.5% HF for 20 s resulted in higher  $\mu$ SBS, as well as visible surface modifications observed under SEM.

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### Comparing the retention of glass ionomer and resin-based fissure sealants



A. Al-Jobair\*

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**Purpose:** The aim of this study was to evaluate and compare the retention of glass ionomer fissure sealant and resin-based fissure sealant.

**Methods and materials:** Thirty children between the ages of 6–9 years, with all four erupted caries-free permanent first molars were selected. Sealants were applied randomly using split mouth design technique. Sixty permanent first molars on one side of the mouth were sealed with Fuji Triage

the cement made of MMA/HEMA liquid and PEMA/silicon powder ( $p < 0.05$ ). The experimental cement containing HEMA into MMA cement liquid had a greater retentive force when it was mixed with either PEMA/PMMA powder or PEMA/silicone powder ( $p < 0.05$ ).

**Conclusion:** Under the experimental conditions, replacing silicone with PMMA in the powder increased the shear strength of the cements and including HEMA in the liquid increased the retentive force of the cement. This study was supported by a Grant-in-Aid for Young Scientists (B) 25861864.

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### Effect of surface treatment on the roughness of all-ceramic materials



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**Purpose:** All-ceramic materials provide excellent mechanical properties and aesthetic outcomes. Amongst other factors, bonding of restorations influences the behaviour under loading conditions and the clinical performance. Different surface treatments can be performed to prepare all-ceramic surfaces for bonding. The aim of this study was to analyze the effect of surface sandblasting and etching on the roughness of hybrid ceramic, glass-ceramic and high translucent zirconia.

**Methods and materials:** Standardized specimens of hybrid ceramic, glass-ceramic and high translucent zirconia (VITA ENAMIC, VITA SUPRINITY and VITA YZ HT, all VITA Zahnfabrik, Bad Säckingen, Germany) with dimensions of  $10 \times 5 \times 17$  mm were prepared. All specimens were polished by a finishing process with 1200 grit SiC. 10 specimens were sandblasted ( $\text{Al}_2\text{O}_3$ ,  $50 \mu\text{m}$ ) or etched with 5% HF (VITA CERAMICS ETCH, VITA Zahnfabrik, Bad Säckingen, Germany) for 60 s. 10 polished specimens served as control. The specimens were observed by SEM (EVO MA10, Carl Zeiss Microscopy, Oberkochen, Germany) and the roughness was measured (Hommel-Tester T8000, ENOPTIK HOMMEL-ETAMIC, Jena, Germany).

**Results:** Polished, sandblasted and etched surfaces showed characteristic surface patterns. Highest results in arithmetic average of the surface roughness (Ra), the mean roughness depth (Rz) and the peak roughness value (Rmax) were found for hybrid ceramic and glass-ceramic with sandblasting.

**Conclusion:** Compared to a polished surface and etching with 5% HF, sandblasting with  $50 \mu\text{m}$   $\text{Al}_2\text{O}_3$ , creates the highest roughness within the hybrid ceramic and the glass-ceramic investigated in this study.

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### Comparison of LTD between Y-TZP and high translucent Y-TZP



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**Purpose:** Zirconia (primarily yttria stabilized tetragonal zirconia polycrystalline ceramics) with its excellent mechanical, biocompatible and aesthetic properties has become of major interest to the dental community. The aim of this study was to compare the kinetics of the tetragonal to monoclinic crystal transformation process of Y-TZP and high translucent Y-TZP (Y-TZP-HT).

**Methods and materials:** The materials under investigation were Y-TZP and Y-TZP-HT (VITA YZ T and VITA YZ HT, both VITA Zahnfabrik, Bad Säckingen, Germany). Standardized specimens ( $n=20$ ) with dimensions of  $14 \times 14 \times 3$  mm were prepared and sintered according to manufacturer's instructions. After autoclaving at  $134^\circ\text{C}$  (Systec DE-23, Systec, Wetztenberg, Germany) the bars were cut on one edge in a  $45^\circ$  angle with FIB (1540XB CrossBeam Focused Ion Beam FE SEM, Carl Zeiss, Jena, Germany) and analyzed at different time periods up to 200 h regarding the amount of monoclinic structures.

**Results:** Both materials showed a low temperature degradation process with different transformation kinetics. The Y-TZP-HT phase transition from tetragonal to monoclinic was twice as fast as that of the Y-TZP material.

**Conclusion:** The kinetics of the transformation process depends on the zirconia material composition. Further studies need to be done in order to investigate the clinical relevance.

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### Effects of Some Modified Coating Methods for Resin Zirconia Bonding



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**Purpose:** To investigate the effects of different surface coating methods for enhancing resin to zirconia adhesion.

**Methods and materials:** Pre-sintered zirconia discs (25 mm in diameter and 1.5 mm in thickness) were prepared and polished under running water. All the samples were randomly



the cement made of MMA/HEMA liquid and PEMA/silicon powder ( $p < 0.05$ ). The experimental cement containing HEMA into MMA cement liquid had a greater retentive force when it was mixed with either PEMA/PMMA powder or PEMA/silicone powder ( $p < 0.05$ ).

**Conclusion:** Under the experimental conditions, replacing silicone with PMMA in the powder increased the shear strength of the cements and including HEMA in the liquid increased the retentive force of the cement. This study was supported by a Grant-in-Aid for Young Scientists (B) 25861864.

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### Effect of surface treatment on the roughness of all-ceramic materials



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**Purpose:** All-ceramic materials provide excellent mechanical properties and aesthetic outcomes. Amongst other factors, bonding of restorations influences the behaviour under loading conditions and the clinical performance. Different surface treatments can be performed to prepare all-ceramic surfaces for bonding. The aim of this study was to analyze the effect of surface sandblasting and etching on the roughness of hybrid ceramic, glass-ceramic and high translucent zirconia.

**Methods and materials:** Standardized specimens of hybrid ceramic, glass-ceramic and high translucent zirconia (VITA ENAMIC, VITA SUPRINITY and VITA YZ HT, all VITA Zahnfabrik, Bad Säckingen, Germany) with dimensions of  $10 \times 5 \times 17$  mm were prepared. All specimens were polished by a finishing process with 1200 grit SiC. 10 specimens were sandblasted ( $\text{Al}_2\text{O}_3$ ,  $50 \mu\text{m}$ ) or etched with 5% HF (VITA CERAMICS ETCH, VITA Zahnfabrik, Bad Säckingen, Germany) for 60 s. 10 polished specimens served as control. The specimens were observed by SEM (EVO MA10, Carl Zeiss Microscopy, Oberkochen, Germany) and the roughness was measured (Hommel-Tester T8000, ENOPTIK HOMMEL-ETAMIC, Jena, Germany).

**Results:** Polished, sandblasted and etched surfaces showed characteristic surface patterns. Highest results in arithmetic average of the surface roughness (Ra), the mean roughness depth (Rz) and the peak roughness value (Rmax) were found for hybrid ceramic and glass-ceramic with sandblasting.

**Conclusion:** Compared to a polished surface and etching with 5% HF, sandblasting with  $50 \mu\text{m}$   $\text{Al}_2\text{O}_3$ , creates the highest roughness within the hybrid ceramic and the glass-ceramic investigated in this study.

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### Comparison of LTD between Y-TZP and high translucent Y-TZP



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**Purpose:** Zirconia (primarily yttria stabilized tetragonal zirconia polycrystalline ceramics) with its excellent mechanical, biocompatible and aesthetic properties has become of major interest to the dental community. The aim of this study was to compare the kinetics of the tetragonal to monoclinic crystal transformation process of Y-TZP and high translucent Y-TZP (Y-TZP-HT).

**Methods and materials:** The materials under investigation were Y-TZP and Y-TZP-HT (VITA YZ T and VITA YZ HT, both VITA Zahnfabrik, Bad Säckingen, Germany). Standardized specimens ( $n=20$ ) with dimensions of  $14 \times 14 \times 3$  mm were prepared and sintered according to manufacturer's instructions. After autoclaving at  $134^\circ\text{C}$  (Systec DE-23, Systec, Wetztenberg, Germany) the bars were cut on one edge in a  $45^\circ$  angle with FIB (1540XB CrossBeam Focused Ion Beam FE SEM, Carl Zeiss, Jena, Germany) and analyzed at different time periods up to 200 h regarding the amount of monoclinic structures.

**Results:** Both materials showed a low temperature degradation process with different transformation kinetics. The Y-TZP-HT phase transition from tetragonal to monoclinic was twice as fast as that of the Y-TZP material.

**Conclusion:** The kinetics of the transformation process depends on the zirconia material composition. Further studies need to be done in order to investigate the clinical relevance.

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### Effects of Some Modified Coating Methods for Resin Zirconia Bonding



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**Purpose:** To investigate the effects of different surface coating methods for enhancing resin to zirconia adhesion.

**Methods and materials:** Pre-sintered zirconia discs (25 mm in diameter and 1.5 mm in thickness) were prepared and polished under running water. All the samples were randomly

divided into five study groups according to the following surface treatments: control group (fully sintered, without further modification), group SA (fully sintered, followed by sandblasting with 50  $\mu\text{m}$  alumina particles), group SS (fully sintered, followed by sandblasting with 110  $\mu\text{m}$  silica-coated alumina particles), group ZS (coated with zirconia and silica particles, then fully sintered), and group ZR (coated with zirconium silicate particles and fully sintered). After ultrasonic cleansing in 70% ethanol solution, each zirconia disc was treated with a silane coupling agent and then bonded with cylindrical resin stubs (3.6 mm in diameter and 3 mm in thickness). There were forty-eight resin stubs prepared in each groups (six on each zirconia disc). In each group, all the specimens were further divided into four sub-groups and subjected to different aging conditions, including initial status, thermocycling for 3000 cycles, thermocycling for 6000 cycles, and thermocycling for 10,000 cycles. Adhesion strength test was carried out after the aging treatments applying shear force. Scanning electron microscope (SEM) examination was conducted for the observation of zirconia surface morphological appearance. The elemental composition of zirconia surface was investigated and analyzed with the energy dispersive X-ray spectrometry (EDX) technique.

**Results:** The lowest initial adhesion value ( $7.8 \pm 2.3$  MPa) was observed in the control group and group SS had the highest initial value ( $17.9 \pm 4.1$  MPa). After aging treatments, adhesion strengths of all the five groups were reduced and all the samples in the control group were debonded. Especially for the thermal cycling for 10,000 cycles, the reductions in shear bond strengths of two sandblasting groups were more than 50%. On the other hand, group ZS and group ZR exhibited the higher resistance to aging effects. In particular, in group ZR, the bond strength was just slightly reduced (13.4%) compared with the initial value and found to be the highest ( $12.9 \pm 1.5$  MPa). Group ZR also had the highest silicon content.

**Conclusion:** Adding silicon content in a coating material might be a promising method of surface treatment for improving the stability of resin zirconia bonding.

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#### Internal void formation on dental composites using ultra-sonic devices



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**Purpose:** The aim of this study was to quantify the internal void formation of four types of composites following the regular and sonic application, using three-dimensional (3D) micro-computed tomography ( $\mu\text{CT}$ ).

**Methods and materials:** Four composites were tested: a regular composite (Herculite, Kerr), a flowable bulk-fill composite (Surefill SDR, Dentsply) and two packable bulk-fill composites (SonicFill, Kerr and Tetric EvoCeram Bulk Fill, Ivoclar

Vivadent). Eight groups were formed following the four composites and two application modes ( $n = 5$ ). Forty polycarbonate plastic, 3D printed cavities, with internal dimension of 5 mm (diameter) and 4 mm (depth) were used in the study. The regular composite filled the cavity in two layers, while the bulk-fill composites were placed in only one increment. For the sonic application, a handpiece was used (SonicFill System, Kerr). Composites were polymerized using a VALO light (Ultradent) in regular mode. The samples were scanned via  $\mu\text{CT}$ . Acquired  $\mu\text{CT}$  data were imported into Amira software for analysis and void volume measurements. Data were analyzed by two-way ANOVA and Pairwise Comparisons (95% confidence Interval).

**Results:** Statistical significance between regular application versus sonic filling was found. Sonic application increased void formation for all the materials tested. Tetric EvoCeram Bulk Fill showed less void formation compared to all groups, followed by Herculite and Surefill SDR, which presented the biggest void formation when applied by sonication.

**Conclusion:** Results suggested that sonic application increased void formation. Tetric EvoCeram Bulk Fill presented the lowest porosity formation.

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#### Five-Year Resin Bond Strengths to Zirconia Ceramics



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**Purpose:** The aim of this *in vitro* study was to evaluate the long-term resin bond strength to zirconia ceramic with the treatment of an MDP-containing zirconia primer.

**Methods and materials:** Yttria-stabilized zirconia ceramics were subjected to air abrading and ultrasonic cleansing, and treated with 1 coat of MDP-containing polymerizable zirconia primer (ZPrime Plus, Bisco). They were randomly divided into 2 groups. In Group 1, ZPrime Plus was light-polymerized (10 s/500 mW/cm<sup>2</sup>). In Group 2, ZPrime Plus was not light-polymerized. Shear bond strength was tested using the ultradent jig method (bonding area 4.5 mm<sup>2</sup>). A dual-cure resin cement, Duolink (Bisco), which was used to fabricate the posts (2 mm high), was light-polymerized (40 s/500 mW/cm<sup>2</sup>). The specimens were then stored in water at 37 °C for up to 5 years, and tested by Instron tester (crosshead-speed 1 mm/min). The data were analyzed statistically by two-way ANOVA and Student-t Tests.

**Results:** Mean shear bond strength on zirconia in MPa (standard deviation). Means within the same column with different letters (a, b), or within the same row with different numbers (1, 2) are statistically different ( $p < 0.05$ ). \* Note: According to manufacturer's Instruction for Use, ZPrime Plus doesn't need to be light-polymerized.

**Conclusion:** After 5 years of aging in 37 °C-water, the resin-zirconia bond strength did not decrease. Light-curing of ZPrime Plus did not change the bond strength (short-term or long-term). A durable resin bond strength to zirconia

divided into five study groups according to the following surface treatments: control group (fully sintered, without further modification), group SA (fully sintered, followed by sandblasting with 50  $\mu\text{m}$  alumina particles), group SS (fully sintered, followed by sandblasting with 110  $\mu\text{m}$  silica-coated alumina particles), group ZS (coated with zirconia and silica particles, then fully sintered), and group ZR (coated with zirconium silicate particles and fully sintered). After ultrasonic cleansing in 70% ethanol solution, each zirconia disc was treated with a silane coupling agent and then bonded with cylindrical resin stubs (3.6 mm in diameter and 3 mm in thickness). There were forty-eight resin stubs prepared in each groups (six on each zirconia disc). In each group, all the specimens were further divided into four sub-groups and subjected to different aging conditions, including initial status, thermocycling for 3000 cycles, thermocycling for 6000 cycles, and thermocycling for 10,000 cycles. Adhesion strength test was carried out after the aging treatments applying shear force. Scanning electron microscope (SEM) examination was conducted for the observation of zirconia surface morphological appearance. The elemental composition of zirconia surface was investigated and analyzed with the energy dispersive X-ray spectrometry (EDX) technique.

**Results:** The lowest initial adhesion value ( $7.8 \pm 2.3$  MPa) was observed in the control group and group SS had the highest initial value ( $17.9 \pm 4.1$  MPa). After aging treatments, adhesion strengths of all the five groups were reduced and all the samples in the control group were debonded. Especially for the thermal cycling for 10,000 cycles, the reductions in shear bond strengths of two sandblasting groups were more than 50%. On the other hand, group ZS and group ZR exhibited the higher resistance to aging effects. In particular, in group ZR, the bond strength was just slightly reduced (13.4%) compared with the initial value and found to be the highest ( $12.9 \pm 1.5$  MPa). Group ZR also had the highest silicon content.

**Conclusion:** Adding silicon content in a coating material might be a promising method of surface treatment for improving the stability of resin zirconia bonding.

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#### Internal void formation on dental composites using ultra-sonic devices



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**Purpose:** The aim of this study was to quantify the internal void formation of four types of composites following the regular and sonic application, using three-dimensional (3D) micro-computed tomography ( $\mu\text{CT}$ ).

**Methods and materials:** Four composites were tested: a regular composite (Herculite, Kerr), a flowable bulk-fill composite (Surefill SDR, Dentsply) and two packable bulk-fill composites (SonicFill, Kerr and Tetric EvoCeram Bulk Fill, Ivoclar

Vivadent). Eight groups were formed following the four composites and two application modes ( $n = 5$ ). Forty polycarbonate plastic, 3D printed cavities, with internal dimension of 5 mm (diameter) and 4 mm (depth) were used in the study. The regular composite filled the cavity in two layers, while the bulk-fill composites were placed in only one increment. For the sonic application, a handpiece was used (SonicFill System, Kerr). Composites were polymerized using a VALO light (Ultradent) in regular mode. The samples were scanned via  $\mu\text{CT}$ . Acquired  $\mu\text{CT}$  data were imported into Amira software for analysis and void volume measurements. Data were analyzed by two-way ANOVA and Pairwise Comparisons (95% confidence Interval).

**Results:** Statistical significance between regular application versus sonic filling was found. Sonic application increased void formation for all the materials tested. Tetric EvoCeram Bulk Fill showed less void formation compared to all groups, followed by Herculite and Surefill SDR, which presented the biggest void formation when applied by sonication.

**Conclusion:** Results suggested that sonic application increased void formation. Tetric EvoCeram Bulk Fill presented the lowest porosity formation.

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**Conclusion:** After 5 years of aging in 37 °C-water, the resin-zirconia bond strength did not decrease. Light-curing of ZPrime Plus did not change the bond strength (short-term or long-term). A durable resin bond strength to zirconia

ceramics was achieved after using ZPrime Plus, an MDP-containing zirconia primer.

Water storage at 37 °C	ZPrime Plus (no light-cure)	ZPrime Plus (light-cure)*
2 h (control)	28.7 (5.7) n=8;ab, 1	25.1 (5.0) n=8;a, 1
2.5 years	23.9 (5.1) n=5;b, 1	32.4 (7.4) n=5;a, 1
3.5 years	33.9 (4.6) n=5;a, 1	30.0 (3.7) n=5;a, 1
5 years	29.2 (8.7) n=6;ab, 1	31.7 (7.2) n=6;a, 1

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### Effectiveness of oral care and lilt on oral mucositis



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<sup>2</sup> São Vicente De Paulo Hospital, Passo Fundo, Brazil

**Purpose:** This study was conducted to determine whether oral care and low-intensity laser therapy (LILT) can reduce the rates of discontinuation of anticancer treatment. Approximately 870,000 new cases of malignant airway and digestive tract tumors are diagnosed annually worldwide. Patients undergoing radiotherapy (RT) for head and neck cancers develop oral mucositis (OM) and other complications as side effects that can lead to discontinuation of anticancer treatment.

**Methods and materials:** We selected patients with carcinoma of the head and neck who underwent radiotherapy with or without chemotherapy (CT) in the São Vicente de Paulo Hospital, Passo Fundo, Brazil. A total of 187 patients were evaluated and divided into two groups: Group I (patients receiving oral care) and Group II (patients not receiving oral care). The patients in Group I were subjected to daily assessments, received guidance, and followed a protocol for dental treatment and adjuvant application of LILT throughout the period they performed RT. The patients in Group II were evaluated and followed but did not receive the proposed treatment.

**Results:** A total of 187 patients were included in this study: 144 males (77%) and 43 females (23%). The mean age was 59.9 ± 12.9 (13–95 years). One-hundred-and-twenty-five patients were included in Group I, and 62 patients were included in Group II. High scores for pain and OM were observed in the patients belonging to group II. Three (2.4%) of the patients in Group I (125 patients) presented an interruption in RT, whereas 20 (34.6%) of the patients in Group II (62 patients) presented an interruption in RT induced by interurrences, such as pain, dehydration, severe OM, immunity and death ( $p < 0.001$ ). The group that received LILT (Group I) presented a lower rate of anti-neoplastic treatment interruption compared with Group II ( $p < 0.001$ ).

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### SEM and bond strength evaluation of adaptation between liners-composites



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**Purpose:** The aim of this *in vitro* study was to evaluate the adaptation between liners and composite restorations by SEM and shear bond strength test.

**Methods and materials:** For SEM study, occlusal dentin surface was wet-polished with 320-grit SiC paper, and then they were randomly divided into 3 groups, and treated with 3 different techniques as shown in Table 1. The specimens were then cut vertically by diamond saw and the interface/surface and wet polished with 600-grit, 1200-grit SiC paper, followed by 1-micron Alumina, and 0.3-micron alumina slurries. The specimens were then cleaned with DI water and dried. Then they were coated with gold sputter and the interface between composite and liner was examined by SEM (JEOL, JSM-6010plus). For shear bond strength study, Dycal or TheraCal disc (6 mm diameter, 3 mm height) were set, and polished with 320-grit SiC paper. Resin composite was bonded to the disc with 3 different techniques as shown in Table 1, by using Ultradent jig shear bond strength test method. The specimens were then stored in 37 °C water for 24 h before breaking by Instron tester (crosshead-speed 1 mm/min). The data were analyzed statistically by one-way ANOVA and Student t-tests.

**Results:** Table 1. Mean shear bond strength (SBS) between liner and composite in MPa (standard deviation). Means with different letters (a, b) are statistically different ( $p < 0.05$ ). \*Dycal (Dentsply) was used as a thin layer and was set in 37 °C oven for 5 min. Both Vitrebond (3M ESPE) and TheraCal LC (Bisco) were used as a thin layer and were set by light-polymerization for 20 s/500 mw/cm<sup>2</sup>. All-Bond Universal (Bisco) was used with 1 coat, and was air dried and light-cured for 10 s. Composite (Aelite All-Purpose Body, Bisco) was light-cured for 40 s.

**Table 1**

Groups	Techniques*	SBS (n = 6)
1	Dentin – Dycal – Vitrebond – AllBond Universal – Composite	0.0 (0.0) <sup>a</sup>
2	Dentin – Dycal – AllBond Universal – Composite	4.5 (1.3) <sup>b</sup>
3	Dentin – TheraCal LC – AllBond Universal – Composite	15.0 (4.1) <sup>c</sup>

**Conclusion:** SEM pictures showed that there was big gap formation between Dycal and Vitrebond, and no gap between Dycal or TheraCal and Composite. The bond strength between

ceramics was achieved after using ZPrime Plus, an MDP-containing zirconia primer.

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Groups	Techniques*	SBS (n = 6)
1	Dentin – Dycal – Vitrebond – AllBond Universal – Composite	0.0 (0.0) <sup>a</sup>
2	Dentin – Dycal – AllBond Universal – Composite	4.5 (1.3) <sup>b</sup>
3	Dentin – TheraCal LC – AllBond Universal – Composite	15.0 (4.1) <sup>c</sup>

**Conclusion:** SEM pictures showed that there was big gap formation between Dycal and Vitrebond, and no gap between Dycal or TheraCal and Composite. The bond strength between

Dycal-Vitrebond-Composite was the lowest, and between TheraCal-Composite was the highest.

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### Bond strength of etch-and-rinse adhesive systems to plasma-treated dentin



R. Hirata<sup>1</sup>, A.P.A. Ayres<sup>2</sup>, L.S. Machado<sup>3</sup>, P.G. Coelho<sup>1</sup>, M. Giannini<sup>2,\*</sup>

<sup>1</sup>New York University, USA

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**Methods and materials:** Twenty-eight third human molar were sectioned to remove the enamel occlusal surface and grounded (600-grit SiC) to expose a flat dentin surface. Teeth were randomly divided into four groups ( $n=7$ ), following the application or not of APP for 30 s after etching with phosphoric acid and two adhesives (XP Bond, Dentsply and Optibond FL, Kerr). After the composite resin build-up, teeth were sectioned perpendicular to the bonded interface to obtain beams specimens. The specimens were tested after 24 h and one year of water storage until failure. Bond strength data were analyzed by three-way ANOVA and Tukey post-hoc test ( $\alpha=0.05$ ).

**Results:** BS means for XP Bond were (in MPa): Control (24 h):  $45.1 \pm 4.6$ ; Control (1 year):  $42.2 \pm 3.4$ , APP (24 h):  $59.0 \pm 10.2$ ; APP (1 year):  $47.1 \pm 13.2$  and BS means for Optibond FL were: Control (24 h):  $68.3 \pm 7.7$ ; Control (1 year):  $56.4 \pm 6.7$ , APP (24 h):  $57.4 \pm 13.7$ ; APP (1 year):  $53.5 \pm 10.9$ .

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### Fluoride release from experimental glass-ionomers made with different resin monomers



H. Shigeta\*, Y. Nagasawa, Y. Hibino, H. Nakajima

Meikai University School of Dentistry, Japan

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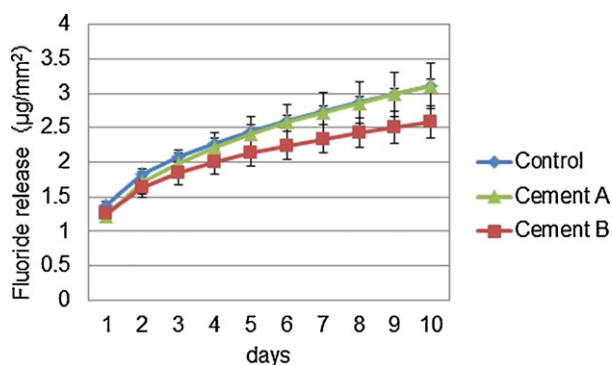


Fig. 1

30 mL of deionized water at 37 °C for up to 10 days. The fluoride released into the solution was quantitated using a fluoride ion-selective electrode for up to 10 days. The solutions in which the specimens were immersed were exchanged every day with fresh deionized water. The data were analyzed by two-way ANOVA followed by Scheffé's test ( $\alpha = 0.05$ ).

**Results:** The cumulative amounts of fluoride released from each cement per unit surface area are shown in the Fig. 1. After 7-day immersion, Cement A and control released greater amounts of fluoride than Cement B ( $p > 0.05$ ).

**Conclusion:** Under the present experimental conditions, the study suggested that the inclusion of the hydrophilic multifunctional monomer in the cement was effective at maintaining fluoride release behavior similar to that of the control cement.

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### Pulpal responses to pulpotomy with ProRoot MTA<sup>®</sup>, RetroMTA<sup>®</sup>, and TheraCal<sup>®</sup>



Y. Shin\*, H. Lee, S.O. Kim, H.S. Lee, H.J. Choi, J.S. Song

Yonsei University, Seoul, South Korea

**Purpose:** This study was conducted to evaluate and compare calcific barrier formation, inflammation, and odontoblastic layer formation of ProRoot MTA<sup>®</sup>, RetroMTA<sup>®</sup> and TheraCal<sup>®</sup> in dog pulpotomy models.

**Methods and materials:** Partial pulpotomies were performed on 60 beagle teeth. The exposed pulp tissues were randomly capped with either ProRoot MTA<sup>®</sup> ( $n = 15$ ), RetroMTA<sup>®</sup> ( $n = 15$ ), TheraCal<sup>®</sup> ( $n = 15$ ), or interim restorative material (IRM) as a negative control ( $n = 15$ ). After 4 weeks, the teeth were extracted and processed for histological and immunohistochemical (IHC) examinations using osteocalcin (OC) and dentin sialoprotein (DSP). Calcific barrier formation, inflammatory reaction, and the odontoblastic layer were evaluated and scored in a blind manner. The areas of newly formed calcific barriers were measured for each group.

**Results:** In most of the ProRoot MTA<sup>®</sup> and RetroMTA<sup>®</sup> specimens, continuous calcific barriers were formed and the pulps contained palisading patterns in the odontoblastic layer that

were free of inflammation. However, the TheraCal<sup>®</sup> specimens had lower quality calcific barrier formation, extensive inflammation, and less favorable odontoblastic layer formation. Overall, areas of newly formed calcific barrier were higher in the ProRoot MTA<sup>®</sup> and RetroMTA<sup>®</sup> specimens than in the TheraCal<sup>®</sup> specimens. Also IHC revealed that OC and DSP were more clearly visible in the ProRoot MTA<sup>®</sup> and RetroMTA<sup>®</sup> specimens than in the TheraCal<sup>®</sup> specimens.

**Conclusion:** RetroMTA<sup>®</sup> could provide an alternative to ProRoot MTA<sup>®</sup>. Both materials produced favorable pulpal responses that were similar in nature, whereas TheraCal<sup>®</sup> produced less favorable pulpal responses.

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### Silane application can influence the bond strength to lithium disilicate



G. Bruzi<sup>1,\*</sup>, A.O. Carvalho<sup>2</sup>, M. Giannini<sup>3</sup>, H.P. Maia<sup>4</sup>, P. Magne<sup>5</sup>

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<sup>2</sup> Federal University of Feira De Santana, Brazil

<sup>3</sup> Campinas State University, Brazil

<sup>4</sup> Federal University of Santa Catarina, Brazil

<sup>5</sup> University of Southern California, United States

**Purpose:** Evaluate the effect of silane application method on the resin-ceramic shear bond strength (SBS).

**Methods and materials:** Ten 2 mm-thick slices of IPS e.max CAD (Ivoclar Vivadent), were crystalized and embedded in acrylic resin and polished with 400 and 600-grit SiC paper. The surface of each block was cleaned and randomly assigned to 5 groups; E/S: hydrofluoric acid (HF) followed by silane application (20 s), air drying and hot drying; E/S2: same as E/S but applied twice (20 s + 20 s) with air drying and hot drying between the applications; E/S3: same as E/S but silane applied 3 times (20 s + 20 s + 20 s) with air drying and hot drying between the applications; E/S+: HF-etching followed by silane application (60 s), air drying and hot air drying, rinsing with boiling water and hot air drying; E/S+s: same as E/S+ but cleaning with steam instead of boiling water. Cylinders of composite resin ( $n = 12$ /slice) (Z100, 3M-ESPE) were bonded with adhesive resin (Optibond FL adhesive, Kerr) in the ceramic surface. SBS testing was carried out after 24 h of storage in water.

**Results:** Bond strength mean values were statistically different: E/S (21.06 MPa) > E/S2 (14.10 MPa) = E/S3 (15.74 MPa) = E/S+ (15.93 MPa) = E/S+s (14.11 MPa).

**Conclusion:** Silane reapplication reduces the bond strength after 24 h testing.

<http://dx.doi.org/10.1016/j.dental.2015.08.022>

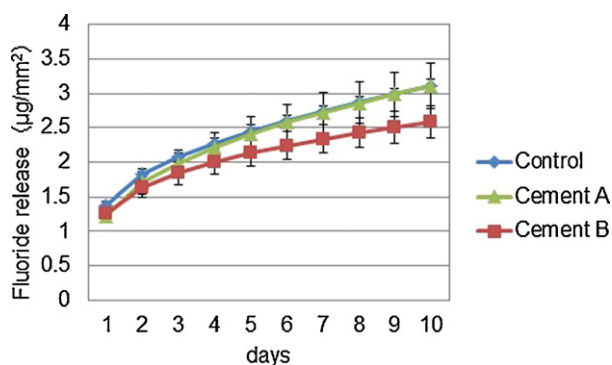


Fig. 1

30 mL of deionized water at 37 °C for up to 10 days. The fluoride released into the solution was quantitated using a fluoride ion-selective electrode for up to 10 days. The solutions in which the specimens were immersed were exchanged every day with fresh deionized water. The data were analyzed by two-way ANOVA followed by Scheffé's test ( $\alpha = 0.05$ ).

**Results:** The cumulative amounts of fluoride released from each cement per unit surface area are shown in the Fig. 1. After 7-day immersion, Cement A and control released greater amounts of fluoride than Cement B ( $p > 0.05$ ).

**Conclusion:** Under the present experimental conditions, the study suggested that the inclusion of the hydrophilic multifunctional monomer in the cement was effective at maintaining fluoride release behavior similar to that of the control cement.

<http://dx.doi.org/10.1016/j.dental.2015.08.020>

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### Pulpal responses to pulpotomy with ProRoot MTA<sup>®</sup>, RetroMTA<sup>®</sup>, and TheraCal<sup>®</sup>



Y. Shin\*, H. Lee, S.O. Kim, H.S. Lee, H.J. Choi, J.S. Song

Yonsei University, Seoul, South Korea

**Purpose:** This study was conducted to evaluate and compare calcific barrier formation, inflammation, and odontoblastic layer formation of ProRoot MTA<sup>®</sup>, RetroMTA<sup>®</sup> and TheraCal<sup>®</sup> in dog pulpotomy models.

**Methods and materials:** Partial pulpotomies were performed on 60 beagle teeth. The exposed pulp tissues were randomly capped with either ProRoot MTA<sup>®</sup> ( $n = 15$ ), RetroMTA<sup>®</sup> ( $n = 15$ ), TheraCal<sup>®</sup> ( $n = 15$ ), or interim restorative material (IRM) as a negative control ( $n = 15$ ). After 4 weeks, the teeth were extracted and processed for histological and immunohistochemical (IHC) examinations using osteocalcin (OC) and dentin sialoprotein (DSP). Calcific barrier formation, inflammatory reaction, and the odontoblastic layer were evaluated and scored in a blind manner. The areas of newly formed calcific barriers were measured for each group.

**Results:** In most of the ProRoot MTA<sup>®</sup> and RetroMTA<sup>®</sup> specimens, continuous calcific barriers were formed and the pulps contained palisading patterns in the odontoblastic layer that

were free of inflammation. However, the TheraCal<sup>®</sup> specimens had lower quality calcific barrier formation, extensive inflammation, and less favorable odontoblastic layer formation. Overall, areas of newly formed calcific barrier were higher in the ProRoot MTA<sup>®</sup> and RetroMTA<sup>®</sup> specimens than in the TheraCal<sup>®</sup> specimens. Also IHC revealed that OC and DSP were more clearly visible in the ProRoot MTA<sup>®</sup> and RetroMTA<sup>®</sup> specimens than in the TheraCal<sup>®</sup> specimens.

**Conclusion:** RetroMTA<sup>®</sup> could provide an alternative to ProRoot MTA<sup>®</sup>. Both materials produced favorable pulpal responses that were similar in nature, whereas TheraCal<sup>®</sup> produced less favorable pulpal responses.

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21

### Silane application can influence the bond strength to lithium disilicate



G. Bruzi<sup>1,\*</sup>, A.O. Carvalho<sup>2</sup>, M. Giannini<sup>3</sup>, H.P. Maia<sup>4</sup>, P. Magne<sup>5</sup>

<sup>1</sup> Federal University of Alfnas, Brazil

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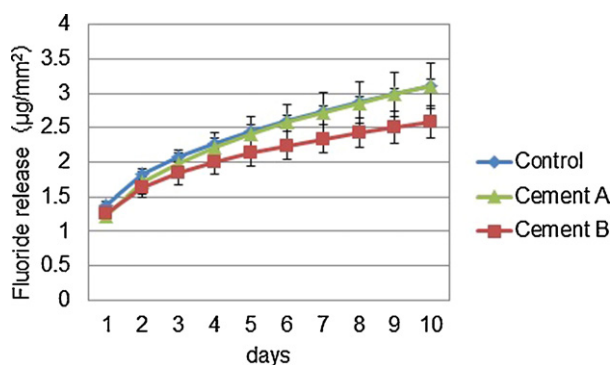


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22

### Flexural strength of experimental highly viscous conventional glass ionomers



Y. Hibino\*, Y. Nagasawa, H. Shigeta, J. Omatsu, H. Nakajima

Meikai University School of Dentistry, Sakado, Japan

**Purpose:** The objective of this study was to examine the flexural strength of experimental highly viscous conventional glass ionomers for restorative filling under various storage conditions.

**Methods and materials:** A commercially available highly viscous conventional glass ionomer (Fuji IX GP EXTRA, GC, A) and an experimental highly viscous conventional glass ionomer (SI-R21402, Shofu, B) were tested. Rectangular specimens (2 mm wide, 2 mm thick and 25 mm long) were prepared according to each manufacturer's recommended ratio (Cement A: P/L = 3.4/1.0, Cement B: P/L = 2.8/1.0, respectively). One hour after the start of mixing for both cements, specimens were stored in deionized water at  $37 \pm 2^\circ\text{C}$  up to 1 week, or were subjected to thermal cycling between  $5^\circ\text{C}$  and  $55^\circ\text{C}$  water up to 3000 cycles for 60s each using thermal cycling equipment (K178-15, Tokyo-Giken). The flexural strengths of each glass ionomer were determined at 1 h, 24 h or 1 week after the start of mixing using a universal testing machine (3366, Instron) at a cross-head speed of 1.0 mm/min using a three-point bending test at  $23 \pm 2^\circ\text{C}$  in air. Five specimens were fabricated and tested for each experimental condition. The results were analyzed using ANOVA/Scheffe's test ( $\alpha = 0.05$ ).

**Results:** Table 1 shows the measured flexural strength (MPa, mean  $\pm$  sd,  $n = 5$ ) for the cements. The flexural strength for Cement A increased significantly with time elapsed after cement mixing ( $p < 0.05$ ). The flexural strength for Cement B increased significantly at 1 week compared to that at 1 h ( $p < 0.05$ ). The flexural strengths of both cements after 3000 thermal cycles were greater than those at 1 h ( $p < 0.05$ ). The strength of Cement A at 24 h after the start of mixing was lower than after 3000 thermal cycles ( $p < 0.05$ ); however, Cement B showed no significant differences in flexural strengths between 24 h immersion in deionized water and 3000 thermal cycles ( $p > 0.05$ ). Statistically no significant differences in flexural strengths of both Cements A and B were observed between thermal cycling and 1 week ( $p > 0.05$ ).

**Conclusion:** Under the present experimental conditions, the flexural strength of highly viscous conventional glass ionomer for restorative filling increased with time. The flex-

ural strength of experimental glass ionomer was comparable to those of commercially available glass ionomer cements. This study was supported by Grants-in-Aid for Scientific Research (C) No. 26462954.

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### Mechanical properties of dentin restored with glass ionomer cements



P.H. Dos Santos<sup>1,\*</sup>, A.P. Guedes<sup>1</sup>, M.D. Moda<sup>1</sup>, T.Y. Suzuki<sup>1</sup>, A.G. Godas<sup>1</sup>, S. Pavan<sup>2</sup>, R.H. Sundfeld<sup>1</sup>, A.L. Briso<sup>1</sup>

<sup>1</sup> São Paulo State University, UNESP, Brazil

<sup>2</sup> FAI University, Brazil

**Purpose:** To evaluate the effect of erosive pH cycling using solutions on the mechanical properties of dentin restored with glass-ionomer materials.

**Methods and materials:** Eighteen bovine dentine slabs (6.0 mm  $\times$  6.0 mm) were restored with Riva Self Cure conventional glass ionomer cement and Riva Light Cure light-cured glass ionomer cement ( $n = 9$ ). The Martens hardness and Eit values were measured before and after immersion in deionized water, citric acid and hydrochloric acid at distances of 10  $\mu\text{m}$ , 30  $\mu\text{m}$ , 50  $\mu\text{m}$ , and 70  $\mu\text{m}$  from the interface.

**Results:** After cycling, the Martens hardness of dentin decreased for all the materials. The distance of 10  $\mu\text{m}$  exhibited lower values compared with the other distances, for both materials, only before erosive pH cycling. The Eit values of the dentin restored with both glass ionomer cements (Riva Self Cure and Riva Light Cure) decreased with increasing distance from the bonding interface; however, after 30  $\mu\text{m}$ , this difference was no longer significant.

**Conclusion:** The fluoride present in the materials interfered without completely preventing dentin demineralization adjacent to restorations.

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### Evaluation of a fluoro-alumino-calcium silicate-based desensitizer using an ultrasonic device



R. Kawamoto\*, H. Endo, T. Takamizawa, M. Miyazaki

Nihon University School of Dentistry, Japan

**Purpose:** The purpose of this study was to evaluate the effect of a fluoro-alumino-calcium silicate-based desensitizer on demineralization of bovine dentin, by measuring changes in ultrasonic velocity to determine the change in mineral content. The ultrasound velocity is a characteristic of a material through which sound is passing, and its importance depends upon its relationship with the elastic modulus.

**Methods and materials:** Desensitizer used in this study was Nanoseal (Nippon Shika Yakuhin, Yamaguchi, Japan). Bovine dentin specimens with and without application of

**Table 1**

Storage condition	Cement A	Cement B
1 h	8.8 $\pm$ 0.9	7.2 $\pm$ 1.1
24 h	13.4 $\pm$ 1.4	13.5 $\pm$ 2.1
1 week	21.3 $\pm$ 2.3	23.5 $\pm$ 4.5
3000 thermal cycles	22.4 $\pm$ 1.7	17.9 $\pm$ 2.4

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### Flexural strength of experimental highly viscous conventional glass ionomers



Y. Hibino\*, Y. Nagasawa, H. Shigeta, J. Omatsu, H. Nakajima

Meikai University School of Dentistry, Sakado, Japan

**Purpose:** The objective of this study was to examine the flexural strength of experimental highly viscous conventional glass ionomers for restorative filling under various storage conditions.

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**Conclusion:** Under the present experimental conditions, the flexural strength of highly viscous conventional glass ionomer for restorative filling increased with time. The flex-

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P.H. Dos Santos<sup>1,\*</sup>, A.P. Guedes<sup>1</sup>, M.D. Moda<sup>1</sup>, T.Y. Suzuki<sup>1</sup>, A.G. Godas<sup>1</sup>, S. Pavan<sup>2</sup>, R.H. Sundfeld<sup>1</sup>, A.L. Briso<sup>1</sup>

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R. Kawamoto\*, H. Endo, T. Takamizawa, M. Miyazaki

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3000 thermal cycles	22.4 $\pm$ 1.7	17.9 $\pm$ 2.4

desensitizer were cut into blocks (4 × 4 × 1-mm), immersed in 0.1 M lactic-acid buffer solution (pH = 4.75, Ca: 0.75 mM, P: 0.45 mM) for 10 min twice daily throughout the test period, and stored in artificial saliva (pH 7.0) between treatments. The transducer was oriented perpendicular to the contact surface of each specimen, in order to obtain the echo signal. The ultrasonic waves propagated from the transducer to the tooth were transmitted through the substrate and were detected by a transmitter set on the opposite side. Six specimens per group were used for each condition, and data were evaluated using one-way ANOVA followed by Tukey HSD test ( $\alpha = 0.05$ ).

**Results:** The ultrasonic velocity of intact bovine dentin in the Control group ranged from 3398 to 3285 m/s, and did not vary significantly with treatment period. The ultrasonic velocity in the demineralization group decreased over time, and was significantly lower than that in the Control group after 7 d (3080–3260 m/s). For one-time application group, slight decrease in ultrasonic velocity was found, but it did not vary significantly with treatment period (3230–3372 m/s). In contrast, an increase in ultrasonic velocity was found in the repeated-application group compared to that of the Control group, but it did not vary significantly with treatment period (3356–3454 m/s).

**Conclusion:** It could be considered that the obliteration of dentinal tubules by repeated application of fluoro-alumino-calcium silicate-based desensitizer prevents demineralization and the occluded dentinal tubules reduce dentinal fluid movement with consequent clinical improvement of dentin hypersensitivity.

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### Clinical evaluation of direct/indirect restorations using the flexible die technique



C.R.G. Torres\*, F. Feitosa, E. Crastechini, M.M. Maia, G.M. Miranda, R.D. Nicolo, A.B. Borges

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**Purpose:** The aim of this study was to evaluate the clinical performance of composite restorations in posterior teeth using direct versus indirect technique with the flexible die.

**Methods and materials:** This study had a randomized split mouth design. Thirty patients were selected according to the inclusion criteria and signed a term of informed consent. Each patient received two medium/large size class II restorations, one made with a direct and the other with an indirect technique. The preparation for direct restoration was restricted to the removal of carious tissue with a round diamond bur, while for indirect, the surrounding walls of the preparations had a minimum divergence of 6°. The direct restorations were performed using a sectional matrix and incremental oblique technique. For indirect, a hemi-arch partial impression with alginate was performed. The model silicone (Die Silicone, Voco) was applied inside the impression, obtaining a flexible die. Composite restorations were constructed over the model using 2 mm increments, each one light cured for 20 s.

After that an additional light/temperature curing cycle was performed for 6 min at 100 °C (Translux CL LightBox, Kulzer). The inner surface was sandblasted with aluminum oxide. The nanohybrid composite GrandioSO (Voco) was utilized for both restorations. All preparations received the selective acid etching of enamel and application of the universal self-etching adhesive Futurabond U (Voco), according to manufacturer's instructions. For luting, the dual-cure cement Bifix QM (Voco) was applied. The restorations were evaluated using the FDI criteria described by Hickel. The evaluations were conducted by two calibrated examiners at baseline, six months, one year and two years after the restorative procedures. The data were analyzed using Kruskal–Wallis test.

**Results:** All patients attended the 6 months, 27 the one year and 25 the two years recall. After two years, both techniques showed 100% acceptable scores for esthetic properties. In relation to functional properties, 100% of acceptable scores were observed for direct restorations and 96% for indirect. In relation to biological scores, 96% for direct were acceptable and 100% for indirect. Non-significant differences were observed between the two techniques ( $p > 0.05$ ) for all properties. After 24 months, one direct restoration was replaced after endodontic treatment necessary to treat a pulpal necrosis. One indirect restoration was repaired because of a fracture at the marginal ridge.

**Conclusion:** We can conclude that after 24-month of intra-oral service, the restorations made with both direct and indirect techniques presented good clinical performance for all parameters analyzed.

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### Bond strengths of dual-cure adhesive resin cements to dentin



M. Nakajima<sup>1,\*</sup>, M. Takahashi<sup>1</sup>, M. Teerapong<sup>1</sup>, S. Thitthaweerat<sup>2</sup>, N. Seki<sup>3</sup>, K. Hosaka<sup>1</sup>, J. Tagami<sup>1</sup>

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**Purpose:** This study was designed to investigate regional bond strengths between dentin and indirect resin composite block bonded with four kinds of dual-cure resin cement.

**Methods and materials:** Twelve extracted unerupted human third molars were used in this study. After removing the occlusal enamel using a low-speed diamond saw, the dentin surface was ground using 600-grit SiC papers to expose a flat dentin surface. Dentin surfaces were applied with dual-cure adhesive resin cements; EsteGem (Tokuyama Dental), RelyX Ultimate (3M ESPE), Panavia F2.0 (Kuraray Noritake Dental) or RelyX Veneer Cement (3M ESPE) according to the manufacturer's instruction, and then restored with an indirect resin block of 2 mm thick (PERLESTE: shade DA2, Tokuyama Dental) and light-cured for 20 s from each five directions

desensitizer were cut into blocks ( $4 \times 4 \times 1$ -mm), immersed in 0.1 M lactic-acid buffer solution (pH = 4.75, Ca: 0.75 mM, P: 0.45 mM) for 10 min twice daily throughout the test period, and stored in artificial saliva (pH 7.0) between treatments. The transducer was oriented perpendicular to the contact surface of each specimen, in order to obtain the echo signal. The ultrasonic waves propagated from the transducer to the tooth were transmitted through the substrate and were detected by a transmitter set on the opposite side. Six specimens per group were used for each condition, and data were evaluated using one-way ANOVA followed by Tukey HSD test ( $\alpha = 0.05$ ).

**Results:** The ultrasonic velocity of intact bovine dentin in the Control group ranged from 3398 to 3285 m/s, and did not vary significantly with treatment period. The ultrasonic velocity in the demineralization group decreased over time, and was significantly lower than that in the Control group after 7 d (3080–3260 m/s). For one-time application group, slight decrease in ultrasonic velocity was found, but it did not vary significantly with treatment period (3230–3372 m/s). In contrast, an increase in ultrasonic velocity was found in the repeated-application group compared to that of the Control group, but it did not vary significantly with treatment period (3356–3454 m/s).

**Conclusion:** It could be considered that the obliteration of dentinal tubules by repeated application of fluoro-alumino-calcium silicate-based desensitizer prevents demineralization and the occluded dentinal tubules reduce dentinal fluid movement with consequent clinical improvement of dentin hypersensitivity.

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After that an additional light/temperature curing cycle was performed for 6 min at  $100^\circ\text{C}$  (Translux CL LightBox, Kulzer). The inner surface was sandblasted with aluminum oxide. The nanohybrid composite GrandioSO (Voco) was utilized for both restorations. All preparations received the selective acid etching of enamel and application of the universal self-etching adhesive Futurabond U (Voco), according to manufacturer's instructions. For luting, the dual-cure cement Bifix QM (Voco) was applied. The restorations were evaluated using the FDI criteria described by Hickel. The evaluations were conducted by two calibrated examiners at baseline, six months, one year and two years after the restorative procedures. The data were analyzed using Kruskal–Wallis test.

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**Conclusion:** We can conclude that after 24-month of intra-oral service, the restorations made with both direct and indirect techniques presented good clinical performance for all parameters analyzed.

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### Bond strengths of dual-cure adhesive resin cements to dentin



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desensitizer were cut into blocks (4 × 4 × 1-mm), immersed in 0.1 M lactic-acid buffer solution (pH = 4.75, Ca: 0.75 mM, P: 0.45 mM) for 10 min twice daily throughout the test period, and stored in artificial saliva (pH 7.0) between treatments. The transducer was oriented perpendicular to the contact surface of each specimen, in order to obtain the echo signal. The ultrasonic waves propagated from the transducer to the tooth were transmitted through the substrate and were detected by a transmitter set on the opposite side. Six specimens per group were used for each condition, and data were evaluated using one-way ANOVA followed by Tukey HSD test ( $\alpha = 0.05$ ).

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**Table 1**

Results	EsteCem	RelyX Ultimate	Panavia F	RelyX Veneer Cement
Center	34.6(8.0) <sup>a</sup> NS	42.3(6.5) <sup>a</sup> $p < 0.05$	19.3(2.8) <sup>b</sup> NS	25.0(7.3) <sup>b</sup> $p < 0.05$
Peripheral	34.6(8.9) <sup>a</sup>	49.3(9.3) <sup>b</sup>	22.6(5.4) <sup>c</sup>	30.3(7.3) <sup>a</sup>

(peripheral sides and overlaid-resin block side) using a light-curing units; Optilux 501 (830 mW/cm<sup>2</sup>). After water storage for 24 h, the bonded teeth were vertically cut perpendicular to the adhesive surface to obtain 16 beams with 0.7 × 0.7 mm thick, under water cooling. The beams were subjected to micro-tensile bond test at cross-head speed of 1 mm/min. The bond strength data were divided into center and peripheral portions under resin block and analyzed using two-way ANOVA and Bonferroni test ( $p < 0.05$ ).

**Results:** The results show [Table 1](#), (mean (SD), MPa). Different letters indicate statistically significant differences. NS = no significant difference ( $p > 0.05$ ).

**Conclusion:** Dual-cure resin cements had higher bond strengths to peripheral portion under indirect resin composite than those to center portion, except for EsteCem. These results would indicate that their bonding performances were influenced by light irradiation energy through indirect resin composite.

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### 3D assessment of dental caries using swept-source optical coherence tomography



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<sup>2</sup>National Center for Geriatrics and Gerontology, Japan

**Purpose:** Optical coherence tomography (OCT) is a non-invasive technique providing cross-sectional images of a tooth structure. OCT helps visualize differences in tissue optical properties, which includes the effects of both optical absorption and scattering. Swept-source OCT (SS-OCT) can construct images by the ultra-high-speed scanning of the time-encoded wavelength of a near-infrared laser. SS-OCT allows for the non-invasive construction of 3D images of biological substrates in a short period of time. Dental caries is an infectious microbial disease that results in localized dissolution and destruction of calcified dental tissues. Despite considerable decline in the incidence of caries, the disease is yet to be completely eradicated, particularly in children and young adults. The aim of this study was to compare the diagnostic accuracy of SS-OCT and dental radiographs in detecting and estimating the depth of caries in posterior teeth *in vivo*.

**Methods and materials:** Unrestored surfaces of premolars and molars with a possibility of caries were selected from 10 patients for this study. This study was approved by the Ethics Committee of the Tokyo Medical and Dental University. After taking dental radiographs, 3D images were constructed

from the surfaces using SS-OCT. The following 5-point rank scale was employed to record the level of caries progression on the basis of bitewing radiographs or SS-OCT images: 0: Sound tooth surface. 1: Superficial enamel demineralization without cavitation. 2: Localized enamel caries. Breakdown due to caries was limited to the enamel thickness. 3: Superficial dentin caries. The lesion depth was beyond DEJ but limited to the outer half of the dentin thickness. 4: Deep dentin caries. The lesion depth was beyond the DEJ and penetrated into the inner half of dentin. The results were compared with clinical observations obtained after the treatment.

**Results:** 3D analysis using SS-OCT could detect the presence of caries that were synthesized based on the backscattered signal. In the case of caries with extensive loss of enamel and dentin creating a hollow space, the upper borders of the hollow space strongly scatter and attenuate light, which is clearly imaged in SS-OCT.

**Conclusion:** SS-OCT appears to be a more reliable and accurate method than dental radiographs for the detection and estimation of the depth of caries in the clinical environment.

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### Protection against discoloration by two OCT anti-erosion products



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University of Geneva, Geneva, Switzerland

**Purpose:** The aim of this *in vitro* study was to determine the protection potential against discoloration of two OCT anti-erosion products on enamel and dentin in comparison to a standard toothpaste and water by means of a spectrophotometer.

**Methods and materials:** A total of 96 samples made out of enamel and 48 of dentin were alternatively immersed in red wine, tee, coffee or water after having been treated by Curodont Protect, Elmex Red tooth paste, Elmex Protection Erosion tooth paste or water (negative control).

**Results:** When enamel samples were measured over a black background,  $\Delta E$  values (T0-T1) varied from 2.2 (0.7) Elmex Red/water to 53.9 (7.6) Elmex Red/red wine. When dentin samples were measured over a black background,  $\Delta E$  values (T0-T1) varied from 5.4 (0.9) Elmex Protection Erosion/water to 61.6 (3.7) Elmex Red/red wine. Statistical analysis was done by mean of repeated measures Anova followed by Tuckey post hoc tests which revealed statistical significant differences ( $p < 0.05$ ).

**Conclusion:** Within the limitations of this study, the application of OCT anti-erosion products did not reduce discoloration, both in enamel and dentin, compared to water or standard toothpaste.

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**Table 1**

Results	EsteCem	RelyX Ultimate	Panavia F	RelyX Veneer Cement
Center	34.6(8.0) <sup>a</sup> NS	42.3(6.5) <sup>a</sup> $p < 0.05$	19.3(2.8) <sup>b</sup> NS	25.0(7.3) <sup>b</sup> $p < 0.05$
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(peripheral sides and overlaid-resin block side) using a light-curing units; Optilux 501 (830 mW/cm<sup>2</sup>). After water storage for 24 h, the bonded teeth were vertically cut perpendicular to the adhesive surface to obtain 16 beams with 0.7 × 0.7 mm thick, under water cooling. The beams were subjected to micro-tensile bond test at cross-head speed of 1 mm/min. The bond strength data were divided into center and peripheral portions under resin block and analyzed using two-way ANOVA and Bonferroni test ( $p < 0.05$ ).

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**Purpose:** Optical coherence tomography (OCT) is a non-invasive technique providing cross-sectional images of a tooth structure. OCT helps visualize differences in tissue optical properties, which includes the effects of both optical absorption and scattering. Swept-source OCT (SS-OCT) can construct images by the ultra-high-speed scanning of the time-encoded wavelength of a near-infrared laser. SS-OCT allows for the non-invasive construction of 3D images of biological substrates in a short period of time. Dental caries is an infectious microbial disease that results in localized dissolution and destruction of calcified dental tissues. Despite considerable decline in the incidence of caries, the disease is yet to be completely eradicated, particularly in children and young adults. The aim of this study was to compare the diagnostic accuracy of SS-OCT and dental radiographs in detecting and estimating the depth of caries in posterior teeth *in vivo*.

**Methods and materials:** Unrestored surfaces of premolars and molars with a possibility of caries were selected from 10 patients for this study. This study was approved by the Ethics Committee of the Tokyo Medical and Dental University. After taking dental radiographs, 3D images were constructed

from the surfaces using SS-OCT. The following 5-point rank scale was employed to record the level of caries progression on the basis of bitewing radiographs or SS-OCT images: 0: Sound tooth surface. 1: Superficial enamel demineralization without cavitation. 2: Localized enamel caries. Breakdown due to caries was limited to the enamel thickness. 3: Superficial dentin caries. The lesion depth was beyond DEJ but limited to the outer half of the dentin thickness. 4: Deep dentin caries. The lesion depth was beyond the DEJ and penetrated into the inner half of dentin. The results were compared with clinical observations obtained after the treatment.

**Results:** 3D analysis using SS-OCT could detect the presence of caries that were synthesized based on the backscattered signal. In the case of caries with extensive loss of enamel and dentin creating a hollow space, the upper borders of the hollow space strongly scatter and attenuate light, which is clearly imaged in SS-OCT.

**Conclusion:** SS-OCT appears to be a more reliable and accurate method than dental radiographs for the detection and estimation of the depth of caries in the clinical environment.

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### Protection against discoloration by two OCT anti-erosion products



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University of Geneva, Geneva, Switzerland

**Purpose:** The aim of this *in vitro* study was to determine the protection potential against discoloration of two OCT anti-erosion products on enamel and dentin in comparison to a standard toothpaste and water by means of a spectrophotometer.

**Methods and materials:** A total of 96 samples made out of enamel and 48 of dentin were alternatively immersed in red wine, tee, coffee or water after having been treated by Curodont Protect, Elmex Red tooth paste, Elmex Protection Erosion tooth paste or water (negative control).

**Results:** When enamel samples were measured over a black background,  $\Delta E$  values (T0-T1) varied from 2.2 (0.7) Elmex Red/water to 53.9 (7.6) Elmex Red/red wine. When dentin samples were measured over a black background,  $\Delta E$  values (T0-T1) varied from 5.4 (0.9) Elmex Protection Erosion/water to 61.6 (3.7) Elmex Red/red wine. Statistical analysis was done by mean of repeated measures Anova followed by Tuckey post hoc tests which revealed statistical significant differences ( $p < 0.05$ ).

**Conclusion:** Within the limitations of this study, the application of OCT anti-erosion products did not reduce discoloration, both in enamel and dentin, compared to water or standard toothpaste.

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### Influence of irradiation on bonding characteristics of self-adhesive resin cements



H. Kurokawa\*, M. Takimoto, K. Shiratsuchi, H. Takenaka, M. Miyazaki

Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

**Purpose:** Self-adhesive resin cements (SARCs) have been marketed to simplify clinical procedures and overcome the technique sensitivity of multiple-step systems. However, their dentin bonding performance in different light irradiation of the cement is not known. The purpose of this study was to monitor the chronological change of dentin bond strengths of SARCs.

**Methods and materials:** Three SARCs; Clearfil SA Cement Automix (SA; Kuraray Noritake Dental), RelyX Unicem 2 Automix (UC; 3M ESPE) and BeautiCem SA (BC; Shofu) were used. Bovine dentin was wet ground with #600 SiC paper. Resin column (4 mm in diameter, 2 mm in height) were cemented and light-irradiated (600 mW/cm<sup>2</sup>), or not irradiated (chemical-cured). The shear bond strengths of 10 specimens per group were measured at a crosshead speed of 1.0 mm/min after 10 min, 1, 6, 12, and 24 h storage in a chamber at 37 ± 1 °C, 90 ± 5 RH %. Statistical analysis was done to test for presence of a significant difference between the mean bond strength at each of test time at a significance level of 0.05. The time at which there was a significant increase in bond strength was identified as “time of increase in bond strength (TIBS)”.

**Results:** The dentin bond strength increased with prolonged specimen storage time. When the specimens were light-irradiated, higher bond strengths were obtained; compared to those specimens without irradiation. Fracture mode of the UC specimens without irradiation changed from adhesive failure to mixed failure after 24 h storage. When the specimens were light irradiated, cohesive failure in resin was observed for UC, and cohesive failure in the cement was observed for BC.

**Conclusion:** From the results of this study, it was indicated that the chronological change in dentin bond strengths of the SARCs was affected by the light irradiation of the specimen. Light irradiation with a sufficient power density is necessary to achieve optimal dentin bond strength, even in materials with a dual-cured setting reaction.

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### Effect of Titania Addition on the Mechanical Properties of Y-TZP



R. Miranda<sup>1</sup>, V. Ussui<sup>2</sup>, D. Lazar<sup>2</sup>, J. Marchi<sup>3</sup>, W. Miranda<sup>1</sup>, P. Cesar<sup>1,\*</sup>

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<sup>3</sup> Federal University of the ABC, Brazil

**Purpose:** To evaluate the effect of adding 30 mol% of titania on the microstructure and mechanical properties of one yttria stabilized tetragonal zirconia polycrystal (Y-TZP).

**Methods and materials:** Y-TZP and Y-TZP/TiO<sub>2</sub> composite were synthesized from precursors (zirconium oxychloride, titanium chloride and yttrium chloride) using a co-precipitation route. The composition of the samples changed depending on the titania content in mol%: zero (control) and 30 (T30). The powders produced were pressed (50 MPa) into pellets (P) and sintered at 1400 °C for 2 h. After polishing with diamond solution of 45 μm, the P (n = 30 for each group) were approximately 12 mm in diameter and 1.0 mm thick. Microstructural analysis was performed using X-ray diffraction (XRD), scanning electron microscopy (SEM) and for the Weibull analysis, the pellets were submitted to biaxial bending with a stress rate of 1 MPa/s in artificial saliva at 37 °C. The effect of titania addition on the mean strength was assessed by Student's t-test (alpha = 0.05), and the Weibull modulus (m) was estimated by the maximum likelihood method.

**Results:** SEM analysis showed that the control group was constituted of zirconia grains with sub-micrometric size. The T30 group showed zirconia grains that were larger than those observed for the control and some of the grains looked like they were projected out of the material surface. XRD analysis identified for the two groups the presence of peaks corresponding to both tetragonal and monoclinic zirconia. For the T30 group, peaks corresponding to zirconium titanate were also identified. The mean strength (MPa) and Weibull moduli are shown in the Table 1. It is possible to note that adding titania to the Y-TZP significantly decreased the mean strength of the material. However, the Weibull modulus was significantly higher for the group containing 30 mol% of titania.

**Table 1**

Parameter	Titania content (mol%)	
	0 (control)	30
Mean strength (MPa)	815 ± 145 <sup>a</sup>	336 ± 38 <sup>b</sup>
Weibull modulus (m)	6.4 (4.7 – 8.5) <sup>b</sup>	11.7 (8.6 – 15.8) <sup>a</sup>



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### Wear resistance of CAD/CAM composite resin blocks



S. Lauvahutanon<sup>1,2</sup>, H. Takahashi<sup>1,\*</sup>, M. Oki<sup>1</sup>, M. Arksornnukit<sup>2</sup>, M. Kanehira<sup>3</sup>, W.J. Finger<sup>3</sup>

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**Conclusion:** The block materials are considered suitable for fabrication of single full crown restorations on premolar teeth.

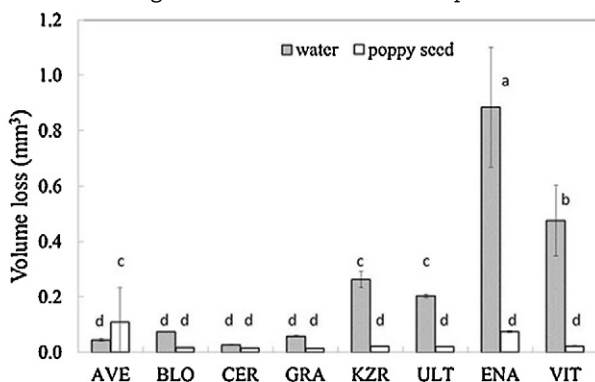


Figure Mean and standard deviation of volume loss in water and in poppy seed. Same lower case letters denote groups that are not significantly different ( $p > 0.05$ ).

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### Sealer wettability on root canal surface after non-thermal plasma therapy



F.F. Lins<sup>1,\*</sup>, M. Prado<sup>2</sup>, R.N. Roizenblit<sup>2</sup>, L.V. Pacheco<sup>2</sup>, C.A.M. Barbosa<sup>2</sup>, R.A. Simão<sup>2</sup>

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**Methods and materials:** Thirty bovine incisors were used. The crown was removed, the root split, and the segments planed. Sixty segments of the cervical third were used. The samples were divided into two groups: Control: Immersed in 6% NaOCl, washed with distilled water, dried, and then immersed in EDTA, washed and dried; and Argon group: after treatment described in the control group, non-thermal argon plasma was applied, for 30 s. The Ramé-hart goniometer was used to measure the contact angle between the surfaces and the sealers AH Plus, Pulp Canal Sealer EWT and Sealapex. For each group, ten samples were evaluated. The sealer dynamic wettability was accompanied and computed during 1 min (sixty measurements with 1 s interval between each measurement). Sealer dynamic wettability (SDW - %) was evaluated using the formula:  $[(\text{initial angle} - \text{final angle}) / \text{initial angle}] \times 100$ . Data were analyzed statistically by Kruskal-Wallis and Mann-Whitney tests ( $p < 0.05$ ).

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### The elastic-plastic nature of fracture in dental resin composites



R. Belli<sup>1,\*</sup>, R. Wolf<sup>2</sup>, A. Petschelt<sup>1</sup>, A. Boccaccini<sup>2</sup>, U. Lohbauer<sup>1</sup>

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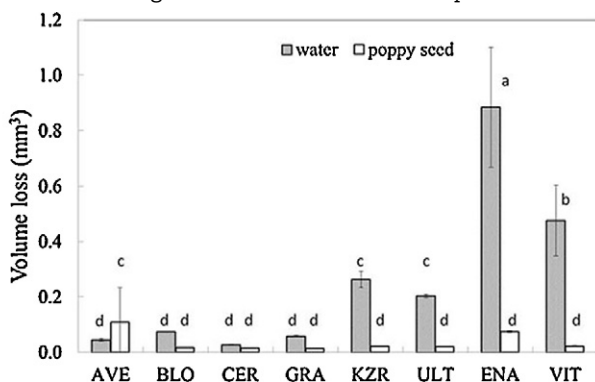


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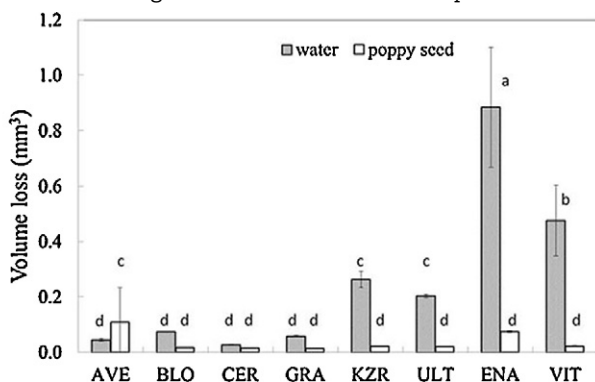


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**Methods and materials:** Six composite resins (AVE, BLO, CER, GRA, KZR, and ULT), one hybrid ceramic (ENA) and one feldspar ceramic (VIT) block materials for CAD/CAM were examined. Six specimens each were tested in a ball-on-disc wear device fitted with a zirconia ball (50 N load, 1.2 Hz) in water for two-body and in poppy seed slurry for three-body wear evaluation. Volume loss after 10k cycles was quantified using a digital CCD microscope. Statistical analysis: ANOVA and Tukey's multiple comparisons ( $\alpha = 0.05$ ).

**Results:** Two-body wear for composite resin blocks was small, hybrid ceramic and ceramic blocks showed larger volume loss. Three-body wear was very low for all materials. All CAD/CAM composite resin block materials investigated displayed low wear compared to previous data reported for direct posterior composites carrying out the same wear test.

**Conclusion:** The block materials are considered suitable for fabrication of single full crown restorations on premolar teeth.

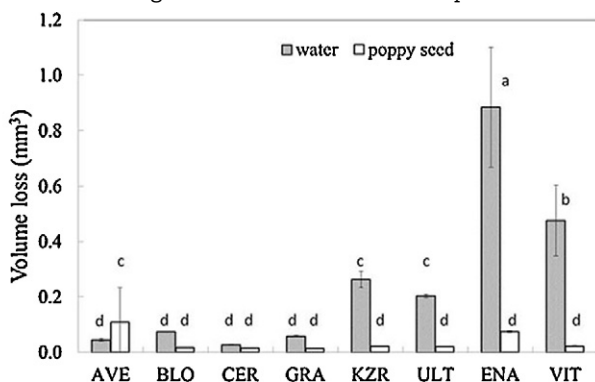


Figure Mean and standard deviation of volume loss in water and in poppy seed. Same lower case letters denote groups that are not significantly different ( $p > 0.05$ ).

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### Sealer wettability on root canal surface after non-thermal plasma therapy



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**Purpose:** Plasma treatment is an effective and clean technology, since the bulk properties of the materials may remain un-altered or well-maintained after the treatment. Argon plasma has been proposed to improve the adhesion process of resin-based materials to dentin. The aim of the present study was to evaluate the sealer wettability on root canal surface after non-thermal plasma, using Argon.

**Methods and materials:** Thirty bovine incisors were used. The crown was removed, the root split, and the segments planed. Sixty segments of the cervical third were used. The samples were divided into two groups: Control: Immersed in 6% NaOCl, washed with distilled water, dried, and then immersed in EDTA, washed and dried; and Argon group: after treatment described in the control group, non-thermal argon plasma was applied, for 30 s. The Ramé-hart goniometer was used to measure the contact angle between the surfaces and the sealers AH Plus, Pulp Canal Sealer EWT and Sealapex. For each group, ten samples were evaluated. The sealer dynamic wettability was accompanied and computed during 1 min (sixty measurements with 1 s interval between each measurement). Sealer dynamic wettability (SDW - %) was evaluated using the formula:  $[(\text{initial angle} - \text{final angle}) / \text{initial angle}] \times 100$ . Data were analyzed statistically by Kruskal-Wallis and Mann-Whitney tests ( $p < 0.05$ ).

**Results:** Argon plasma improved statistically the SDW of AH Plus, however had no effect in the Pulp Canal Sealer EWT and Sealapex wettability.

**Conclusion:** Non-thermal Plasma improved the dynamic wettability of the resin-based sealer AH Plus. Supported by Capes and CNPq.

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### The elastic-plastic nature of fracture in dental resin composites



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**Purpose:** The mechanical behavior of dental composites is usually studied within the realm of linear-elastic behavior, where fast fracture is induced, e.g., for the measurement of Strength and Fracture Toughness. However, loading in the oral environment is rarely held statically for long periods of

time, such as under clenching and bruxism. Therein plays the viscoelastic nature of dental composites an important role, which may contribute with a significant plastic component to the fracture event. Here we aim to measure the Fracture Toughness of nano-hybrid dental composites using the conventional Linear-Elastic Fracture Mechanics (LEFM, Kc) and the Elastic-Plastic Fracture Mechanics (EPFM, KJc) approaches.

**Methods and materials:** Compact Tension specimens ( $n=5$ ) were produced according to the ASTM 1820-13 for three composites: GrandioSO, GrandioSO Flow and GrandioSO Heavy Flow (all Voco, Germany). Specimens were stored dry or in water for 2 months and tested at 1 mm/min or 0.01 mm/min. Micro strain-gauges were bonded to one side of the specimens to record the uniaxial deformation with high precision. The single-edge-notched-beam (SENB) method under 3-point bending was performed following the ISO 13856 for comparison.

**Results:** No plastic deformation was observed upon fast-fracture (1 mm/min), yielding Kc values of 1.54 (0.06), 0.87 (0.05) and 0.96 (0.03) MPam0.5 for GrandioSO, GrandioSO Flow and GrandioSO Heavy Flow, respectively. Under low strain rates (0.01 mm/min) KJc values for the same composites were 2.55 (0.09), 2.17 (0.1) and 2.13 (0.3) MPam0.5 due to a significant plastic contribution to the fracture energy (Jpl). Higher Jpl were observed for the Flowable composites. After 2 months in water KJc values increased significantly.

**Conclusion:** At low strain rates the fracture behavior dental resin composites contains a significant plastic component and should be assessed using the EPFM approach. Low-viscosity monomers and water storage increase the plastic deformation during fracture, thereby toughening resin composites.

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### Effect of water storage on stress distribution in root-filled premolars



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**Purpose:** Studies indicated that degradation occurred at the dentin-adhesive interface after long-term water storage. The aim of this finite elemental stress analysis (FEA) study was to determine the effect of water storage on stress-distributions in root-filled premolars restored with composite resin using either by self-etch or total-etch adhesive system.

**Methods and materials:** Four premolar tooth FEA models were created. The models were included root filling, MOD cavity and composite-resin restorations. The cavities were assumed as treated by a self-etch or a total-etch adhesive system. The teeth were assumed as stored in water for 18 months. Change in the elastic properties of the dentin-adhesive interfaces after 24 h and after 18 months was obtained from the literature. These properties were applied to the FEA models. A 300 N load was applied from the functional cusps of the models. For FEA analysis, the SolidWorks/Cosmosworks structural-analysis program was used. Results were presented considering von Mises criteria.

**Results:** Stresses at the cervical region were increased by the time at the load application side of the main tooth models (self-etch: 84.11 MPa to 87.51 MPa; total-etch: 100.24 MPa to 120.8 MPa). When the adhesive interfaces (hybrid layer, adhesive layer) and dentin were evaluated separately, the stresses near the root canal orifices were increased with time in both models however this change was more noticeable in the total-etch group. Stresses at the cavity corners were decreased in the total-etch model (in adhesive layer) while self-etch model showed the opposite (in hybrid layer).

**Conclusion:** Degradation of the adhesive interfaces has an effect on stresses. This effect changes according to the adhesive system used (total-etch or self-etch).

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### Effect of chemical solvents on flexural properties of bulk-fill composites



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**Purpose:** The aim of this study was to measure and compare flexural properties (strength,  $s$ ; modulus, Eflexural; Weibull modulus,  $m$ ; and probability of failure) of bulk-fill resin composites after immersion in different chemical solvents.

**Methods and materials:** Seven bulk-fill resin composites (SonicFill™ [ScF], Kerr; Tetric EvoCeram® Bulk Fill [TEC], Ivoclar Vivadent; X-tra base® [XB], VOCO; everX Posterior™ [EXP], GC; SureFil® SDR™ flow [SDRF], Dentsply DeTrey; Filtek™ Bulk Fill [FBF], 3M ESPE; Venus® Bulk Fill [VBF], Heraeus Kulzer) were studied. Four groups ( $n=20$ ) of bar-shaped specimens (25 mm × 2 mm × 2 mm) were prepared after 4 × light-curing exposures for 20 s, each side. Then, they were stored in the following solvents: (Methyl Ethyl Ketone [MEK], Heptane [H], 99% Ethanol [E]; Sigma-Aldrich, and Distilled water) for two weeks. The flexural properties: ( $s$ , Eflexural,  $m$ ) were determined in a three-point bending test using a universal testing machine, according to ISO/DIN 4049:1998, at crosshead speed 1 mm/min and 23 °C. The data were analyzed by two-way ANOVA and Tukey's HSD post hoc test.

**Results:** Two-way ANOVA showed a high significant difference of the flexural properties among the bulk-fill composites and the solvents as well as the interaction between them ( $p=0.000$ ). The highest flexural strength ( $s$ ) and modulus (Eflexural) were observed in ScF when it was stored in water, 165.77 (4.64) MPa and 10.3 (0.62) GPa, while the lowest values were noted in FBF and VBF when they were stored in Heptane and MEK, 73.65 (0.54) MPa; 3.45 (0.59) GPa and 74.11 (6.2) MPa; 3.06 (0.55) GPa, respectively. Weibull modulus ranged between highest value, 37.7 and 31.6 for ScF and TEC, in water; and lowest values, 12.1 for VBF and 12.4 for FBF, in water and MEK, respectively. The flexural strength and modulus values varied among the different solvents depending on the composition of the bulk-fill composites. Their flexural properties decrease in the solvents following the order: ScF > TEC > XB > EXP > SDRF > FBF > VBF.

time, such as under clenching and bruxism. Therein plays the viscoelastic nature of dental composites an important role, which may contribute with a significant plastic component to the fracture event. Here we aim to measure the Fracture Toughness of nano-hybrid dental composites using the conventional Linear-Elastic Fracture Mechanics (LEFM, Kc) and the Elastic-Plastic Fracture Mechanics (EPPM, KJc) approaches.

**Methods and materials:** Compact Tension specimens ( $n=5$ ) were produced according to the ASTM 1820-13 for three composites: GrandioSO, GrandioSO Flow and GrandioSO Heavy Flow (all Voco, Germany). Specimens were stored dry or in water for 2 months and tested at 1 mm/min or 0.01 mm/min. Micro strain-gauges were bonded to one side of the specimens to record the uniaxial deformation with high precision. The single-edge-notched-beam (SENB) method under 3-point bending was performed following the ISO 13856 for comparison.

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**Conclusion:** At low strain rates the fracture behavior dental resin composites contains a significant plastic component and should be assessed using the EPPM approach. Low-viscosity monomers and water storage increase the plastic deformation during fracture, thereby toughening resin composites.

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### Effect of water storage on stress distribution in root-filled premolars



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<sup>2</sup> Yuzuncuyil University, Van, Turkey

**Purpose:** Studies indicated that degradation occurred at the dentin-adhesive interface after long-term water storage. The aim of this finite elemental stress analysis (FEA) study was to determine the effect of water storage on stress-distributions in root-filled premolars restored with composite resin using either by self-etch or total-etch adhesive system.

**Methods and materials:** Four premolar tooth FEA models were created. The models were included root filling, MOD cavity and composite-resin restorations. The cavities were assumed as treated by a self-etch or a total-etch adhesive system. The teeth were assumed as stored in water for 18 months. Change in the elastic properties of the dentin-adhesive interfaces after 24 h and after 18 months was obtained from the literature. These properties were applied to the FEA models. A 300 N load was applied from the functional cusps of the models. For FEA analysis, the SolidWorks/Cosmosworks structural-analysis program was used. Results were presented considering von Mises criteria.

**Results:** Stresses at the cervical region were increased by the time at the load application side of the main tooth models (self-etch: 84.11 MPa to 87.51 MPa; total-etch: 100.24 MPa to 120.8 MPa). When the adhesive interfaces (hybrid layer, adhesive layer) and dentin were evaluated separately, the stresses near the root canal orifices were increased with time in both models however this change was more noticeable in the total-etch group. Stresses at the cavity corners were decreased in the total-etch model (in adhesive layer) while self-etch model showed the opposite (in hybrid layer).

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### Effect of chemical solvents on flexural properties of bulk-fill composites



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**Purpose:** The aim of this study was to measure and compare flexural properties (strength,  $s$ ; modulus, Eflexural; Weibull modulus,  $m$ ; and probability of failure) of bulk-fill resin composites after immersion in different chemical solvents.

**Methods and materials:** Seven bulk-fill resin composites (SonicFill™ [ScF], Kerr; Tetric EvoCeram® Bulk Fill [TEC], Ivoclar Vivadent; X-tra base® [XB], VOCO; everX Posterior™ [EXP], GC; SureFil® SDR™ flow [SDRF], Dentsply DeTrey; Filtek™ Bulk Fill [FBF], 3M ESPE; Venus® Bulk Fill [VBF], Heraeus Kulzer) were studied. Four groups ( $n=20$ ) of bar-shaped specimens (25 mm × 2 mm × 2 mm) were prepared after 4 × light-curing exposures for 20 s, each side. Then, they were stored in the following solvents: (Methyl Ethyl Ketone [MEK], Heptane [H], 99% Ethanol [E]; Sigma-Aldrich, and Distilled water) for two weeks. The flexural properties: ( $s$ , Eflexural,  $m$ ) were determined in a three-point bending test using a universal testing machine, according to ISO/DIN 4049:1998, at crosshead speed 1 mm/min and 23 °C. The data were analyzed by two-way ANOVA and Tukey's HSD post hoc test.

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### Effect of water storage on stress distribution in root-filled premolars



S. Belli<sup>1,\*</sup>, O. Eraslan<sup>1</sup>, G. Eskitascioglu<sup>2</sup>

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### Effect of chemical solvents on flexural properties of bulk-fill composites



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**Purpose:** The aim of this study was to measure and compare flexural properties (strength,  $s$ ; modulus, Eflexural; Weibull modulus,  $m$ ; and probability of failure) of bulk-fill resin composites after immersion in different chemical solvents.

**Methods and materials:** Seven bulk-fill resin composites (SonicFill™ [ScF], Kerr; Tetric EvoCeram® Bulk Fill [TEC], Ivoclar Vivadent; X-tra base® [XB], VOCO; everX Posterior™ [EXP], GC; SureFil® SDR™ flow [SDRF], Dentsply DeTrey; Filtek™ Bulk Fill [FBF], 3M ESPE; Venus® Bulk Fill [VBF], Heraeus Kulzer) were studied. Four groups ( $n=20$ ) of bar-shaped specimens (25 mm × 2 mm × 2 mm) were prepared after 4 × light-curing exposures for 20 s, each side. Then, they were stored in the following solvents: (Methyl Ethyl Ketone [MEK], Heptane [H], 99% Ethanol [E]; Sigma–Aldrich, and Distilled water) for two weeks. The flexural properties: ( $s$ , Eflexural,  $m$ ) were determined in a three-point bending test using a universal testing machine, according to ISO/DIN 4049:1998, at crosshead speed 1 mm/min and 23 °C. The data were analyzed by two-way ANOVA and Tukey's HSD post hoc test.

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**Conclusion:** Ketone based solvent has the strongest softening effect on bulk-fill composites that contained lesser filler volume fraction and more resin components, thus minimizing their resistance to fracture. Moreover, the type of the resin has an effect on the flexural properties of these composites when immersed in the corresponding solvents.

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### Effect of surface treatment with commercial silane coupling agents



K. Ohashi\*, K. Miyake, H. Yamaguchi, A. Teranaka, T. Shiiya, K. Tomiyama, Y. Mukai, T. Nihei

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**Purpose:** The purpose of this study was to evaluate the clinical characteristics, tensile bond strength (TBS) and water resistance of eight commercial ceramic primers [seven commercial silane coupling agents (IN, MO, PL, RE, CP, TO and CS) and one alumina zirconia primer (AZ)].

**Methods and materials:** Glass plates were used as the adhered. The surfaces of the glass plates were treated according to the manufacturer's instruction using microbrushes for each group of ceramic primers. The modified glass surface and the stainless steel rod were manually held together to achieve bonding with the resin composite. Samples were then divided into two different immersion groups. One group was stored in distilled water at 37 °C for 7 days, and the other group was subjected to thermal cycling. The contact angles were obtained by dropping the resin monomer onto glass plates treated with methods described above. The TBS of resin composite and the wettability of resin monomer to glass surfaces treated with silane coupling agents was investigated, with 3-methacryloxypropyltrimethoxysilane (MPS, Shinetsu) used as a control. The values obtained from the experiments were analyzed using one-way ANOVA followed by Tukey's multiple comparison test ( $p < 0.05$ ).

**Results:** No significant difference was observed in TBS between MPS and the commercial ceramic primers except for AZ ( $p > 0.05$ ). However, after thermal stress, four products (CP, TO, CS and AZ) exhibited significantly lower TBS when compared with those stored in distilled water ( $p < 0.05$ ). Six products (IN, MO, RE, CP, TO and CS) displayed significantly higher contact angles between the treated glass and the resin monomer than MPS ( $p < 0.05$ ).

**Conclusion:** The results of the present study demonstrate that there is no significant difference in immediate bond strength among commercial silane coupling agents, but there is a significant difference in the water resistance of these bonds, showing characteristic variations depending on the products. It was suggested that the use of catalysts in silane coupling agents should be further evaluated to obtain optimum performance of these products.

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### Micro-shear bond strength of resin cement to glass ceramics



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**Purpose:** The aim of this study was to evaluate of influence of surface treatments on bond strength of resin cement (NX3, Kerr Dental) to lithium disilicate (e.Max CAD, Ivoclar-Vivadent) [LD] and lithium silicate reinforced by zirconia (Suprinity, VITA) [SZ] ceramics.

**Methods and materials:** CAD/CAM blocks were sectioned using a diamond disc under refrigerated water irrigation in a metallographic precision cutter, obtaining 104 ceramic plates. Specimens were manually grounded using a sequence of SiC abrasive papers until grit 2000, crystallized and randomly assigned to following surface treatments ( $n = 10$ ): (A) aluminum oxide sandblasting; (B) 9.5% hydrofluoric acid etching (HF) for 10 s; (C) HF for 20 s and (D) HF for 30 s. After treatments, plates were submitted to ultra-sonic bath for 5 min prior to silane and applications. A resin cement cylinder (3, 40 mm high and 1.80 diameter) was bonded to plate surface and light-cured with polywave LED curing unit. Bond strength was determined by micro-shear bond strength test ( $\mu$ SBS) and data were subjected to two-way ANOVA and Tukey's test (5%). For topography analysis after treatments, plates ( $n = 3$ ) were mounted in metallic stubs and gold sputtered for scanning electron microscopy (SEM) observation at  $\times 1000$  and  $\times 5000$  magnification.

**Results:** LD and SZ showed higher  $\mu$ SBS (MPa) values when subjected HF for 20 s: 30.6 (12.8) and 35.6 (12.2), respectively. The lowest values were observed when plates were sandblasted: 7.0 (3.4) for LD and 6.6 (2.2) for SZ. Different surface treatments resulted in different surface modifications observed in SEM. No significant differences were obtained between CAD/CAM materials.

**Conclusion:** Surface treatment of LD and SZ plates with 9.5% HF for 20 s resulted in higher  $\mu$ SBS, as well as visible surface modifications observed under SEM.

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### Comparing the retention of glass ionomer and resin-based fissure sealants



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**Purpose:** The aim of this study was to evaluate and compare the retention of glass ionomer fissure sealant and resin-based fissure sealant.

**Methods and materials:** Thirty children between the ages of 6–9 years, with all four erupted caries-free permanent first molars were selected. Sealants were applied randomly using split mouth design technique. Sixty permanent first molars on one side of the mouth were sealed with Fuji Triage

**Conclusion:** Ketone based solvent has the strongest softening effect on bulk-fill composites that contained lesser filler volume fraction and more resin components, thus minimizing their resistance to fracture. Moreover, the type of the resin has an effect on the flexural properties of these composites when immersed in the corresponding solvents.

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**Conclusion:** The results of the present study demonstrate that there is no significant difference in immediate bond strength among commercial silane coupling agents, but there is a significant difference in the water resistance of these bonds, showing characteristic variations depending on the products. It was suggested that the use of catalysts in silane coupling agents should be further evaluated to obtain optimum performance of these products.

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### Micro-shear bond strength of resin cement to glass ceramics



F.J.F. Rigolin\*, R.R. Pacheco, C.M. Rizzatti-Barbosa, M. Giannini

State University of Campinas, Piracicaba Dental School, Piracicaba, Brazil

**Purpose:** The aim of this study was to evaluate of influence of surface treatments on bond strength of resin cement (NX3, Kerr Dental) to lithium disilicate (e.Max CAD, Ivoclar-Vivadent) [LD] and lithium silicate reinforced by zirconia (Suprinity, VITA) [SZ] ceramics.

**Methods and materials:** CAD/CAM blocks were sectioned using a diamond disc under refrigerated water irrigation in a metallographic precision cutter, obtaining 104 ceramic plates. Specimens were manually grounded using a sequence of SiC abrasive papers until grit 2000, crystallized and randomly assigned to following surface treatments ( $n = 10$ ): (A) aluminum oxide sandblasting; (B) 9.5% hydrofluoric acid etching (HF) for 10 s; (C) HF for 20 s and (D) HF for 30 s. After treatments, plates were submitted to ultra-sonic bath for 5 min prior to silane and applications. A resin cement cylinder (3, 40 mm high and 1.80 diameter) was bonded to plate surface and light-cured with polywave LED curing unit. Bond strength was determined by micro-shear bond strength test ( $\mu$ SBS) and data were subjected to two-way ANOVA and Tukey's test (5%). For topography analysis after treatments, plates ( $n = 3$ ) were mounted in metallic stubs and gold sputtered for scanning electron microscopy (SEM) observation at  $\times 1000$  and  $\times 5000$  magnification.

**Results:** LD and SZ showed higher  $\mu$ SBS (MPa) values when subjected HF for 20 s: 30.6 (12.8) and 35.6 (12.2), respectively. The lowest values were observed when plates were sandblasted: 7.0 (3.4) for LD and 6.6 (2.2) for SZ. Different surface treatments resulted in different surface modifications observed in SEM. No significant differences were obtained between CAD/CAM materials.

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### Comparing the retention of glass ionomer and resin-based fissure sealants



A. Al-Jobair\*

King Saud University, Riyadh, Saudi Arabia

**Purpose:** The aim of this study was to evaluate and compare the retention of glass ionomer fissure sealant and resin-based fissure sealant.

**Methods and materials:** Thirty children between the ages of 6–9 years, with all four erupted caries-free permanent first molars were selected. Sealants were applied randomly using split mouth design technique. Sixty permanent first molars on one side of the mouth were sealed with Fuji Triage

**Conclusion:** Ketone based solvent has the strongest softening effect on bulk-fill composites that contained lesser filler volume fraction and more resin components, thus minimizing their resistance to fracture. Moreover, the type of the resin has an effect on the flexural properties of these composites when immersed in the corresponding solvents.

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### Effect of surface treatment with commercial silane coupling agents



K. Ohashi\*, K. Miyake, H. Yamaguchi, A. Teranaka, T. Shiiya, K. Tomiyama, Y. Mukai, T. Nihei

Kanagawa Dental University, Japan

**Purpose:** The purpose of this study was to evaluate the clinical characteristics, tensile bond strength (TBS) and water resistance of eight commercial ceramic primers [seven commercial silane coupling agents (IN, MO, PL, RE, CP, TO and CS) and one alumina zirconia primer (AZ)].

**Methods and materials:** Glass plates were used as the adhered. The surfaces of the glass plates were treated according to the manufacturer's instruction using microbrushes for each group of ceramic primers. The modified glass surface and the stainless steel rod were manually held together to achieve bonding with the resin composite. Samples were then divided into two different immersion groups. One group was stored in distilled water at 37 °C for 7 days, and the other group was subjected to thermal cycling. The contact angles were obtained by dropping the resin monomer onto glass plates treated with methods described above. The TBS of resin composite and the wettability of resin monomer to glass surfaces treated with silane coupling agents was investigated, with 3-methacryloxypropyltrimethoxysilane (MPS, Shinetsu) used as a control. The values obtained from the experiments were analyzed using one-way ANOVA followed by Tukey's multiple comparison test ( $p < 0.05$ ).

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**Results:** Comparison of the two sealants at the end of one-year showed complete retention of 36% of glass ionomer sealant compared to 32% of resin-based sealant ( $P > 0.05$ ). Complete sealant loss was seen in 18% of the teeth in each material.

**Conclusion:** It was concluded that neither glass ionomer sealant nor resin-based sealant was superior to each other in the retention. Thus, both materials seem to be equally appropriate for the clinical application as a fissure sealant material.

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### Novel hydroxyapatite nanorods improve the anti-caries efficacy of enamel infiltrants



V.P. Feitosa<sup>1,\*</sup>, D.M.A. Neto<sup>2</sup>, E.V. Carvalho<sup>2</sup>, G. Mele<sup>3</sup>, L. Carbone<sup>4</sup>, S. Sauro<sup>1</sup>, P.B.A. Fechine<sup>2</sup>, L.K. Rodrigues<sup>1</sup>

<sup>1</sup> Federal University of Ceará, Department of Restorative Dentistry, Fortaleza, Brazil

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<sup>4</sup> UOS Pisa, CNR-IPCF, Pisa Italy; 5 CEU-Cardenal Herrera University, Department of Dental Biomaterials and Minimally Invasive Dentistry, Valencia, Spain

**Purpose:** To promote the nano-controlled synthesis of hydroxyapatite (HAp) controlling its crystallinity, crystal size and morphology. Furthermore, the HAp nanoparticles were incorporated into enamel resin infiltrants and other purposes were to survey the degree of conversion, level of resin infiltration and infiltrated enamel micro-hardness before and after pH cycling.

**Methods and materials:** HAp nanoparticles were synthesized using co-precipitation and hydrothermal (0 h, 2 h or 5 h) route and were structurally characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM). These nanoparticles were incorporated (10 wt%) into experimental enamel infiltrants to obtain an innovative material with adequate applicability and preventive potential. The degree of conversion (DC) was assessed by FTIR. Enamel caries lesions were created in extracted human molars and were infiltrated (Control, HAp-0 h, HAp-2 h and HAp-5 h) following the protocol of commercial enamel resin infiltrants. The level of resin

infiltration into demineralized enamel was evaluated using scanning electron microscopy (SEM). The treated specimens underwent pH cycling challenge to simulate recurrent caries. Knoop microhardness of infiltrated enamel specimens was analyzed before and after pH cycling. Data were statistically analyzed using ANOVA and Tukey test ( $p < 0.05$ ).

**Results:** XRD depicted approximately 11, 24 and 30 nm crystallite size for HAp-0 h, HAp-2 h and HAp-5 h respectively which was confirmed by the TEM micrographs. HAp-2 h and HAp-5 h presented higher crystallinity and nanorod shape whilst HAp-0 h was amorphous. HAp-2 h and HAp-5 h provided higher degree of conversion than the control resin and HAp-0 h infiltrant. Infiltrant penetration was similar among all materials. HAp-2 h and HAp-5 h achieved a protective role in avoiding demineralization of the treated enamel in contrast to control infiltrant (no HAp) and amorphous HAp-0 h which depicted significant microhardness reduction after pH cycling.

**Conclusion:** The incorporation of more crystalline hydroxyapatite nanorods (produced by 2/5 h hydrothermal treatment) clearly surpassed the conventional enamel infiltrant in terms of protection against recurrent caries and acid challenges. Moreover, crystalline hydroxyapatite nanorods improve the polymerization due to an increase of the degree of conversion.

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### Internal and marginal adaptation of bulk-filled composites



S.H. Park\*, J.J. Jung, S.H. Han

Yonsei University, Seoul, Republic of Korea

**Purpose:** The purpose of this study was to compare the marginal & internal adaptation of flowable bulk fill resin-based composites (FB-RBCs), non-flowable bulk fill resin-based composite (NFB-RBCs), and universal incremental-fill RBCs in MOD cavities in vitro.

**Methods and materials:** A Class II MOD cavity was prepared on 40 extracted sound lower molars. In Group 1 (control group), the preparation was filled with Filtek Z350 (Z3, 3M ESPE, St Paul, MN, USA) using the incremental filling technique. The FB-RBCs, SDR (SD, Group 2) (Dentsply Caulk, Milford, DE, USA) and Venus Bulk Fill (VB, Group 3) (Heraeus Kulzer, Dormagen, Germany), were placed in the core portion of the cavity first and Z350 was filled in the remaining cavity. The NFB-RBCs, Tetric N-Ceram Bulkfill (TB, Group 4) (Ivoclar Vivadent, Schaan, Liechtenstein) and SonicFill (SF, Group 5) (Kerr, West Collins, Orange, CA, USA) were bulk-filled into the preparation. Images of the magnified marginal area were captured under 100× magnification before and after thermo-mechanical loading and the percentage ratio of the imperfect margin (%IM) was calculated. Gaps, cracks in the enamel layer, and chippings of composites, enamel or dentine, were all considered to be imperfect margins. After thermo-mechanical load cycling, micro-CT images were taken cross-sectionally. Internal adaptation was expressed as imperfect margin % at the interface (IM%). On the images of micro-CT, imperfect margin% was measured at following four interfaces: Buccal wall of proximal

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## Abstracts of the Academy of Dental Materials Annual Meeting, 7–10 October 2015 – Hawaii, USA

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### A 3D-printed TCP/HA osteoconductive scaffold for vertical bone augmentation

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Switzerland

**Purpose:** OsteoFlux<sup>®</sup> (OF) is a 3D printed porous block of layered strands of tricalcium phosphate (TCP) and hydroxyapatite. Its porosity and interconnectivity are defined and it can be readily shaped to conform to the bone bed's morphology. We investigated the performance of OF as a scaffold to promote the vertical growth of cortical bone in a sheep calvarial model.

**Methods and materials:** Six titanium hemispheres were filled with OF, OF+ bmp2 (100 µg), Bio-Oss (particulate bovine bone, BO) or Ceros (particulate TCP, CO) and placed onto the calvaria of 12 adult sheep (6 hemispheres/sheep). Histomorphometric analyses were performed after 8 and 16 weeks.

**Results:** OF led to substantial vertical bone growth by 8 weeks and outperformed BO and CO by a factor of 2 yielding OF 22% ± 2.1; BO 11.5% ± 1.9; CO 12.9% ± 2.1 total new bone. 3 mm away from the bony bed, OF led to a 4-fold increase in new bone relative to BO and CO ( $n=8$ ,  $p < 0.002$ ). At 16 weeks, OF, BO and CO behaved similarly and showed marked new bone synthesis. A moderate degradation was observed at 16 weeks for all bone substitutes. Addition of bmp2 in OF scaffolds led to a dramatic improvement in local bone metabolism and material resorption that was 3 times higher as compared to OF alone.

**Conclusion:** When compared to existing bone substitutes, OF enhances vertical bone growth during the first 2 months after implantation in a sheep calvarial model. The controlled porous structure translated in a high osteoconductivity and resulted in a bone mass 3 mm above the bony bed that was 4-times greater than that obtained with standard substitutes.



Acceleration of bone development kinetics by addition of bmp2 showed that the material has the ability to resorb in large proportions. These results are promising but must be confirmed in clinical tests.

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### Facial analysis by 3D-stereophotogrammetric imaging: Reproducibility and clinical application

R. Ceinos\*, M.-F. Bertrand, E. Medioni, L.  
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Odontologie Nice, France

**Purpose:** The goal of this study was to assess the reproducibility of an innovative method for facial analysis with 3D-stereophotogrammetry (3D-spg). This tool is then put into practice in a clinical case for aesthetic purposes.

**Methods and materials:** Twelve subjects with no obvious malocclusion participated in this study. For each of them, four photographs were acquired using the LifeViz<sup>TM</sup>, an absolute calibration 3D-spg system mounted on a tripod. The optical system was composed of a double-lens beamsplitter coupled to an inverted polarization double flash. Then, the three-dimensional facial reconstruction was obtained. The images were analyzed with the DermaPix<sup>TM</sup> image management software. Distance and image centring between each shot were standardized thanks to ground markings and a laser pointer. The landmarks used in this study were selected according to the definitions given by Farkas, the pioneer of modern anthropometry. Distances between landmarks were recorded by two different operators and each operator repeated the



glass ionomer sealant, and sixty contra-lateral permanent first molars were sealed with Clinpro, a resin-based sealant. Evaluation of sealant retention was performed every 4 months over one year. The data obtained was tabulated and subjected to statistical analysis using the Chi-square test.

**Results:** Comparison of the two sealants at the end of one-year showed complete retention of 36% of glass ionomer sealant compared to 32% of resin-based sealant ( $P > 0.05$ ). Complete sealant loss was seen in 18% of the teeth in each material.

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### Novel hydroxyapatite nanorods improve the anti-caries efficacy of enamel infiltrants



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**Purpose:** To promote the nano-controlled synthesis of hydroxyapatite (HAp) controlling its crystallinity, crystal size and morphology. Furthermore, the HAp nanoparticles were incorporated into enamel resin infiltrants and other purposes were to survey the degree of conversion, level of resin infiltration and infiltrated enamel micro-hardness before and after pH cycling.

**Methods and materials:** HAp nanoparticles were synthesized using co-precipitation and hydrothermal (0 h, 2 h or 5 h) route and were structurally characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM). These nanoparticles were incorporated (10 wt%) into experimental enamel infiltrants to obtain an innovative material with adequate applicability and preventive potential. The degree of conversion (DC) was assessed by FTIR. Enamel caries lesions were created in extracted human molars and were infiltrated (Control, HAp-0 h, HAp-2 h and HAp-5 h) following the protocol of commercial enamel resin infiltrants. The level of resin

infiltration into demineralized enamel was evaluated using scanning electron microscopy (SEM). The treated specimens underwent pH cycling challenge to simulate recurrent caries. Knoop microhardness of infiltrated enamel specimens was analyzed before and after pH cycling. Data were statistically analyzed using ANOVA and Tukey test ( $p < 0.05$ ).

**Results:** XRD depicted approximately 11, 24 and 30 nm crystallite size for HAp-0 h, HAp-2 h and HAp-5 h respectively which was confirmed by the TEM micrographs. HAp-2 h and HAp-5 h presented higher crystallinity and nanorod shape whilst HAp-0 h was amorphous. HAp-2 h and HAp-5 h provided higher degree of conversion than the control resin and HAp-0 h infiltrant. Infiltrant penetration was similar among all materials. HAp-2 h and HAp-5 h achieved a protective role in avoiding demineralization of the treated enamel in contrast to control infiltrant (no HAp) and amorphous HAp-0 h which depicted significant microhardness reduction after pH cycling.

**Conclusion:** The incorporation of more crystalline hydroxyapatite nanorods (produced by 2/5 h hydrothermal treatment) clearly surpassed the conventional enamel infiltrant in terms of protection against recurrent caries and acid challenges. Moreover, crystalline hydroxyapatite nanorods improve the polymerization due to an increase of the degree of conversion.

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### Internal and marginal adaptation of bulk-filled composites



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**Purpose:** The purpose of this study was to compare the marginal & internal adaptation of flowable bulk fill resin-based composites (FB-RBCs), non-flowable bulk fill resin-based composite (NFB-RBCs), and universal incremental-fill RBCs in MOD cavities in vitro.

**Methods and materials:** A Class II MOD cavity was prepared on 40 extracted sound lower molars. In Group 1 (control group), the preparation was filled with Filtek Z350 (Z3, 3M ESPE, St Paul, MN, USA) using the incremental filling technique. The FB-RBCs, SDR (SD, Group 2) (Dentsply Caulk, Milford, DE, USA) and Venus Bulk Fill (VB, Group 3) (Heraeus Kulzer, Dormagen, Germany), were placed in the core portion of the cavity first and Z350 was filled in the remaining cavity. The NFB-RBCs, Tetric N-Ceram Bulkfill (TB, Group 4) (Ivoclar Vivadent, Schaan, Liechtenstein) and SonicFill (SF, Group 5) (Kerr, West Collins, Orange, CA, USA) were bulk-filled into the preparation. Images of the magnified marginal area were captured under 100× magnification before and after thermo-mechanical loading and the percentage ratio of the imperfect margin (%IM) was calculated. Gaps, cracks in the enamel layer, and chippings of composites, enamel or dentine, were all considered to be imperfect margins. After thermo-mechanical load cycling, micro-CT images were taken cross-sectionally. Internal adaptation was expressed as imperfect margin % at the interface (IM%). On the images of micro-CT, imperfect margin% was measured at following four interfaces: Buccal wall of proximal



box (BWIM%); Lingual wall of proximal box (LWIM%); Gingival floor of proximal box (GFIM%); Mesial floor of pulpal cavity (MFIM%); Distal floor of pulpal cavity (DFIM%). To compare the difference, both two-way and one-way ANOVA with Scheffe test was applied at 95% confidence interval.

**Results:** As for marginal adaptation, %IM was in the order of Group 4, 5 = Group 1, 3 = Group 2 ( $p < 0.05$ ). As for internal adaptation, %IM was in the order of group 4,5 = group 1,2 = group 3 ( $p < 0.05$ ). %IM was higher after thermo-mechanical loading than before loading in marginal adaptation ( $p < 0.05$ ). As for cavity walls, BWIM%, MFIM% < GFIM%, MFIM%, DFIM% ( $p < 0.05$ ) in the internal adaptation.

**Conclusion:** NFB-RBCs showed the same or better marginal & internal adaptation than FB-RBCs.

Groups	IM% (marginal adaptation)	IM% (marginal adaptation)	IM% (internal adaptation)
	Before loading	After loading	After loading
1	4.49 <sup>a</sup>	20.44 <sup>ab</sup>	32.90 <sup>a</sup>
2	2.64 <sup>a</sup>	24.76 <sup>b</sup>	37.03 <sup>b</sup>
3	1.75 <sup>a</sup>	22.05 <sup>ab</sup>	40.95 <sup>b</sup>
4	4.07 <sup>a</sup>	12.73 <sup>a</sup>	32.94 <sup>a</sup>
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### Microhardness evaluation of enamel submitted to bleaching with hydrogen peroxide



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**Purpose:** The aim of this study was to conduct an in vitro evaluation of the microhardness of dental enamel submitted to a bleaching treatment with high and low concentrations of hydrogen peroxide (HP) agents.

**Methods and materials:** Sixty blocks of enamel were obtained from third human molars, which were then embedded and flattened. The blocks were bleached with six different agents ( $n = 10$ ). The following agents of high HP concentration (for in-office use) were evaluated: Whiteness HP Maxx/FGM (35% HP), Whiteness HP Blue/FGM (35% HP + 2% calcium gluconate), Pola Office+/SDI (37.5% HP + 5% potassium nitrate) and Opalescence Boost/Ultradent (38% HP + 1.1% ion fluoride + 3% potassium nitrate), whereas the following agents of low HP concentration (for home use) were evaluated: Pola Day/SDI (9.5% HP) and White Class 10%/FGM (10% HP + potassium nitrate + calcium + fluoride). The manufacturer's instructions for each bleaching agent were followed. Two bleaching treatment options were applied, depending on the HP concentration. The treatments with high HP

concentration were applied in 3 sessions, with a time interval of 7 days, whereas those with low HP concentration were applied for 14 days. The enamel blocks were immersed in artificial saliva between sessions. The Knoop microhardness measurements were taken on the surface of the enamel before, during (in the period after the first application up to after the third application of high HP concentration, or respectively after the 14th application of low HP concentration) and 14 days after the end of the treatment, at six different times.

**Results:** The two-way analysis of variance showed that the microhardness values were influenced by both the whitening agent used ( $p < 0.001$ ) and the time of application ( $p < 0.001$ ). The Tukey test showed that the enamel bleached with Whiteness HP Maxx or White Class 10% presented lower microhardness values than with the other agents. The highest enamel microhardness values for all the bleaching agents were found up to the 2nd application of high HP concentration, or respectively after the 8th application of low HP concentration, after which time there was a reduction in values up to the end of the treatment.

**Conclusion:** The concentration, composition and application protocol of each bleaching agent can influence the enamel microhardness values, in that microhardness decreases as a function of time, regardless of the agent used.

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### Calcium and phosphorous content in enamel submitted to bleaching treatments



A.V.D. Pinto\*, E.C. Bridi, E.F. Martinez, F.L.B. Amaral, F.M.G. França, C.P. Turssi, F.M. Flório, R.T. Basting

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**Purpose:** The aim of this in vitro study was to evaluate the calcium and phosphorous content in human dental enamel before, during and after bleaching treatments with high and low hydrogen peroxide (HP) concentrations

**Methods and materials:** Forty-eight sound human third molars were used. Their roots were embedded in polystyrene resin, and immersed for 7 days in an artificial saliva solution. They were distributed into six groups ( $n = 8$ ) to receive the bleaching treatments. The agents of high HP concentration (for in-office use) evaluated were: Whiteness HP Maxx/FGM (35% HP), Whiteness HP Blue/FGM (35% HP + 2% calcium gluconate), Pola Office+/SDI (37.5% HP + 5% potassium nitrate) and Opalescence Boost/Ultradent (38% HP + 1.1% ion fluoride + 3% potassium nitrate); the agents of low HP concentration (for home use) evaluated were: Pola Day/SDI (9.5% HP) and White Class 10%/FGM (10% HP + potassium nitrate + calcium + fluoride). The treatments with high HP concentration were applied in 3 sessions, with a time interval of 7 days, whereas those with low HP concentration were applied for 14 days. A spectrophotometry was used to determine calcium and phosphorous content by using the enamel microbiopsy technique. Enamel microbiopsies were

box (BWIM%); Lingual wall of proximal box (LWIM%); Gingival floor of proximal box (GFIM%); Mesial floor of pulpal cavity (MFIM%); Distal floor of pulpal cavity (DFIM%). To compare the difference, both two-way and one-way ANOVA with Scheffe test was applied at 95% confidence interval.

**Results:** As for marginal adaptation, %IM was in the order of Group 4, 5 = Group 1, 3 = Group 2 ( $p < 0.05$ ). As for internal adaptation, %IM was in the order of group 4,5 = group 1,2 = group 3 ( $p < 0.05$ ). %IM was higher after thermo-mechanical loading than before loading in marginal adaptation ( $p < 0.05$ ). As for cavity walls, BWIM%, MFIM% < GFIM%, MFIM%, DFIM% ( $p < 0.05$ ) in the internal adaptation.

**Conclusion:** NFB-RBCs showed the same or better marginal & internal adaptation than FB-RBCs.

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### Microhardness evaluation of enamel submitted to bleaching with hydrogen peroxide



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**Results:** The two-way analysis of variance showed that the microhardness values were influenced by both the whitening agent used ( $p < 0.001$ ) and the time of application ( $p < 0.001$ ). The Tukey test showed that the enamel bleached with Whiteness HP Maxx or White Class 10% presented lower microhardness values than with the other agents. The highest enamel microhardness values for all the bleaching agents were found up to the 2nd application of high HP concentration, or respectively after the 8th application of low HP concentration, after which time there was a reduction in values up to the end of the treatment.

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### Calcium and phosphorous content in enamel submitted to bleaching treatments



A.V.D. Pinto\*, E.C. Bridi, E.F. Martinez, F.L.B. Amaral, F.M.G. França, C.P. Turssi, F.M. Flório, R.T. Basting

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### Calcium and phosphorous content in enamel submitted to bleaching treatments



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**Methods and materials:** Forty-eight sound human third molars were used. Their roots were embedded in polystyrene resin, and immersed for 7 days in an artificial saliva solution. They were distributed into six groups ( $n = 8$ ) to receive the bleaching treatments. The agents of high HP concentration (for in-office use) evaluated were: Whiteness HP Maxx/FGM (35% HP), Whiteness HP Blue/FGM (35% HP + 2% calcium gluconate), Pola Office+/SDI (37.5% HP + 5% potassium nitrate) and Opalescence Boost/Ultradent (38% HP + 1.1% ion fluoride + 3% potassium nitrate); the agents of low HP concentration (for home use) evaluated were: Pola Day/SDI (9.5% HP) and White Class 10%/FGM (10% HP + potassium nitrate + calcium + fluoride). The treatments with high HP concentration were applied in 3 sessions, with a time interval of 7 days, whereas those with low HP concentration were applied for 14 days. A spectrophotometry was used to determine calcium and phosphorous content by using the enamel microbiopsy technique. Enamel microbiopsies were

performed before the bleaching treatment (baseline), during it (in the period after the first application up to after the third application of high HP concentration, or respectively after the 14th application of low HP concentration), and 14 days after the end of the treatment, at six different times. For the purpose of statistical analysis, the Tukey-Kramer test for repeated measures was used.

**Results:** Teeth submitted to bleaching with Pola Office + Opalescence Boost, Pola Day and White Class 10% showed a significant decrease in calcium concentrations in enamel over time ( $p < 0.0001$ ), whereas a significant decrease in phosphorous was observed over time for enamel treated with Pola Office + and White Class 10% ( $p < 0.0001$ ). In regard to the other agents, the concentrations of calcium and phosphorous in enamel were not affected over time. Furthermore, there were no significant differences between calcium and phosphorous concentrations for the bleaching agents ( $p = 0.28$  for calcium;  $p = 0.08$  for phosphorous) among the six different periods studied.

**Conclusion:** It can be concluded that the application protocol, the concentrations and the compositions of bleaching agents can influence the calcium and phosphorous content in enamel, with a decrease in this content over time, for some bleaching agents.

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#### Bleaching with peroxides delivered in custom-made or prefilled disposable trays



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**Purpose:** The aim of this study was to conduct an *in vivo* evaluation of tooth sensitivity, gingival irritation and tray comfort, regarding the use of different trays for home-use bleaching techniques, based on hydrogen peroxide (HP) or carbamide peroxide (CP) delivered in custom-made or prefilled disposable trays.

**Methods and materials:** Seventy-five patients with no tooth sensitivity and no gingival irritation at the beginning of the experiment were included in this study to receive a bleaching treatment. The patients were randomly distributed to receive one of the following bleaching agents ( $n = 25$ ): 10% HP delivered in prefilled disposable trays (Opalescence Trés White Supreme 10% Mint/Ultradent), 9.5% HP delivered in custom-made trays (Pola Day/SDI), and 10% CP delivered in custom-made trays (Opalescence PF 10% Mint/Ultradent). The custom-made mouth trays were made by one researcher using flexible vinyl acetate that was 1.0 mm thick. The custom trays were cut 1 mm beyond the clinical crown length. Bleaching treatment with HP was performed for 30 min per day, for 14 days, whereas bleaching with CP was performed for 8 h per night, for 14 days. Tooth sensitivity and tray comfort were evaluated by visual analogical scale at the 7th and 14th days of treatment. Gingival irritation was evaluated as absent, localized irritation

or generalized irritation. The Friedman, Kruskal-Wallis and Wilcoxon tests were applied to the results.

**Results:** There was an increase in tooth sensitivity for all bleaching agents at the 7th day of treatment (Friedman;  $p < 0.001$ ), but no differences in sensitivity between the 7th and 14th days. There were no significant differences for tooth sensitivity and gingival irritation among bleaching agents at baseline, 7th and 14th days of treatment (Kruskal-Wallis;  $p > 0.05$ ). At the 7th and 14th days, perception of tray comfort was similar for the bleaching agents (Kruskal-Wallis;  $p > 0.05$ ), and there were no differences among the time periods for each bleaching agent (Wilcoxon;  $p > 0.05$ ). For gingival irritation, there were no differences among time intervals for each bleaching agent (Friedman;  $p < 0.05$ ).

**Conclusion:** There was an increase in tooth sensitivity after 7 days of bleaching treatment, but sensitivity then remained stable up to the 14th day of treatment. No gingival irritation was observed for the different agents during bleaching treatment. The tray comfort was perceived as comfortable, regardless of the hydrogen peroxide or carbamide peroxide agent delivered in the custom-made or prefilled disposable trays.

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#### Efficacy of at-home bleaching: Meta-analysis



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**Purpose:** To compare the color change produced by tray-delivered carbamide peroxide [CP] versus hydrogen peroxide products [HP] for at-home bleaching through a systematic review and meta-analysis.

**Methods and materials:** MEDLINE via PubMeb, Scopus, Web of Science, LILACS, BBO and Cochrane Library and Grey literature were searched without restrictions. The abstracts of the IADR (1990–2014), unpublished and ongoing trials registries were also searched. Dissertations and theses were explored using the ProQuest Dissertations and Periodicos Capes Theses databases. We included randomized clinical trials that compared tray-delivered CP versus HP for at-home dental bleaching. The color change in shade guide units (SGU) and  $\Delta E$  were the primary outcomes, and tooth sensitivity and gingival irritation were the secondary outcomes. The risk of bias tool of the Cochrane Collaboration was used for quality assessment.

**Results:** Data: After duplicates removal, 1379 articles were identified. After title and abstract screening, 29 studies remained. Fifteen studies were further excluded remaining 14 studies for qualitative analyses and 6 for the meta-analysis of the primary and secondary outcomes. For  $\Delta E$ , the standardized mean difference was  $-0.38$  (95% CI  $-0.62$  to  $-0.15$ ), which favoured tray-delivered CP products ( $p < 0.001$ ). The color change in SGU ( $p = 0.06$ ), tooth sensitivity ( $p = 0.83$ ) and gingival irritation ( $p = 0.62$ ) was not significant different between groups.

performed before the bleaching treatment (baseline), during it (in the period after the first application up to after the third application of high HP concentration, or respectively after the 14th application of low HP concentration), and 14 days after the end of the treatment, at six different times. For the purpose of statistical analysis, the Tukey-Kramer test for repeated measures was used.

**Results:** Teeth submitted to bleaching with Pola Office + Opalescence Boost, Pola Day and White Class 10% showed a significant decrease in calcium concentrations in enamel over time ( $p < 0.0001$ ), whereas a significant decrease in phosphorous was observed over time for enamel treated with Pola Office + and White Class 10% ( $p < 0.0001$ ). In regard to the other agents, the concentrations of calcium and phosphorous in enamel were not affected over time. Furthermore, there were no significant differences between calcium and phosphorous concentrations for the bleaching agents ( $p = 0.28$  for calcium;  $p = 0.08$  for phosphorous) among the six different periods studied.

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**Methods and materials:** Seventy-five patients with no tooth sensitivity and no gingival irritation at the beginning of the experiment were included in this study to receive a bleaching treatment. The patients were randomly distributed to receive one of the following bleaching agents ( $n = 25$ ): 10% HP delivered in prefilled disposable trays (Opalescence Trés White Supreme 10% Mint/Ultradent), 9.5% HP delivered in custom-made trays (Pola Day/SDI), and 10% CP delivered in custom-made trays (Opalescence PF 10% Mint/Ultradent). The custom-made mouth trays were made by one researcher using flexible vinyl acetate that was 1.0 mm thick. The custom trays were cut 1 mm beyond the clinical crown length. Bleaching treatment with HP was performed for 30 min per day, for 14 days, whereas bleaching with CP was performed for 8 h per night, for 14 days. Tooth sensitivity and tray comfort were evaluated by visual analogical scale at the 7th and 14th days of treatment. Gingival irritation was evaluated as absent, localized irritation

or generalized irritation. The Friedman, Kruskal-Wallis and Wilcoxon tests were applied to the results.

**Results:** There was an increase in tooth sensitivity for all bleaching agents at the 7th day of treatment (Friedman;  $p < 0.001$ ), but no differences in sensitivity between the 7th and 14th days. There were no significant differences for tooth sensitivity and gingival irritation among bleaching agents at baseline, 7th and 14th days of treatment (Kruskal-Wallis;  $p > 0.05$ ). At the 7th and 14th days, perception of tray comfort was similar for the bleaching agents (Kruskal-Wallis;  $p > 0.05$ ), and there were no differences among the time periods for each bleaching agent (Wilcoxon;  $p > 0.05$ ). For gingival irritation, there were no differences among time intervals for each bleaching agent (Friedman;  $p < 0.05$ ).

**Conclusion:** There was an increase in tooth sensitivity after 7 days of bleaching treatment, but sensitivity then remained stable up to the 14th day of treatment. No gingival irritation was observed for the different agents during bleaching treatment. The tray comfort was perceived as comfortable, regardless of the hydrogen peroxide or carbamide peroxide agent delivered in the custom-made or prefilled disposable trays.

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#### Efficacy of at-home bleaching: Meta-analysis



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<sup>2</sup> State University of Ponta Grossa, Brazil

<sup>3</sup> Federal University of Rio De Janeiro, Brazil

**Purpose:** To compare the color change produced by tray-delivered carbamide peroxide [CP] versus hydrogen peroxide products [HP] for at-home bleaching through a systematic review and meta-analysis.

**Methods and materials:** MEDLINE via PubMeb, Scopus, Web of Science, LILACS, BBO and Cochrane Library and Grey literature were searched without restrictions. The abstracts of the IADR (1990–2014), unpublished and ongoing trials registries were also searched. Dissertations and theses were explored using the ProQuest Dissertations and Periodicos Capes Theses databases. We included randomized clinical trials that compared tray-delivered CP versus HP for at-home dental bleaching. The color change in shade guide units (SGU) and  $\Delta E$  were the primary outcomes, and tooth sensitivity and gingival irritation were the secondary outcomes. The risk of bias tool of the Cochrane Collaboration was used for quality assessment.

**Results:** Data: After duplicates removal, 1379 articles were identified. After title and abstract screening, 29 studies remained. Fifteen studies were further excluded remaining 14 studies for qualitative analyses and 6 for the meta-analysis of the primary and secondary outcomes. For  $\Delta E$ , the standardized mean difference was  $-0.38$  (95% CI  $-0.62$  to  $-0.15$ ), which favoured tray-delivered CP products ( $p < 0.001$ ). The color change in SGU ( $p = 0.06$ ), tooth sensitivity ( $p = 0.83$ ) and gingival irritation ( $p = 0.62$ ) was not significant different between groups.

performed before the bleaching treatment (baseline), during it (in the period after the first application up to after the third application of high HP concentration, or respectively after the 14th application of low HP concentration), and 14 days after the end of the treatment, at six different times. For the purpose of statistical analysis, the Tukey-Kramer test for repeated measures was used.

**Results:** Teeth submitted to bleaching with Pola Office + Opalescence Boost, Pola Day and White Class 10% showed a significant decrease in calcium concentrations in enamel over time ( $p < 0.0001$ ), whereas a significant decrease in phosphorous was observed over time for enamel treated with Pola Office + and White Class 10% ( $p < 0.0001$ ). In regard to the other agents, the concentrations of calcium and phosphorous in enamel were not affected over time. Furthermore, there were no significant differences between calcium and phosphorous concentrations for the bleaching agents ( $p = 0.28$  for calcium;  $p = 0.08$  for phosphorous) among the six different periods studied.

**Conclusion:** It can be concluded that the application protocol, the concentrations and the compositions of bleaching agents can influence the calcium and phosphorous content in enamel, with a decrease in this content over time, for some bleaching agents.

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#### Bleaching with peroxides delivered in custom-made or prefilled disposable trays



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**Purpose:** The aim of this study was to conduct an *in vivo* evaluation of tooth sensitivity, gingival irritation and tray comfort, regarding the use of different trays for home-use bleaching techniques, based on hydrogen peroxide (HP) or carbamide peroxide (CP) delivered in custom-made or prefilled disposable trays.

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**Purpose:** To compare the color change produced by tray-delivered carbamide peroxide [CP] versus hydrogen peroxide products [HP] for at-home bleaching through a systematic review and meta-analysis.

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**Conclusion:** Tray-delivered CP gels showed a better whitening efficacy than HP-based products, although the difference did not reach the threshold for clinical identification. Both whitening systems demonstrated equal level of gingival irritation and tooth sensitivity.

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### Anti-demineralization effect of novel S-PRG filler containing varnishes on dentin



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Kanagawa Dental University, Yokosuka, Kanagawa, Japan

**Purpose:** There are many fluoride varnishes on the market which prevent dental caries. Recently, surface pre-reacted glass-ionomer (S-PRG) filler which releases ions including fluoride, strontium, sodium, boron, aluminum and silicate ions, was developed. In this study, we investigated anti-demineralization effects of novel S-PRG filler containing varnishes on dentin *in vitro*.

**Methods and materials:** Thirty-five bovine root specimens with a flat dentin surface were prepared. A 3 × 2 mm test surface was made with acid-resistant nail varnish on each dentin surface. Seven specimens were allocated to each of five treatment groups were as follows; (1) MI Varnish (MIV: NaF 5% + CPP-ACP; GC America), (2) F Varnish (FV: NaF 5%; Bee Brand Medico Dental), (3) PRG Varnish I (PV: S-PRG filler 40%; Shofu), (4) PRG Varnish II (PVF: NaF 5% + S-PRG filler 40%; Shofu), (5) Control group (CONT), acid-resistant nail varnish was applied instead of the material. PV and PVF were novel varnishes. Each of five experimental materials was applied to 3 × 1 mm of the test surface with a brush, and the other half of the surface was left as exposed lesion area for demineralization. The specimens were immersed in 8% methylcellulose gel demineralization system (1.5 mM Ca, 0.9 mM P, 50 mM acetic acid, pH5.0) for 1 week at 37 °C. Mineral profiles and integrated mineral loss (IML: vol% × μm) of lesions adjacent to the materials were obtained by transversal microradiography and analytical software. Statistical difference of IML was analyzed with one-way ANOVA and Tukey's HSD multiple comparison test with  $\alpha = 0.05$ .

**Results:** The TMR image of CONT revealed a mineral profile with a thin surface layer and a severe body of lesion. In contrast, PVF showed comparatively high mineral content over 40 vol% at the surface layer, and a lesion body of about 20 vol%. PV (3,386 ± 292) and PVF (2,855 ± 419) showed significantly lower IML than CONT (4,282 ± 483). Furthermore, the IML of PVF was significantly lower than those of MIV (3,835 ± 474) and FV (3,982 ± 314). There were no significant differences between PV and PVF. Fluoride and strontium ions released from S-PRG filler would react with hydroxyapatite to form fluoroapatite, strontium apatite and/or fluoridated apatite. Also, strontium and sodium ions released from this filler would buffer the acidic condition.

**Conclusion:** This investigation indicated that the novel S-PRG filler containing varnishes, especially, the varnish

including NaF had superior anti-demineralization effects, compared with conventional varnishes, on root dentin.

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### Influence of light-irradiation on polymerization shrinkage of recent bulk-fill composites



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**Purpose:** Bulk-fill composites have been applied for direct composite restoration recently. The purpose of this study was to examine the influence of the light irradiation condition on polymerization shrinkage of typical bulk-fill composites.

**Methods and materials:** Three typical recent bulk-fill composites; Filtek Bulk Fill (FBF, 3M ESPE), SDR (SDR, Dentsply), Tetric N-Ceram Bulk Fill (TET, Ivoclar vivadent), were selected and compared with two types of popular composite; a universal composite; Filtek Supreme Ultra (FSU, 3M ESPE) and a flowable composite; Filtek Supreme Ultra Flowable (FSF, 3M ESPE). Each composite was filled into a translucent acrylic mold (f 4 × 8 mm) and then cured by three different light irradiation methods, HL: irradiated with a halogen light source (Optilux 501, Kerr, 750 mW/cm<sup>2</sup> × 20 s), HP: irradiated with high-power mode (1800 mW/cm<sup>2</sup> × 3 s) or SS: soft-start mode (1000 mW/cm<sup>2</sup> × 2 s + 1800 mW/cm<sup>2</sup> × 3 s) using a LED light source (G-Light Prima, GC). Linear polymerization shrinkage was measured using a diode-laser displacement sensor (HL-C105B-BK, SUNX) during 0–180 s ( $n = 5$ ). The data were analyzed using two-way ANOVA and Tukey's  $q$ -test.

**Results:** The mean values of linear polymerization shrinkage (S.D.) in % under HL/HP/SS methods were FBF: 1.14(0.05)/1.51(0.14)/1.16(0.18), SDR: 1.12(0.30)/1.32(0.27)/1.27(0.19), TET: 1.37(0.29)/1.73(0.33)/1.39(0.15), FSU: 1.26(0.26)/1.60(0.28)/1.18(0.31) and FSF: 2.14(0.35)/2.71(0.33)/2.17(0.29). Both factors in composite and light irradiation method influenced shrinkage rate significantly at  $p < 0.01$  and there was no interaction effect between those of two factors. There was no significant difference in shrinkage among FBF, SDR, TET and FSU. The values of those four composite were although statistically smaller than that of FSF at  $p < 0.01$ . The rates of HL and SS light irradiations were significantly smaller than the value of HP method at  $p < 0.01$ .

**Conclusion:** The linear polymerization shrinkage of typical recent bulk-fill resin composites was similar to or smaller than the values of a universal resin composite and a flowable composite in this study. Both light irradiation methods with a halogen light source (750 mW/cm<sup>2</sup> × 20 s) and soft-start mode (1000 mW/cm<sup>2</sup> × 2 s + 1800 mW/cm<sup>2</sup> × 3 s) using a LED light source were effective for reduction of polymerization shrinkage compared with high-power mode (1800 mW/cm<sup>2</sup>) using a LED light source.

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### Bonding property of resin cement to composite resin crown



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Tokyo Medical and Dental University, Tokyo, Japan

**Purpose:** Direct Crowns made of a composite resin material recently developed by 3M ESPE. These crowns maintain the coronal morphology even in an uncured state. The advantage is that the entire process of crown fabrication until cementing and delivery can be completed chairside on the same day. There has not been a detailed study on how adhesion with resin cement is affected by bonding performed immediately after polymerization of composite resin. Thus, this study examined the effects of time period after crown fabrication on the bonding property.

**Methods and materials:** This study used a combination of composite resin and resin cement from the same manufacturer. The following items were used: 3M ESPE Direct Crowns with RelyX Unicem 2 Automix self-adhesive resin cement and Kuraray Noritake Dental Estenia C&B with Panavia F 2.0. Discs of Estenia C&B and Direct Crowns were fabricated (10 mm diameter and 3 mm thickness). They were divided into groups with bonding performed immediately after fabrication of discs (0h-Direct Crown group and 0h-Estenia C&B group) and groups with bonding performed 48 h later (48 h-Direct Crown group and 48h-Estenia C&B group). A microtensile bond strength test was performed after bonding. Subsequently, fracture surface morphology of samples was examined by scanning electron microscopy. ATR-FTIR (Attenuated total reflection-Fourier transform infrared spectroscopy) analysis was performed on Direct Crowns.

**Results:** The adhesive strength was significantly higher in the 0h-Direct Crown group ( $76.6 \pm 13.4$  MPa) compared with the 0h-Estenia C&B group ( $60.9 \pm 14.8$  MPa), the 48 h-Direct Crown group ( $45.8 \pm 9.7$  MPa), and the 48 h-Estenia C&B group ( $53.7 \pm 12.5$  MPa). When fracture surfaces were examined after the microtensile bond strength test, the 0h-Direct Crown group had many mixed fractures but no fracture at the interface between composite resin and resin cement. The other three groups had mainly mixed fractures and fractures at the interface between composite resin and resin cement. The results of ATR-FTIR analysis suggest that the degree of surface polymerization was low for Direct Crowns immediately after fabrication compared with Direct Crowns 24 h later.

**Conclusion:** The results of this study suggest that Direct Crowns are effective in clinical use with their high adhesion due to bonding immediately after fabrication.

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### Influence of different whitening intervals on tooth whitening



M. Hasegawa\*, D. Kita, A. Ishikawa

Nippon Dental University, Japan

**Purpose:** With a desire to dental aesthetic, tooth whitening is generally used widely as one step of dental treatment without involving the tooth cutting. At-home whitening, which is used at home by the patient, is often selected from convenience. However patients often forget to perform whitening. In this study, we evaluated the effect of tooth whitening of two different intervals of at-home whitening.

**Methods and materials:** TiON take home (GC co.) was used as the whitening agent. 30 Participants who consented to the study contents and their upper incisor tooth weren't treated in crown restoration or resin restoration (labial surface) and weren't planning to be performed for dental treatment during study duration, were selected. Participants were divided equally into two different whitening interval groups at random. Group1 were treated at-home whitening 14 times every day ( $n=15$ ). Group2 were treated at-home whitening 14 times every three days ( $n=15$ ). Whitening procedure was followed with manufacturer's instructions. Before and after whitening treatment,  $L^*$ ,  $a^*$ , and  $b^*$  were measured using a colorimeter (Shadepilot, Dentsply). Color differences  $\Delta E^*ab$  between before and after whitening were calculated, and independent t-test was performed.

**Results:** The color difference  $\Delta E^*ab$  of Group 1 were 5.58.  $\Delta E^*ab$  of Group 2 were 4.55. There was significant difference between Groups 1 and 2 ( $p < 0.05$ ).

**Conclusion:** The color difference  $\Delta E^*ab$  of Group1 and Group2 were more than 4, it was confirmed that sufficient whitening effects can be achieved on clinical use. Furthermore, without forgetting to do whitening every day was more effective than to do whitening every three days. It seemed that whitening material was easier to penetrate the tooth.

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### Primary bone healing of autologous grafts



M.E. Draenert<sup>1,\*</sup>, A. Draenert<sup>2</sup>, R. Hickel<sup>1</sup>

<sup>1</sup> Clinic for Restorative Dentistry and Periodontology, Ludwig-Maximilian University of Munich, Germany

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**Purpose:** If an autologous graft is implanted in a bony bed, the continuity of the bone structure around the implant is interrupted and an interface is created, between the graft and the surrounding bone. In case of a press-fit implantation, a static stress deforms the bone according to its stiffness. In case of a cancellous bone graft, the graft is deformed in a compact recipient bed, in case of a compact graft in a more spongy-structured bed; the strain adapted bone remodelling is focused

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### Bonding property of resin cement to composite resin crown



O. Aramaki\*, R. Takahashi, T. Wada, M. Uo, J. Tagami

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to the recipient bone bed. Revascularization and bone formation depend upon the structure of bed and graft. The question of this study was how the revascularization and time-dependent bone formation can be analyzed under stable press-fit implantation.

**Methods and materials:** Contact healing of a cancellous graft was studied in 18 giant rabbits using a standardized animal model of the distal femur. In the center of the patellar groove, a 4.60-mm-large graft cylinder from the contralateral side was inserted into a 4.50-mm-large defect; both were created using a wet-grinding surgical procedure with diamond-coated instruments. The press fit was based on a difference in diameter of 1/10 of a millimeter. The titer dynamic of bone formation was investigated applying the polychromatic sequential labeling according to Rahn and Perren. The stages were 1, 4, 6, 9 (4 animals) and 11 weeks (2 animals). Non-decalcified histology was performed of a complete sequence of serial cuts perpendicularly to the transplant. The documentation was done in the HIIFL (High Intensity Incident Fluorescent Light) microscope developed by the ZOW Munich.

**Results:** There was a fast bone healing observed: after 8 days, the bony ingrowth of press-fit inserted graft cylinders reached 1500  $\mu\text{m}$ , as indicated in the yellow label of oxytetracycline. The osseous integration was complete after 4 weeks. During the following weeks (6–11) remodelling activities were observed.

**Conclusion:** The primary bone healing of press-fit inserted grafts starts five days after the operation and is complete after 4 weeks. The newly formed bone represents mature lamellar bone formations.

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### Shade of resin-based luting agents: Impact on final porcelain color



A.P. Perroni, C. Amaral, M.R. Kaizer, N. Boscato, R.R. Moraes\*

Federal University of Pelotas, Brazil

**Purpose:** This study investigated the influence of different shades of flowable resin-based composites used as luting agents on the final shade of enamel and/or dentin porcelain veneers.

**Methods and materials:** The porcelain veneers were placed over A2 and B1 simulated dental substrates as backgrounds. One mm monolithic (enamel – E1.0, or dentin D1.0) and bilayer (D0.5E0.5) porcelain discs were paired with flowable composite films (100  $\mu\text{m}$  thickness). The composite films had different shades (A1, A2, B1, WO, or IL). The veneer + luting agent specimens ( $n=10$  per group) had the CIE  $L^*a^*b^*$  individual color coordinates measured with a spectrophotometer. Color measurements were carried out over white and black standard backgrounds, as well as over the A2 and B1 simulated dental substrates. The translucency parameter of all porcelain and luting agent specimens alone or their combinations were measured. CIEDE2000 color variations were calculated to analyze the shade matching of the porcelain-luting agent pairs over

A2 and B1 substrates. Data were analyzed by calculating 95% confidence intervals.

**Results:** For porcelain discs alone, the group E1.0 was considerably more translucent than groups D1.0 and D0.5E0.5, among which the most opaque was D0.5E0.5. Among the luting agent films alone, only IL presented significant higher translucency than the others. For the combinations porcelain + luting agent, groups with E1.0 as porcelain veneer were the most translucent, while groups with D1.0 as veneer had the highest opacity. Within the same porcelain veneer, the WO luting agent showed the lowest translucency, while A1, A2, B1, and IL had minor differences. Results for color variation of the porcelain veneers + luting agents over the A2 simulated dental substrate showed that the porcelain veneer D1.0 had the lowest color variation, indicating better shade matching than the porcelain veneers E1.0 and D0.5E0.5. The overall best shade matching with A2 dental substrate was yielded by the combination D1.0 veneer + WO luting agent, while the poorest shade matching was observed for E1.0 veneer, with little influence of the luting agent. D0.5E0.5 porcelain veneer showed the best shade matching on B1 substrate. The overall best shade matching for substrate B1 was obtained by combination of D0.5E0.5 porcelain and WO luting agent.

**Conclusion:** In conclusion, the combination of porcelain veneer (enamel or dentin) with luting agents of different shades affected the final appearance of the restorations over distinct simulated tooth substrates. Shade selection of the luting agent might impact the shade matching of porcelain for a natural looking restoration.

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### Shrinkage and shrinkage stress control in highly-filled nanogel-modified composite



H. Lu<sup>1,\*</sup>, X. Jin<sup>1</sup>, M. Barros<sup>2</sup>, J.W. Stansbury<sup>2,3</sup>

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<sup>2</sup> University of Colorado School of Dental Medicine, Aurora, USA

<sup>3</sup> University of Colorado Boulder, Department of Chemical & Biological Eng., Boulder, USA

**Purpose:** The importance of minimizing disruption to interfacial bonding between composite and substrate cannot be overstated. Restricted volumetric shrinkage, especially during the rapid photopolymerization irradiated with high-output lamps, may result in excessive shrinkage stress at composite ~ adhesive ~ tooth interphases. This study aims to evaluate the impact of nanogel in experimental universal type composite's mechanical properties, shrinkage, shrinkage stress, & real-time modulus development during photo-curing.

**Methods and materials:** The isobornyl methacrylate (IBMA)/UDMA-based reactive nanogels were prepared and characterized as described previously in studies from the Stansbury lab. After nanogel dispersion in an isosorbide-based urethane dimethacrylate resin matrix at various levels (10–25 wt.%), experimental composites were prepared in double-pleinary mixer under vacuum. Shrinkage stress was measured with ADAF's cantilever-beam based Tensometer

to the recipient bone bed. Revascularization and bone formation depend upon the structure of bed and graft. The question of this study was how the revascularization and time-dependent bone formation can be analyzed under stable press-fit implantation.

**Methods and materials:** Contact healing of a cancellous graft was studied in 18 giant rabbits using a standardized animal model of the distal femur. In the center of the patellar groove, a 4.60-mm-large graft cylinder from the contralateral side was inserted into a 4.50-mm-large defect; both were created using a wet-grinding surgical procedure with diamond-coated instruments. The press fit was based on a difference in diameter of 1/10 of a millimeter. The titer dynamic of bone formation was investigated applying the polychromatic sequential labeling according to Rahn and Perren. The stages were 1, 4, 6, 9 (4 animals) and 11 weeks (2 animals). Non-decalcified histology was performed of a complete sequence of serial cuts perpendicularly to the transplant. The documentation was done in the HIIFL (High Intensity Incident Fluorescent Light) microscope developed by the ZOW Munich.

**Results:** There was a fast bone healing observed: after 8 days, the bony ingrowth of press-fit inserted graft cylinders reached 1500  $\mu\text{m}$ , as indicated in the yellow label of oxytetracycline. The osseous integration was complete after 4 weeks. During the following weeks (6–11) remodelling activities were observed.

**Conclusion:** The primary bone healing of press-fit inserted grafts starts five days after the operation and is complete after 4 weeks. The newly formed bone represents mature lamellar bone formations.

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### Shrinkage and shrinkage stress control in highly-filled nanogel-modified composite



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**Purpose:** The importance of minimizing disruption to interfacial bonding between composite and substrate cannot be overstated. Restricted volumetric shrinkage, especially during the rapid photopolymerization irradiated with high-output lamps, may result in excessive shrinkage stress at composite ~ adhesive ~ tooth interphases. This study aims to evaluate the impact of nanogel in experimental universal type composite's mechanical properties, shrinkage, shrinkage stress, & real-time modulus development during photo-curing.

**Methods and materials:** The isobornyl methacrylate (IBMA)/UDMA-based reactive nanogels were prepared and characterized as described previously in studies from the Stansbury lab. After nanogel dispersion in an isosorbide-based urethane dimethacrylate resin matrix at various levels (10–25 wt.%), experimental composites were prepared in double-planet mixer under vacuum. Shrinkage stress was measured with ADAF's cantilever-beam based Tensometer

( $n=3$ ). The densities of cured and uncured composites were used to calculate polymerization shrinkage by using Helium Pycnometer ( $n=3$ ). Dynamic oscillatory shear tests were conducted on a rheometer equipped with high-pressure mercury lamp and 400–500 nm filter. The real-time storage shear modulus ( $G'$ ) and loss shear modulus ( $G''$ ) evolution of the composites were recorded. Specimens for ISO4049 flexural strength and modulus (25 mm  $\times$  2 mm  $\times$  2 mm) were prepared and cured using a QTH lamp ( $\sim 1050$  mW/cm<sup>2</sup>) ( $n=6$ ). Specimens were stored in DI-water at 37 °C for 1-day before Instron testing at 0.75 mm/min. For 3-point fracture toughness test, a notch ( $\sim 2.4$  mm deep and 0.4 mm wide) was prepared in center of 25 mm  $\times$  5 mm  $\times$  2 mm specimen ( $n=6$ ). Specimens were then fractured on Instron under 3-point bending mode at 0.5 mm/min. Data were analyzed with one-way ANOVA/Tukey's test ( $\alpha=0.05$ ).

**Results:** As shown in the table, in these highly filled experimental composite systems, the incorporation of only  $\sim 2.1$  to 5.6 wt% IBMA/UDMA nanogel in the composite led to substantial reduction of polymerization shrinkage (up to 23%) and particularly shrinkage stress (up to 41%), without any sacrifice of flexural modulus. Slight decrease of flexural strength and fracture toughness are observed when nanogel loading levels exceed 4.3 wt%; however, even for composite with the highest nanogel loading (5.63 wt%), the flexural strength, flexural modulus, and fracture toughness are still comparable or better than leading commercial dental composites. Photo-rheological study also revealed delayed  $G'/G''$  crossover (gelation point) resulted from the nanogel addition.

**Conclusion:** In the highly filled experimental composite systems studied, the reactive nanogel demonstrated quite effective polymerization shrinkage and shrinkage stress mediation, with no or minimum compromise of overall mechanical strengths and modulus.

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### Correlating polymerization stress with C-factor/filler contents at various instrumental compliances



Z. Wang<sup>1</sup>, F.A. Landis<sup>2</sup>, A.A.M. Giuseppetti<sup>3</sup>, M.Y.M. Chiang<sup>1,\*</sup>

<sup>1</sup> Biosystems and Biomaterials Division, National Institute of Standards and Technology, Gaithersburg, USA

<sup>2</sup> Department of Chemistry, Penn State University – York Campus, York, USA

<sup>3</sup> Dr. Anthony Volpe Research Center, American Dental Association Foundation, Gaithersburg, USA

**Purpose:** Using a NIST-developed instrument to investigate complex effects of cavity configuration (C-factor, the configuration factor) on PS and the dependence of PS on filler content of composites under various instrumental compliances

**Methods and materials:** Experimental composites comprised of typical dental resins filled with inorganic fillers were synthesized and tested. The filler was mixed with the resins in 15 wt.% increments to obtain filler contents of 0–75 wt.% (0–53 vol.%). The instrumental compliances ranging from 0.33–2186.1  $\mu\text{m}/\text{N}$  were obtained. Various C-factors for the cylindrical specimen were achieved by adjusting either the diameter or height of the samples. Also, the design of the instrument allows for the desired compliance to be easily altered by varying the composite sample position along the cantilever beam of instrument thereby replicating the compliance of the prepared tooth cavity.

**Results:** The results indicate that, for a general trend, PS decreased with increasing C-factor when measured by the

Experimental composite	Control	EXP-1	EXP-2	EXP-3	EXP-4	EXP-5
Nanogel wt% in resin	0%	10%	15%	20%	22.5%	25%
Nanogel wt% in Composite	0%	2.10%	3.23%	4.30%	4.95%	5.63%
Flexural strength (MPa)	160 $\pm$ 5 <sup>A</sup>	158 $\pm$ 4 <sup>A</sup>	160 $\pm$ 2 <sup>A</sup>	145 $\pm$ 4 <sup>B</sup>	138 $\pm$ 5 <sup>B</sup>	138 $\pm$ 6 <sup>B</sup>
Flexural modulus (MPa)	12335 $\pm$ 212 <sup>A</sup>	12663 $\pm$ 268 <sup>A</sup>	12728 $\pm$ 394 <sup>A</sup>	11760 $\pm$ 324 <sup>A</sup>	11693 $\pm$ 301 <sup>A</sup>	11967 $\pm$ 1286 <sup>A</sup>
3-Point fracture toughness (MPa $\cdot\text{m}^{1/2}$ )	2.58 $\pm$ 0.09 <sup>A</sup>	2.53 $\pm$ 0.04 <sup>A</sup>	2.55 $\pm$ 0.08 <sup>A</sup>	2.24 $\pm$ 0.0 <sup>C</sup>	2.38 $\pm$ 0.09 <sup>B</sup>	2.27 $\pm$ 0.05 <sup>B,C</sup>
Shrinkage stress (MPa)	2.70 $\pm$ 0.13 <sup>A</sup>	2.21 $\pm$ 0.02 <sup>B</sup>	2.02 $\pm$ 0.0 <sup>C</sup>	1.79 $\pm$ 0.04 <sup>D</sup>	1.75 $\pm$ 0.01 <sup>D,E</sup>	1.59 $\pm$ 0.09 <sup>E</sup>
Shrinkage stress reduction	N/A	18%	25%	34%	35%	41%
Volume shrinkage (%)	2.70 $\pm$ 0.11 <sup>A</sup>	2.44 $\pm$ 0.07 <sup>B</sup>	2.45 $\pm$ 0.03 <sup>B</sup>	2.11 $\pm$ 0.0 <sup>C</sup>	2.07 $\pm$ 0.1 <sup>C</sup>	2.09 $\pm$ 0.1 <sup>C</sup>
Volume shrinkage reduction	N/A	10%	10%	22%	23%	23%

Within each row, results not connected by same letter are significantly different ( $\alpha=0.05$ ).

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instrument set at high instrumental compliance. However, this trend gradually diminished and eventually reversed (PS increased with increasing C-factor) by decreasing the compliance. Also, the results show that the effect of filler content on PS highly depends upon the instrumental compliance. Within the clinic-relevant range of compliances tested, the PS increased with increasing filler content when measured in low compliance region of the instrument. In an intermediate instrumental compliance range, the measured PS was essentially independent of the filler content, while at



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**Conclusion:** The study not only provides a full picture of the PS/C-factor and PS/filler content correlations but also explains the inconsistent trends reported in the literature. From the perspective of clinical significance, our study demonstrates that the current concept of C-factor in dentistry, i.e. higher C-factor produces higher PS due to reduced flow capacity of the confined materials, can be misleading. Instead, the influence of compliance relevant to specific clinical cavity on the relationship of C-factor and PS should be considered. The compliance-dependent effect of filler content on PS suggests: (1) for materials comparison in terms of PS, the specific compliance at which the comparison being done should always be reported and (2) clinically, composites with relatively lower filler content could be selected for such cavities with relatively lower compliance (e.g. a Class I cavity with thick tooth walls or the basal part in a cavity) and vice versa in order to reduce the final PS.

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### Novel powder-liquid type high-performance PMMA/MMA based resin



J. Tanaka<sup>1,\*</sup>, M. Irie<sup>1</sup>, J.W. Stansbury<sup>2</sup>

<sup>1</sup> Okayama University, Japan

<sup>2</sup> University of Colorado, USA

**Purpose:** This investigation seeks to evaluate the characteristic mechanical properties of a novel powder-liquid type denture base material using the elastic paste prepared by the mixing of poly(methyl methacrylate) (PMMA) powder and liquid monomeric mixture of methyl methacrylate (MMA) and 1,1,1-trimethylolpropane trimethacrylate (TMPTMA).

**Methods and materials:** The elastic paste was prepared by the mixing of PMMA powder (M.W.:  $5 \times 10^5$ , Particle size: 40–80  $\mu\text{m}$ ) and MMA-TMPTMA liquid at the P/L ratio of 2.0 (g/ml) and allowing MMA-TMPTMA to react physically with PMMA in a sealed jar until a doughy consistency. The dough was then packed into a Teflon mold ( $2 \times 2 \times 25$  mm). The open surfaces were covered with Mylar sheets and slide glasses, clamped and thermo-polymerized with a dry heat curing unit (ST-110B1, Espec; 65 °C/60 min + 100 °C/90 min). The flexural properties were measured in a universal testing machine (Instron 5544) in three-point bend testing (span: 20 mm, cross-head speed: 0.5 mm/min) after 24 h storage in air ( $n=6$ ). The conversion of the MMA-TMPTMA mixture in the paste was determined based on the methacrylate group concentration by near-infrared spectrometry (PerkinElmer Spectrum 2000).

**Results:** The flexural properties of powder-liquid type PMMA/MMA-TMPTMA resin depended on the fraction of TMPTMA with the highest flexural strength ( $147 \pm 4$  MPa) in the formulation with 40 vol% TMPTMA. About 2.5 in three methacryloyl groups of TMPTMA were polymerized in MMA-TMPTMA (40) based on the total conversion of 89%. These characteristic flexural properties of PMMA/MMA-TMPTMA

resin are associated with the semi-interpenetrating polymer network structure, where cross-linked density changes continuously for, the polymer alloy layer composed of poly(MMA-co-TMPTMA) with extremely high density cross-linked network structure and PMMA near the surface of the original PMMA powder particles.

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### Evaluation of wear resistance of coating materials on GI restorative



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**Results:** The wear depth of each coating materials are shown in the table. It was suggested that EQUIA Forte coat forms the thinnest layer and has the highest wear resistance than other coating materials. However, Scotchbond Universal was completely worn. And Ketac Glaze formed the thickest layer.

**Conclusion:** This research indicated that EQUIA Forte coat will form thin layer and have high wear resistance. It will meet the requirement for coating material. In clinical case, EQUIA Forte coat is superior to other coating materials.

Wear depth [micrometer], (S.D.)

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**Conclusion:** The study not only provides a full picture of the PS/C-factor and PS/filler content correlations but also explains the inconsistent trends reported in the literature. From the perspective of clinical significance, our study demonstrates that the current concept of C-factor in dentistry, i.e. higher C-factor produces higher PS due to reduced flow capacity of the confined materials, can be misleading. Instead, the influence of compliance relevant to specific clinical cavity on the relationship of C-factor and PS should be considered. The compliance-dependent effect of filler content on PS suggests: (1) for materials comparison in terms of PS, the specific compliance at which the comparison being done should always be reported and (2) clinically, composites with relatively lower filler content could be selected for such cavities with relatively lower compliance (e.g. a Class I cavity with thick tooth walls or the basal part in a cavity) and vice versa in order to reduce the final PS.

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	Initial thickness	Wear depth
EQF	31.67 (3.81)	11.00 (3.61)
KMS	39.33 (1.15)*	39.33 (1.15)**
KMG	92.67 (6.81)**	46.67 (7.37)**

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### Effects of light-curing method and bonding on different-type composite/wall adaptation



T. Yoshikawa\*, M. Morigami, A. Sadr

Tokyo Medical and Dental University, Tokyo, Japan

**Purpose:** The aim of this study was to evaluate the effects of the light curing method and bonding system on different type resin composite adaptation to the cavity wall.

**Methods and materials:** Cylindrical cavities were prepared on the buccal cervical regions. The cavities were restored using Clearfil tri-S Bond ND Quick (Kuraray Noritake Dental) or Clearfil Mega Bond (Kuraray Noritake Dental) adhesive system and filled with Clearfil AP-X (shade A3: Kuraray Noritake Dental) or Clearfil Photo Bright (shade US: Kuraray Noritake Dental) resin composite. The shades of resin composites used in this study, A3, correspond to Vita classical shade. Clearfil AP-X has decreased contrast ratio (opacity) and Clearfil Photo Bright has increased contrast ratio (opacity) during polymerization. The resins were cured using the conventional light curing method (600 mW/cm<sup>2</sup> for 40 s) or slow-start light curing method (270 mW/cm<sup>2</sup> for 10 s + 5 s interval + 600 mW/cm<sup>2</sup> for 30 s). After thermal cycling, dye penetration tests around the cavity wall on cut surface were carried out. Dye penetration length was calculated as a percentage of the total cavity wall length. Data (n = 6) were analyzed using Mann-Whitney U test and Kruskal-Wallis test.

**Results:** Result of Clearfil Mega bond was significantly improved resin composite adaptation to the cavity wall compared with that of Clearfil tri-S Bond for both resin composites (p < 0.05). The slow-start curing method resulted in significantly improved resin composite adaptation to the cavity wall compared with the conventional method for Clearfil Photo Bright resin composites (p < 0.05) (Table 1).

**Table 1 – Cavity-wall gap formation (%: mean (SD)).**

Light curing method	Clearfil AP-X tri-S Bond		Clearfil Photo Bright tri-S Bond	
	ND Quick	Mega Bond	ND Quick	Mega Bond
600 mW/cm <sup>2</sup> 40 s	57.1 (13.0) <sup>A,B</sup>	17.0 (12.6) <sup>A</sup>	25.7 (8.4) <sup>a,B</sup>	8.9 (7.8) <sup>a,B</sup>
270 mW/cm <sup>2</sup> 10 s + 5-s interval + 600 mW/cm <sup>2</sup> 30 s	41.8 (7.4) <sup>C,D,E</sup>	4.1 (7.6) <sup>C</sup>	1.2 (2.9) <sup>a,D</sup>	0 <sup>a,E</sup>

Intergroup data designated with same superscript small letters for each light curing method are significantly different (p < 0.05). Intergroup data designated with same superscript capital letters for each resin composite are significantly different (p < 0.05).

**Conclusion:** Clearfil Mega Bond with the light-cured resin composite, which had increased contrast ratio during polymerization, showed complete cavity-wall adaptation when using the slow-start curing method. Supported by Grant #22592115; #25462950 from JSPS and GCOE Program, FRMDRTB.

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### Influence of fibre-reinforcement on light-transmittance and microhardness of bulk-fill resin-composites



A. Al-Haddad\*, J. Satterthwaite, N. Silikas

University of Manchester, UK

**Purpose:** (i) To measure the top and bottom initial and 48 h post-cure Vickers hardness (VH) for one bulk-fill resin-composite after incorporating different reinforcing fibres, and compare the bottom/top hardness ratio among them, and (ii) to investigate the effect of fibre reinforcement on the penetration of curing light indicated by irradiance (IR) and total energy (ET) values delivered to specimens' bottom surface.

**Methods and materials:** Fifty rectangular-shaped specimens (5 × 5 × 3 mm), representing five main groups (n = 10), were made from a bulk-fill resin-composite (X-tra fill/Voco) and reinforced with four different reinforcing fibres. No fibres was used to reinforce Group C (control), while pre-impregnated (EverstickNet/Stick-Tech) and non-impregnated (StickNet/Stick-Tech) bidirectional E-glass fibres, plasma-treated woven polyethylene fibres (Constuct/Kerr) and unidirectional E-glass fibres (Stick/Stick-Tech) were incorporated to reinforce groups (EN, SN, P, S) respectively. An LED light (Elipar<sup>TM</sup>S10/3M-ESPE) was utilized to cure all specimens (20 s) from the top surface only. Microhardness tester (FM-700, Kawasaki, Japan) was used (300 g load, 10 s dwell) to measure Vickers hardness number (VHN) of top and bottom surfaces immediately post-cure for half of specimens, while the remaining was tested after water storage (48 h/37 °C). Three measurements per surface were taken, and the bottom/top ratio was calculated. Laboratory spectrometer (MARC Resin Calibrator, Blue-Light Analytics) was employed to measure mean IR (mW/cm<sup>2</sup>) and ET (J/cm<sup>2</sup>) delivered to specimens' bottom surface during curing. Series of independent/paired t-test and one-way ANOVA (Tukey's Post-hoc) were used to detect any significant difference among the groups (α = 0.05).

**Results:** Group C had the lowest top-bottom initial (64.4–63.3) and post-cure (75.2–73.1) VHN but the highest bottom/top ratio (98.5%), IR (207) and ET (4.2). For fibre-reinforced groups, Group SN had the highest top-bottom VHN (76.8–74.7), bottom/top ratio (97.2%), IR (190) and ET (3.9) while Group S had the lowest values (72.4–67.7, 93.5%, 183, 3.7 respectively). Fibre-reinforcement significantly improved top-bottom VHN but reduced bottom/top ratio, IR and ET delivered to the bottom surface. Storage condition significantly enhanced microhardness for all groups.

**Conclusion:** Incorporating reinforcing fibres has the potential to improve microhardness of the bulk-fill resin-composite without distressing its post-cure polymerisation. However, it tends to attenuate light intensity and transmittance to bottom

	Initial thickness	Wear depth
EQF	31.67 (3.81)	11.00 (3.61)
KMS	39.33 (1.15)*	39.33 (1.15)**
KMG	92.67 (6.81)**	46.67 (7.37)**

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### Ultra-fast photopolymerization of experimental composites: DEA and FT-NIRS measurement comparison



L.D. Randolph<sup>1</sup>, W.M. Palin<sup>2</sup>, J. Steinhaus<sup>3</sup>, B. Moeginger<sup>3</sup>, G. Leloup<sup>1</sup>, J.G. Leprince<sup>1,\*</sup>

<sup>1</sup> Université Catholique De Louvain, Bruxelles, Belgium

<sup>2</sup> University of Birmingham, UK

<sup>3</sup> Bonn-Rhine-Sieg University of Applied Sciences, Rheinbach, Germany

**Purpose:** Polymerization kinetics of ultra-fast photopolymerizations in model resin composites using a monoacyl phosphine oxide (Lucirin-TPO, MAPO) as a photoinitiator have been shown to proceed substantially faster compared with conventional Camphorquinone/Amine systems (CQ). Monitoring such reactions requires many data points to be collected per second. In the present work we investigated the relevance of combining dielectric analysis (DEA) and near-infrared spectroscopy (FT-NIRS) to monitor polymerizations in MAPO and CQ-based composites, under clinically relevant conditions.

**Methods and materials:** Four experimental resin composites were prepared based on 20/80, 40/60, 60/40 or 80/20 mol% BisGMA/TegDMA resins, using either CQ or MAPO in equimolar concentrations (CQ/DMAEMA (0.20/0.80 wt%) or TPO (0.42 wt%). The resins were filled to 75 wt%. 2 mm thick layers of material were photo-polymerized with spectral outputs specific to each photoinitiator (395–415 nm for MAPO or 455–485 nm for CQ). DEA measured changes in ionic viscosity (Nion) and was measured through the variations in the electric field emitted from a comb-like electrode placed beneath the material (data collection rate, 20 s<sup>-1</sup>). FT-NIRS was performed in transmission mode, by measuring the decrease in absorption peak related to C=C bonds (6165 cm<sup>-1</sup>, data collection rate = 3 s<sup>-1</sup>). In both cases, measurements were carried out in triplicates.

**Results:** The monitoring of kinetics in MAPO 20/80 by FT-NIRS could not be carried out due to an insufficient collection rate, while DEA was not limited. Initial viscosity (N0ion) increased exponentially with BisGMA content (R<sup>2</sup> = 0.99), impacting the maximum rate of change dNmaxion/dt (R<sup>2</sup> = 0.95), in MAPO-composites. The comparatively slower kinetics of CQ-composites (0.04 < R<sub>pmax</sub> < 0.27 s<sup>-1</sup> compared to 0.37–0.51 s<sup>-1</sup>) could be monitored using FT-NIRS, but proved difficult with DEA due to the extensive conversion occurring post gelation where high noise was observed, in contrast with that of MAPO. Final conversion in MAPO-composites was either equal to (20/80) or higher than their CQ counterparts. If there is space, general comparison of DC values for both methods would be useful for the Abstract.

**Conclusion:** FT-NIR spectroscopy and DEA are complementary methods in the measurement of ultra-fast

photo-polymerization kinetics of highly filled resin-composite systems cured in thick layers. The complementary use of DEA and FT-NIRS allowed for a more comprehensive characterization of their curing kinetics, DEA being more adapted to initial stages, while FT-NIRS is more suited after gelation. DEA indirectly informed on the system viscosity and FT-NIRS allowed for the determination of functional group conversion.

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WITHDRAWN

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### 3D metrological information from fatigue fractured composite surfaces



U. Lohbauer\*, C. Itze, R. Belli

University of Erlangen-Nuernberg, Erlangen, Germany

**Purpose:** Fractographic examination of clinically failed restorations is extremely difficult in terms of load history interpretation. Fatigue fractures are hardly to distinguish from fast fractures and so the energy involved in the fracture event is difficult to approximate. Metrology in three dimensions might help to classify fracture surfaces as fatigued or fast fractured events. The aim of the study was to collect relevant parameters (amplitude and hybrid parameters) from differently fractured composite surfaces, to rank their explanatory power in terms of energy involved in the fracture process.

**Methods and materials:** The resin composite GrandioSO (VOCO, Cuxhaven, Germany) was used to manufacture four-point bending specimens according to ISO 4049. The specimens were fractured (FS in [MPa] (SD) at different cross-head speeds: 5 MPa/s @dry, 0.05 MPa/s @dry, 0.05 MPa/s @wet, 104 fatigue cycles at 0.5 Hz @ wet/14 d, staircase method (FFS). All specimens were stored 24 h at 37 °C prior to testing. A non-contact profilometer (CT100, CyberTechnologies, Ingolstadt, Germany) equipped with a confocal white-light spotsensor (vertical res: 20 nm) was used for mapping all fractured surfaces (stepsize x-y: 5 μm). The following parameters were collected from different regions on the fractured surfaces: Sa (Average Roughness [μm]), Sp (Max. Peak Height [μm]), Sv (Max. Valley Depth [μm]), Sku (Kurtosis), Sdp (Root Mean Square (RMS) Surface Slope [1/mm<sup>2</sup>]), Ssc (mean summit curvature [1/mm]), FD (fractal dimension, box counting). ANOVA/S-N-K statistics were applied in order to distinguish at a level of alpha = 0.05.

**Results:** Results are presented in the Table. Letters indicate statistically homogenous subsets within columns (alpha = 0.05).

**Conclusion:** FS decreased with water storage, particularly after cyclic loading (FFS). Amplitude parameters Sa,



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**Purpose:** Polymerization kinetics of ultra-fast photopolymerizations in model resin composites using a monoacyl phosphine oxide (Lucirin-TPO, MAPO) as a photoinitiator have been shown to proceed substantially faster compared with conventional Camphorquinone/Amine systems (CQ). Monitoring such reactions requires many data points to be collected per second. In the present work we investigated the relevance of combining dielectric analysis (DEA) and near-infrared spectroscopy (FT-NIRS) to monitor polymerizations in MAPO and CQ-based composites, under clinically relevant conditions.

**Methods and materials:** Four experimental resin composites were prepared based on 20/80, 40/60, 60/40 or 80/20 mol% BisGMA/TegDMA resins, using either CQ or MAPO in equimolar concentrations (CQ/DMAEMA (0.20/0.80 wt%) or TPO (0.42 wt%). The resins were filled to 75 wt%. 2 mm thick layers of material were photo-polymerized with spectral outputs specific to each photoinitiator (395–415 nm for MAPO or 455–485 nm for CQ). DEA measured changes in ionic viscosity (Nion) and was measured through the variations in the electric field emitted from a comb-like electrode placed beneath the material (data collection rate, 20 s<sup>-1</sup>). FT-NIRS was performed in transmission mode, by measuring the decrease in absorption peak related to C=C bonds (6165 cm<sup>-1</sup>, data collection rate = 3 s<sup>-1</sup>). In both cases, measurements were carried out in triplicates.

**Results:** The monitoring of kinetics in MAPO 20/80 by FT-NIRS could not be carried out due to an insufficient collection rate, while DEA was not limited. Initial viscosity (N0ion) increased exponentially with BisGMA content (R<sup>2</sup> = 0.99), impacting the maximum rate of change dNmaxion/dt (R<sup>2</sup> = 0.95), in MAPO-composites. The comparatively slower kinetics of CQ-composites (0.04 < R<sub>pmax</sub> < 0.27 s<sup>-1</sup> compared to 0.37–0.51 s<sup>-1</sup>) could be monitored using FT-NIRS, but proved difficult with DEA due to the extensive conversion occurring post gelation where high noise was observed, in contrast with that of MAPO. Final conversion in MAPO-composites was either equal to (20/80) or higher than their CQ counterparts. If there is space, general comparison of DC values for both methods would be useful for the Abstract.

**Conclusion:** FT-NIR spectroscopy and DEA are complementary methods in the measurement of ultra-fast

photo-polymerization kinetics of highly filled resin-composite systems cured in thick layers. The complementary use of DEA and FT-NIRS allowed for a more comprehensive characterization of their curing kinetics, DEA being more adapted to initial stages, while FT-NIRS is more suited after gelation. DEA indirectly informed on the system viscosity and FT-NIRS allowed for the determination of functional group conversion.

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WITHDRAWN

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### 3D metrological information from fatigue fractured composite surfaces



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**Purpose:** Fractographic examination of clinically failed restorations is extremely difficult in terms of load history interpretation. Fatigue fractures are hardly to distinguish from fast fractures and so the energy involved in the fracture event is difficult to approximate. Metrology in three dimensions might help to classify fracture surfaces as fatigued or fast fractured events. The aim of the study was to collect relevant parameters (amplitude and hybrid parameters) from differently fractured composite surfaces, to rank their explanatory power in terms of energy involved in the fracture process.

**Methods and materials:** The resin composite GrandioSO (VOCO, Cuxhaven, Germany) was used to manufacture four-point bending specimens according to ISO 4049. The specimens were fractured (FS in [MPa] (SD) at different cross-head speeds: 5 MPa/s @dry, 0.05 MPa/s @dry, 0.05 MPa/s @wet, 104 fatigue cycles at 0.5 Hz @ wet/14 d, staircase method (FFS). All specimens were stored 24 h at 37 °C prior to testing. A non-contact profilometer (CT100, CyberTechnologies, Ingolstadt, Germany) equipped with a confocal white-light spotsensor (vertical res: 20 nm) was used for mapping all fractured surfaces (stepsize x-y: 5 μm). The following parameters were collected from different regions on the fractured surfaces: Sa (Average Roughness [μm]), Sp (Max. Peak Height [μm]), Sv (Max. Valley Depth [μm]), Sku (Kurtosis), Sdp (Root Mean Square (RMS) Surface Slope [1/mm<sup>2</sup>]), Ssc (mean summit curvature [1/mm]), FD (fractal dimension, box counting). ANOVA/S-N-K statistics were applied in order to distinguish at a level of alpha = 0.05.

**Results:** Results are presented in the Table. Letters indicate statistically homogenous subsets within columns (alpha = 0.05).

**Conclusion:** FS decreased with water storage, particularly after cyclic loading (FFS). Amplitude parameters Sa,

Sp, Sv correlate well with FS while Sku, Sdq, Ssc, and FD seem not to reflect the amount of energy released during fracture. Sdp however, indicates a smoother surface texture for wet storage. Ssc, a useful indicator for elastic/plastic deformation, shows a more brittle morphology upon dry storage.

the shear stress concentration at the adhesive interface was recorded in 30 MPa at lingual cervical area and in 20 MPa at buccal cervical area. On the other hand, when load was applied from lingual to buccal at buccal cusp, the interfacial shear stress concentration increased (50 MPa at buccal cervical area

Test conditions	FS/FFS	FD	S <sub>a</sub>	S <sub>p</sub>	S <sub>v</sub>	S <sub>ku</sub>	S <sub>dq</sub>	S <sub>sc</sub>
5MPa/s@dry	131.3 <sup>a</sup> (26.5)	2.081 <sup>b,c</sup> (0.05)	1.25 <sup>d</sup> (0.4)	12.56 (6.0)	10.35 <sup>f</sup> (5.4)	6.0 <sup>h</sup> (2.9)	0.26 <sup>k</sup> (0.1)	105.9 <sup>m</sup> (29.1)
0.05MPa/s@dry	125.6 <sup>a</sup> (13.5)	2.049 <sup>b,c</sup> (0.05)	1.23 <sup>d</sup> (0.2)	10.1 (2.5)	8.73 <sup>f</sup> (2.5)	4.64 <sup>j</sup> (1.0)	0.27 <sup>k</sup> (0.1)	117.2 <sup>m</sup> (27.6)
0.05MPa/s@wet	101.7 (15.4)	2.024 <sup>b</sup> (0.19)	0.90 (0.3)	7.31 <sup>e</sup> (2.3)	6.94 <sup>g</sup> (2.7)	4.40 <sup>j</sup> (1.5)	0.21 <sup>l</sup> (0.0)	96.9 <sup>n</sup> (18.0)
FFS@wet	63.2 (12.2)	2.109 <sup>c</sup> (0.085)	0.61 (0.1)	6.90 <sup>e</sup> (2.5)	5.78 <sup>g</sup> (1.4)	4.94 <sup>h,j</sup> (2.3)	0.19 <sup>l</sup> (0.0)	89.5 <sup>n</sup> (19.9)

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### Stress distribution in resin-core build-up tooth under different load directions



T. Hatayama<sup>1,\*</sup>, M. Nakajima<sup>1</sup>, K. Hosaka<sup>1</sup>, K. Kainose<sup>1</sup>, N. Wakabayashi<sup>2</sup>, J. Tagami<sup>1</sup>

<sup>1</sup> Cariology and Operative Dentistry, Tokyo Medical and Dental University, Japan

<sup>2</sup> Removable Partial Prosthodontics, Tokyo Medical and Dental University, Japan

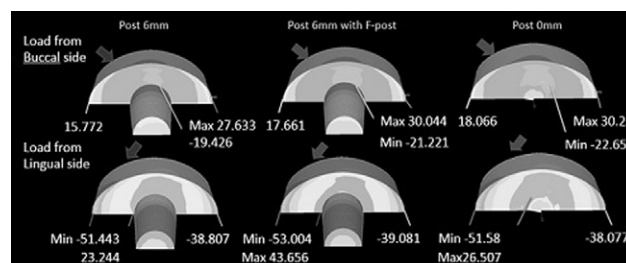
**Purpose:** Stress concentration at adhesive interface would cause fracture of resin-dentin bond in resin-core build-up tooth, leading to destruction of restorative material and/or restored tooth. The purpose of this study was to investigate shear stress distribution at adhesive interface in endodontically-treated tooth restored with resin core with or without fiber post under different load direction.

**Methods and materials:** Finite element models consisted of a mandibular second premolar tooth with root filling, periodontal ligament and surrounding alveolar bone. The mandibular bone was modelled as a cancellous block with 2.0 mm-thick cortical bone. The post preparation was modelled with a simplified circular external cross section 2.0 mm in diameter in the root. Part of the tooth structure was replaced with a full coverage ceramic crown without ferrule. Resin-core was designed with no post preparation and 6.0 mm post preparation embedding with or without a fiber post. Each model had mesiodistal symmetrical boundary conditions applied and was meshed by approximately 100000 hexahedral elements determined by preliminary convergence tests (ANSYS 11.0; ANSYS Inc., Cononsburg, PA, USA). All materials were assumed to be perfectly bonded each other. A total axial load of 400 N was applied to the tip of buccal cusp 45° obliquely from buccal to lingual or from lingual to buccal. The shear stress distribution at the interface between post&core and dentin were calculated for all simulations.

**Results:** (1) Interfacial stress in core part at the cervical area: When load was applied from buccal to lingual at buccal cusp,

and 40 MPa at lingual cervical area). Resin build-up method did not affect these shear stress concentrations in adhesive interface at the cervical area. (2) Interfacial stress in post part: There were no differences in the shear stress distribution in adhesive interface at the post area between the load directions.

**Conclusion:** Load from lingual to buccal at buccal cusp caused a higher concentration of shear stress at cervical area than that from buccal to lingual at buccal cusp. Post preparation method could not reduce interfacial stress concentration at the cervical area.



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### DMSO improves long-term dentin bonding of etch-and-rinse and self-etch adhesives



T.H.S. Stape<sup>1</sup>, L. Tjäderhane<sup>2,\*</sup>, A. Szesz<sup>3</sup>, A.D. Loguercio<sup>3</sup>, L.R.M. Martins<sup>1</sup>

<sup>1</sup> Piracicaba Dental School, University of Campinas, Brazil

<sup>2</sup> Institute of Dentistry, University of Oulu, Oulu, Finland

<sup>3</sup> School of Dentistry, University Estadual De Ponta Grossa, Ponta Grossa, Brazil

**Purpose:** Dimethyl sulfoxide (DMSO) is a solvent that has been shown to improve the bond strength and reduce nanoleakage with 2-step etch-and-rinse (2-ER) adhesive, and improve the adhesive penetration with 3-ER and 2-step self-etch (2-SE) adhesives. The aim of this study was to examine

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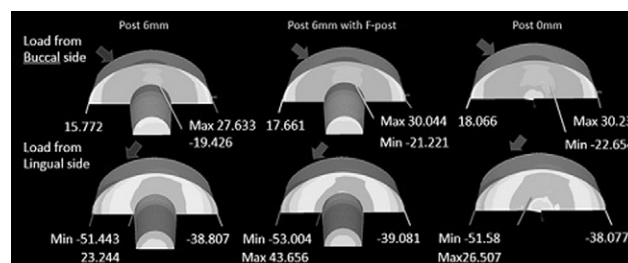
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**Results:** (1) Interfacial stress in core part at the cervical area: When load was applied from buccal to lingual at buccal cusp,

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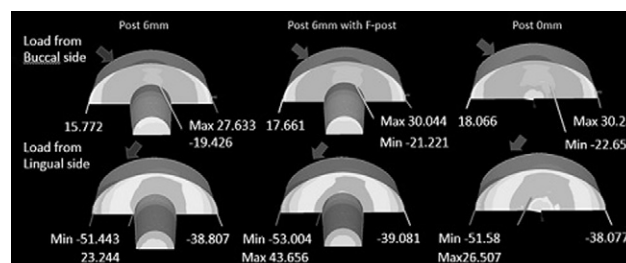
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**Results:** (1) Interfacial stress in core part at the cervical area: When load was applied from buccal to lingual at buccal cusp,

and 40 MPa at lingual cervical area). Resin build-up method did not affect these shear stress concentrations in adhesive interface at the cervical area. (2) Interfacial stress in post part: There were no differences in the shear stress distribution in adhesive interface at the post area between the load directions.

**Conclusion:** Load from lingual to buccal at buccal cusp caused a higher concentration of shear stress at cervical area than that from buccal to lingual at buccal cusp. Post preparation method could not reduce interfacial stress concentration at the cervical area.



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### DMSO improves long-term dentin bonding of etch-and-rinse and self-etch adhesives



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**Purpose:** Dimethyl sulfoxide (DMSO) is a solvent that has been shown to improve the bond strength and reduce nanoleakage with 2-step etch-and-rinse (2-ER) adhesive, and improve the adhesive penetration with 3-ER and 2-step self-etch (2-SE) adhesives. The aim of this study was to examine

the effect of DMSO dentin pretreatment on the immediate and long-term bonding of 3-ER and 2-SE adhesives.

**Methods and materials:** A standardized smear layer was created on 40 human middle-dentin surfaces. Specimens were assigned to the following groups ( $n=10/\text{group}$ ): Scotch-bond MultiPurpose (SBMP: 3M ESPE); SBMP + DMSO; Clearfil SE Bond (CSE: Kuraray); CSE + DMSO. In SBMP + DMSO, dentin was pretreated with 50  $\mu\text{L}$  of 50% DMSO (Sigma-Aldrich) over acid-etched dentin, and in CSE + DMSO DMSO was applied over smear-covered dentin. The controls were treated with water. Otherwise, adhesive procedures were performed according to manufacturers' instructions. Composite build-up was done with Z250 (3M ESPE). Dentin beams were cut and used for the following analysis: degree of conversion (DC) inside the hybrid layer with micro-Raman spectrometer after 24 h; and microtensile bond strength ( $\mu\text{TBS}$ : MPa), fracturographic analysis and interfacial nanoleakage evaluation with  $\text{AgNO}_3$  nanoparticles (% of leakage of the total sample width) after 24 h and 2 years storage in artificial saliva. Statistical analysis was performed with one-way ANOVA (DC) or Repeated Measures ANOVA ( $\mu\text{TBS}$ ) followed by Tukey's test, or with Wilcoxon Signed Ranks test and Kruskal-Wallis with Mann-Whitney test (fracture analysis and nanoleakage), depending whether or not the data followed normal distribution.

**Results:** 50% DMSO pretreatment significantly increased DC with CSE ( $p<0.05$ ) but not with SBMP. DMSO increased 24 h  $\mu\text{TBS}$  significantly with SBMP ( $p<0.05$ ) but not with CSE. After 2 years,  $\mu\text{TBS}$  was significantly lower than 24 h values with both adhesives without DMSO ( $p<0.05$ ). 2-year  $\mu\text{TBS}$  was significantly higher with CSE + DMSO ( $p<0.05$ ), but not with SBMP + DMSO when compared to respective 24 h values; and both adhesives had significantly higher 2-year  $\mu\text{TBS}$  values than respective groups without DMSO ( $p<0.05$ ). At 24 h, there were no differences between the groups in adhesive failure rates, and CSE and CSE + DMSO had significantly less nanoleakage than SBMP and SBMP + DMSO ( $p<0.01$ ) (Table 1). In all groups, adhesive fractures and nanoleakage increased significantly after 2 years in storage ( $p<0.01$ ). However, at 2

years DMSO significantly reduced adhesive fractures with both adhesives, and nanoleakage with SBMP ( $p<0.01$ ; Table 1).

**Conclusion:** 50% DMSO pretreatment improves the hybrid layer quality and durability of both 3-ER and 2-SE adhesives.

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### Effect of silane on bond strength to lithium disilicate ceramic



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Univeridade Federal De Santa Catarina, Brazil

**Purpose:** To evaluate the effect of silane on the microtensile bond strength ( $\mu\text{TBS}$ ) of lithium disilicate glass-ceramic to self-adhesive and conventional resin cements.

**Methods and materials:** Eight ceramic blocks –11 mm long  $\times$  9 mm wide  $\times$  4 mm thick- (IPS e.max Press, Ivoclar Vivadent AG, Schann, Liechtenstein) were fabricated and divided into 4 groups ( $n=2$ ), according to silane application and cement tested. All ceramics surfaces were etched with hydrofluoric acid 10% (Condac, FGM, SC, Brazil) for 20s, rinsed and air-dried. Then, in groups 1 and 3 one layer of silane coupling agent (Monobond S, Ivoclar Vivadent AG, Schann, Liechtenstein) was applied for 60s and air-dried. Composite resin blocks were fabricated with same dimensions of ceramic blocks (Filtek Z350, 3M ESPE, St. Paul, MN, USA) and cemented to them. Self-adhesive resin cement (SpeedCem, Ivoclar Vivadent AG, Schann, Liechtenstein) was utilized in groups 1 and 2, and conventional etch-and-rinse resin cement (Variolink II, Ivoclar Vivadent AG, Schann, Liechtenstein) in groups 3 and 4. After 24 h in distilled water at 37 °C, the specimens were sectioned into sticks (0.8 mm<sup>2</sup>) for  $\mu\text{TBS}$ . Data were statistically analyzed with two-way ANOVA ( $p=0.05$ ). Fractured specimens were examined under optical microscopy at 40 $\times$  magnification.

**Results:** Silanization resulted in higher  $\mu\text{TBS}$  compared to groups without silane ( $p<0.05$ ). No significant differences were found between the conventional resin cement and the self-adhesive resin cement ( $p=0.787$ ).

**Conclusion:** Silanization appear to be crucial for resin bonding to a lithium disilicate glass ceramic, regardless of the resin cement used. The self-adhesive resin cement performed as well as the conventional resin cement.

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**Table 1 – Adhesive fractures (% of all fractures) and nanoleakage (mean percentage [SD] of leakage of the whole width of specimen).**

Group	24 h	2 years
Fractures		
SBMP	43.1Aa	74.1Ab
SBMP + DMSO	31.0Aa	35.7Bb
Clearfil	34.5Aa	64.9Ab
Clearfil + DMSO	27.1Aa	39.0Ab
Leakage		
SBMP	21.5 (4.9)Aa	42.7 (9.0)Ab
SBMP + DMSO	15.9 (3.1)Ba	27.0 (6.3)Bb
Clearfil	9.0 (1.7)Ca	21.8 (5.0)BCb
Clearfil + DMSO	8.8 (1.5)Ca	20.1 (3.9)Cb

Different uppercase letters indicate significant difference between the groups within each time point (column). Different lowercase letters indicate significant difference between the time points within each group (row).

the effect of DMSO dentin pretreatment on the immediate and long-term bonding of 3-ER and 2-SE adhesives.

**Methods and materials:** A standardized smear layer was created on 40 human middle-dentin surfaces. Specimens were assigned to the following groups ( $n=10/\text{group}$ ): Scotch-bond MultiPurpose (SBMP: 3M ESPE); SBMP + DMSO; Clearfil SE Bond (CSE: Kuraray); CSE + DMSO. In SBMP + DMSO, dentin was pretreated with 50  $\mu\text{L}$  of 50% DMSO (Sigma–Aldrich) over acid-etched dentin, and in CSE + DMSO DMSO was applied over smear-covered dentin. The controls were treated with water. Otherwise, adhesive procedures were performed according to manufacturers' instructions. Composite build-up was done with Z250 (3M ESPE). Dentin beams were cut and used for the following analysis: degree of conversion (DC) inside the hybrid layer with micro-Raman spectrometer after 24 h; and microtensile bond strength ( $\mu\text{TBS}$ : MPa), fracturographic analysis and interfacial nanoleakage evaluation with  $\text{AgNO}_3$  nanoparticles (% of leakage of the total sample width) after 24 h and 2 years storage in artificial saliva. Statistical analysis was performed with one-way ANOVA (DC) or Repeated Measures ANOVA ( $\mu\text{TBS}$ ) followed by Tukey's test, or with Wilcoxon Signed Ranks test and Kruskal–Wallis with Mann–Whitney test (fracture analysis and nanoleakage), depending whether or not the data followed normal distribution.

**Results:** 50% DMSO pretreatment significantly increased DC with CSE ( $p<0.05$ ) but not with SBMP. DMSO increased 24 h  $\mu\text{TBS}$  significantly with SBMP ( $p<0.05$ ) but not with CSE. After 2 years,  $\mu\text{TBS}$  was significantly lower than 24 h values with both adhesives without DMSO ( $p<0.05$ ). 2-year  $\mu\text{TBS}$  was significantly higher with CSE + DMSO ( $p<0.05$ ), but not with SBMP + DMSO when compared to respective 24 h values; and both adhesives had significantly higher 2-year  $\mu\text{TBS}$  values than respective groups without DMSO ( $p<0.05$ ). At 24 h, there were no differences between the groups in adhesive failure rates, and CSE and CSE + DMSO had significantly less nanoleakage than SBMP and SBMP + DMSO ( $p<0.01$ ) (Table 1). In all groups, adhesive fractures and nanoleakage increased significantly after 2 years in storage ( $p<0.01$ ). However, at 2

years DMSO significantly reduced adhesive fractures with both adhesives, and nanoleakage with SBMP ( $p<0.01$ ; Table 1).

**Conclusion:** 50% DMSO pretreatment improves the hybrid layer quality and durability of both 3-ER and 2-SE adhesives.

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### Effect of silane on bond strength to lithium disilicate ceramic



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**Purpose:** To evaluate the effect of silane on the microtensile bond strength ( $\mu\text{TBS}$ ) of lithium disilicate glass-ceramic to self-adhesive and conventional resin cements.

**Methods and materials:** Eight ceramic blocks –11 mm long  $\times$  9 mm wide  $\times$  4 mm thick- (IPS e.max Press, Ivoclar Vivadent AG, Schann, Liechtenstein) were fabricated and divided into 4 groups ( $n=2$ ), according to silane application and cement tested. All ceramics surfaces were etched with hydrofluoric acid 10% (Condac, FGM, SC, Brazil) for 20s, rinsed and air-dried. Then, in groups 1 and 3 one layer of silane coupling agent (Monobond S, Ivoclar Vivadent AG, Schann, Liechtenstein) was applied for 60s and air-dried. Composite resin blocks were fabricated with same dimensions of ceramic blocks (Filtek Z350, 3M ESPE, St. Paul, MN, USA) and cemented to them. Self-adhesive resin cement (SpeedCem, Ivoclar Vivadent AG, Schann, Liechtenstein) was utilized in groups 1 and 2, and conventional etch-and-rinse resin cement (Variolink II, Ivoclar Vivadent AG, Schann, Liechtenstein) in groups 3 and 4. After 24 h in distilled water at 37 °C, the specimens were sectioned into sticks (0.8 mm<sup>2</sup>) for  $\mu\text{TBS}$ . Data were statistically analyzed with two-way ANOVA ( $p=0.05$ ). Fractured specimens were examined under optical microscopy at 40 $\times$  magnification.

**Results:** Silanization resulted in higher  $\mu\text{TBS}$  compared to groups without silane ( $p<0.05$ ). No significant differences were found between the conventional resin cement and the self-adhesive resin cement ( $p=0.787$ ).

**Conclusion:** Silanization appear to be crucial for resin bonding to a lithium disilicate glass ceramic, regardless of the resin cement used. The self-adhesive resin cement performed as well as the conventional resin cement.

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**Table 1 – Adhesive fractures (% of all fractures) and nanoleakage (mean percentage [SD] of leakage of the whole width of specimen).**

Group	24 h	2 years
Fractures		
SBMP	43.1Aa	74.1Ab
SBMP + DMSO	31.0Aa	35.7Bb
Clearfil	34.5Aa	64.9Ab
Clearfil + DMSO	27.1Aa	39.0Ab
Leakage		
SBMP	21.5 (4.9)Aa	42.7 (9.0)Ab
SBMP + DMSO	15.9 (3.1)Ba	27.0 (6.3)Bb
Clearfil	9.0 (1.7)Ca	21.8 (5.0)BCb
Clearfil + DMSO	8.8 (1.5)Ca	20.1 (3.9)Cb

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### Physical and chemical properties of new endodontic sealers

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**Purpose:** This study aimed to develop and evaluate physical and chemical properties of three experimental root canal sealers based on MTA and resin salicylate.

**Methods and materials:** The experimental cements were composed by one base paste and three different catalyst pastes. The base paste contained the synthesized component 1,3-butylene glycol disalicylate and the bismuth oxide. The 1,3-butylene glycol disalicylate was synthesized by the transesterification reaction of methyl salicylate characterized by Nuclear Magnetic Resonance Spectroscopy and by Fourier Transform Infrared Spectroscopy. Three different catalyst pastes were formulated by changing the amount of MTA and the type of calcium phosphate (hydroxyapatite-HA or dibasic calcium phosphate dehydrate-DCPD) in weight. (G1) Resimpol 8 (RP8) + titanium dioxide (TiO<sub>2</sub>) + MTA; (G2) RP8 + TiO<sub>2</sub> + MTA + HA and (G3) RP8 + TiO<sub>2</sub> + MTA + DCPD. MTA Fillapex (Angelus) was used as control. Some physical properties were analyzed such as radiopacity (aluminum step wedge), solubility and water absorption (percentage weight variation). Data were submitted to statistical analysis using one-way ANOVA with Tukey test ( $p < 0.05$ ).

**Results:** Statistically similar values of radiopacity were found between MTA Fillapex (7.1 mm Al) and the experimental root canal sealers (G1 – 6.7 mm Al, G2 – 6.5 mm Al, G3 – 6.5 mm Al). MTA Fillapex showed the lowest mean values for solubility (19.47%) and water absorption (13.83%). Regarding the experimental materials, the G2 and G3 showed the highest mean values of solubility (28.02% and 26.19%) and water absorption (23.14% and 22.66%). These properties analyzed showed values close to those recommended by the specifications.

**Conclusion:** The experimental sealers tested presented satisfactory physical properties to be used as root-end filling materials.

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### Properties of resin composites containing natural antimicrobial components

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**Purpose:** The objective of this *in vitro* study was to evaluate the effect of the addition of two natural antimicrobial components (A and B), derived from Brazilian propolis, on the flexural strength, flexural modulus and the amount of *Streptococcus mutans* biofilm formation on resin composites.

**Methods and materials:** Two natural antimicrobial components (A and B) were incorporated in different concentrations into experimental resin composites. A was incorporated in concentrations of 1 mM and 5 mM and B at 5 mM, 25 mM and 50 mM. One resin composite with no addition served as the control. All the resin composites were prepared with the same formulations: BISGMA and TEGDMA (equal parts by weight), camphorquinone (0.6 mol%), EDMAB (1.2 mol%), BHT (0.05 wt%), 0.7 μm barium borosilicate glass (65 wt%) and 0.005 μm fumed silica (5 wt%). Six bars (20 mm × 2 mm × 2 mm) were light cured (3 points/20 s each – Valo, Ultradent) and tested in bending on a universal testing machine (Q-Test/MTS System Corp., MN, USA) at 0.5 mm/min after aging for 24 h. Four 10 mm × 2 mm discs were produced in the same way for each group and a biofilm of *S. mutans* was grown on their surface. After five days, the biofilm was collected and processed to calculate the dry weight of the formed biofilm.

**Results:** Table 1.

**Table 1 – Flexural strength (MPa), flexural modulus (GPa) and dry weight (mg).**

	Flexural strength	Flexural Modulus	Dry weight
Control	119.8 (6.6) <sup>a</sup>	4802.0 (230.8) <sup>a</sup>	11.4 (2.2) <sup>a</sup>
A (1 mM)	119.9 (16.4) <sup>a</sup>	4636.2 (831.4) <sup>ab</sup>	8.0 (1.8) <sup>ab</sup>
A (5 mM)	96.8 (18.9) <sup>a</sup>	4562.7 (270.4) <sup>ab</sup>	8.5 (1.6) <sup>ab</sup>
B (5 mM)	115.9 (13.7) <sup>a</sup>	4470 (569.5) <sup>ab</sup>	6.1 (0.5) <sup>b</sup>
B (25 mM)	101.8 (16.5) <sup>a</sup>	4071.4 (176.7) <sup>ab</sup>	7.4 (0.9) <sup>b</sup>
B (50 mM)	96.4 (9.6) <sup>a</sup>	3851.8 (320.3) <sup>b</sup>	8.9 (2.3) <sup>ab</sup>

Means having similar letters are not significantly different.

**Conclusion:** The incorporation of these natural components into composites at the concentrations tested did not reduce the flexural strength, but reduced the modulus for B at 50 mM. The addition of the compounds demonstrated some antibacterial effect, which resulted in a significant reduction in biofilm formation for the B component at 5 and 25 mM.

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### Physical and chemical properties of new endodontic sealers

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**Methods and materials:** Two natural antimicrobial components (A and B) were incorporated in different concentrations into experimental resin composites. A was incorporated in concentrations of 1 mM and 5 mM and B at 5 mM, 25 mM and 50 mM. One resin composite with no addition served as the control. All the resin composites were prepared with the same formulations: BISGMA and TEGDMA (equal parts by weight), camphorquinone (0.6 mol%), EDMAB (1.2 mol%), BHT (0.05 wt%), 0.7  $\mu$ m barium borosilicate glass (65 wt%) and 0.005  $\mu$ m fumed silica (5 wt%). Six bars (20 mm  $\times$  2 mm  $\times$  2 mm) were light cured (3 points/20 s each – Valo, Ultradent) and tested in bending on a universal testing machine (Q-Test/MTS System Corp., MN, USA) at 0.5 mm/min after aging for 24 h. Four 10 mm  $\times$  2 mm discs were produced in the same way for each group and a biofilm of *S. mutans* was grown on their surface. After five days, the biofilm was collected and processed to calculate the dry weight of the formed biofilm.

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### Biaxial flexural strength and light transmission of bulk-fill composite resins



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**Purpose:** The aims of this study were to assess the light transmission (LT) and determine the biaxial flexural strength (BFS) and flexural modulus (FM) at different depths of one regular and four bulk-fill composites.

**Methods and materials:** Eighty human third molar teeth were bonded to composite resin with the Adper Scotch-bond Multipurpose (SBMP), Adper Single Bond 2 (SB2), Clearfil SE Bond (CSE) or Clearfil S3 Bond (CS3) adhesive systems and water stored for 24 h and six months ( $n=10$ ). Three bonded teeth were selected for nanohardness and seven for  $\mu$ TBS. Data were analyzed by two-way ANOVA and Tukey's tests ( $\alpha=0.05$ ). Spearman correlations between nanohardness/modulus and  $\mu$ TBS values were also calculated.

**Results:** The LT decreased as the composite thickness increased for all of the materials, and no significant differences in the LT were observed, although the SDR sample was more translucent than the HER composite. Furthermore, HER was the unique material with lower BFS values at greater depths. Conversely, HER exhibited a higher FM than SDR.

**Conclusion:** The bulk-fill composites investigated exhibited high LT. Although an increase in the composite thickness reduced the LT, the BFS of the bulk-fill composites was not compromised despite the decreased irradiation at greater depths.

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### Effect of inorganic fillers and post-polymerization methods in experimental composites



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**Purpose:** This study evaluated *in-vitro* the influence of different proportions of inorganic fillers and post-polymerization methods on the hardness and roughness of experimental composites used in indirect technique.

**Methods and materials:** Four experimental composites were prepared containing 30 wt.% of organic matrix and

70 wt.% of filler in following proportions: only barium glass particles Ba-Al-Si (C1), barium glass Ba-Al-Si/silica SiO<sub>2</sub> 90:10 (C2), barium glass Ba-Al-Si/silica SiO<sub>2</sub> 80:20 (C3) and barium glass Ba-Al-Si/silica SiO<sub>2</sub> 70:30 (C4). Silicon matrices (5 mm diameter  $\times$  2 mm thick) were fulfilled with experimental composites (C1–C4) and photoactivated using Bluephase-G2 (energy dose-36J). After that, the specimens were subdivided according to the protocol of post-polymerization ( $n=10$ ): control (P0-without post-polymerization process); Beta Visio unit 470 mW/cm<sup>2</sup> for 15 min (P1); 200 ml water for 5 min in a microwave at 500 W (P2). Specimens were stored in deionized water at 37 °C for 24 h, polished with 600-, 800- and 1200-grit SiC papers and 3  $\mu$ m and 1  $\mu$ m diamond paste and cloth and placed in ultrasound for 20 min. Hardness was determined by Knoop indentation at top and bottom surfaces in three locations under load of 50 g for 5 s and the ratio of bottom/top hardness values were calculated. Surface roughness (Ra) analysis was performed at 0.05 mm/s (0.25 mm cut-off) in three locations. Data were submitted to two-way ANOVA and Tukey's test ( $\alpha=5\%$ ).

**Results:** Interaction was observed between experimental composites X post-polymerization ( $p<0.01$ ) in the ratio of bottom/top hardness. The highest ratio of bottom/top hardness value was found for P1 in C1, C2, C3 and C4. C1 showed no significant difference between post-polymerization protocol ( $p>0.01$ ). C2/P1 ( $1.10 \pm 0.20$ ) presented the highest ratio of bottom/top hardness with significant difference from C2/P0 ( $0.86 \pm 0.25$ ). The lowest ratio of bottom/top hardness was found for C3/P0 ( $0.28 \pm 0.06$ ), significantly different from C3/P1 ( $0.95 \pm 0.19$ ) and C3/P2 ( $0.65 \pm 0.34$ ). C4/P1 ( $0.94 \pm 0.06$ ) presented significant difference for C4/P0 ( $0.59 \pm 0.23$ ) and C4/P2 ( $0.64 \pm 0.23$ ). For surface roughness no interaction was observed and post-polymerization indicated significant difference. P0 showed the highest roughness value ( $0.48 \pm 0.54$ ), significantly different from P1 ( $0.26 \pm 0.15$ ) and P2 ( $0.25 \pm 0.12$ ).

**Conclusion:** The different proportions of inorganic fillers and additional polymerization methods influenced the hardness and roughness of experimental composites. The best experimental composite was C1 and C2 and post-polymerization protocol was Beta Visio.

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### Irradiance assessment of damaged dental light guides by spectrometer-based methods



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**Purpose:** Damaged or resin contaminated light guides may interfere on the irradiance delivery compromising the physical properties, optimal polymerization and eventual clinical performance of resin composite restorations. There is a lack of accurate and reliable devices for determining curing light output. The current method for evaluating irradiance, radiometers, has been demonstrated to be inaccurate. In addi-

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70 wt.% of filler in following proportions: only barium glass particles Ba-Al-Si (C1), barium glass Ba-Al-Si/silica SiO<sub>2</sub> 90:10 (C2), barium glass Ba-Al-Si/silica SiO<sub>2</sub> 80:20 (C3) and barium glass Ba-Al-Si/silica SiO<sub>2</sub> 70:30 (C4). Silicon matrices (5 mm diameter  $\times$  2 mm thick) were fulfilled with experimental composites (C1–C4) and photoactivated using Bluephase-G2 (energy dose-36J). After that, the specimens were subdivided according to the protocol of post-polymerization ( $n=10$ ): control (P0-without post-polymerization process); Beta Visio unit 470 mW/cm<sup>2</sup> for 15 min (P1); 200 ml water for 5 min in a microwave at 500 W (P2). Specimens were stored in deionized water at 37 °C for 24 h, polished with 600-, 800- and 1200-grit SiC papers and 3  $\mu$ m and 1  $\mu$ m diamond paste and cloth and placed in ultrasound for 20 min. Hardness was determined by Knoop indentation at top and bottom surfaces in three locations under load of 50 g for 5 s and the ratio of bottom/top hardness values were calculated. Surface roughness (Ra) analysis was performed at 0.05 mm/s (0.25 mm cut-off) in three locations. Data were submitted to two-way ANOVA and Tukey's test ( $\alpha=5\%$ ).

**Results:** Interaction was observed between experimental composites X post-polymerization ( $p<0.01$ ) in the ratio of bottom/top hardness. The highest ratio of bottom/top hardness value was found for P1 in C1, C2, C3 and C4. C1 showed no significant difference between post-polymerization protocol ( $p>0.01$ ). C2/P1 ( $1.10 \pm 0.20$ ) presented the highest ratio of bottom/top hardness with significant difference from C2/P0 ( $0.86 \pm 0.25$ ). The lowest ratio of bottom/top hardness was found for C3/P0 ( $0.28 \pm 0.06$ ), significantly different from C3/P1 ( $0.95 \pm 0.19$ ) and C3/P2 ( $0.65 \pm 0.34$ ). C4/P1 ( $0.94 \pm 0.06$ ) presented significant difference for C4/P0 ( $0.59 \pm 0.23$ ) and C4/P2 ( $0.64 \pm 0.23$ ). For surface roughness no interaction was observed and post-polymerization indicated significant difference. P0 showed the highest roughness value ( $0.48 \pm 0.54$ ), significantly different from P1 ( $0.26 \pm 0.15$ ) and P2 ( $0.25 \pm 0.12$ ).

**Conclusion:** The different proportions of inorganic fillers and additional polymerization methods influenced the hardness and roughness of experimental composites. The best experimental composite was C1 and C2 and post-polymerization protocol was Beta Visio.

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### Irradiance assessment of damaged dental light guides by spectrometer-based methods



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**Purpose:** Damaged or resin contaminated light guides may interfere on the irradiance delivery compromising the physical properties, optimal polymerization and eventual clinical performance of resin composite restorations. There is a lack of accurate and reliable devices for determining curing light output. The current method for evaluating irradiance, radiometers, has been demonstrated to be inaccurate. In addi-

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### Biaxial flexural strength and light transmission of bulk-fill composite resins



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**Purpose:** The aims of this study were to assess the light transmission (LT) and determine the biaxial flexural strength (BFS) and flexural modulus (FM) at different depths of one regular and four bulk-fill composites.

**Methods and materials:** Eighty human third molar teeth were bonded to composite resin with the Adper Scotch-bond Multipurpose (SBMP), Adper Single Bond 2 (SB2), Clearfil SE Bond (CSE) or Clearfil S3 Bond (CS3) adhesive systems and water stored for 24 h and six months ( $n=10$ ). Three bonded teeth were selected for nanohardness and seven for  $\mu$ TBS. Data were analyzed by two-way ANOVA and Tukey's tests ( $\alpha=0.05$ ). Spearman correlations between nanohardness/modulus and  $\mu$ TBS values were also calculated.

**Results:** The LT decreased as the composite thickness increased for all of the materials, and no significant differences in the LT were observed, although the SDR sample was more translucent than the HER composite. Furthermore, HER was the unique material with lower BFS values at greater depths. Conversely, HER exhibited a higher FM than SDR.

**Conclusion:** The bulk-fill composites investigated exhibited high LT. Although an increase in the composite thickness reduced the LT, the BFS of the bulk-fill composites was not compromised despite the decreased irradiation at greater depths.

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### Effect of inorganic fillers and post-polymerization methods in experimental composites



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**Purpose:** This study evaluated *in-vitro* the influence of different proportions of inorganic fillers and post-polymerization methods on the hardness and roughness of experimental composites used in indirect technique.

**Methods and materials:** Four experimental composites were prepared containing 30 wt.% of organic matrix and

70 wt.% of filler in following proportions: only barium glass particles Ba-Al-Si (C1), barium glass Ba-Al-Si/silica SiO<sub>2</sub> 90:10 (C2), barium glass Ba-Al-Si/silica SiO<sub>2</sub> 80:20 (C3) and barium glass Ba-Al-Si/silica SiO<sub>2</sub> 70:30 (C4). Silicon matrices (5 mm diameter  $\times$  2 mm thick) were fulfilled with experimental composites (C1–C4) and photoactivated using Bluephase-G2 (energy dose-36J). After that, the specimens were subdivided according to the protocol of post-polymerization ( $n=10$ ): control (P0-without post-polymerization process); Beta Visio unit 470 mW/cm<sup>2</sup> for 15 min (P1); 200 ml water for 5 min in a microwave at 500 W (P2). Specimens were stored in deionized water at 37 °C for 24 h, polished with 600-, 800- and 1200-grit SiC papers and 3  $\mu$ m and 1  $\mu$ m diamond paste and cloth and placed in ultrasound for 20 min. Hardness was determined by Knoop indentation at top and bottom surfaces in three locations under load of 50 g for 5 s and the ratio of bottom/top hardness values were calculated. Surface roughness (Ra) analysis was performed at 0.05 mm/s (0.25 mm cut-off) in three locations. Data were submitted to two-way ANOVA and Tukey's test ( $\alpha=5\%$ ).

**Results:** Interaction was observed between experimental composites X post-polymerization ( $p<0.01$ ) in the ratio of bottom/top hardness. The highest ratio of bottom/top hardness value was found for P1 in C1, C2, C3 and C4. C1 showed no significant difference between post-polymerization protocol ( $p>0.01$ ). C2/P1 ( $1.10 \pm 0.20$ ) presented the highest ratio of bottom/top hardness with significant difference from C2/P0 ( $0.86 \pm 0.25$ ). The lowest ratio of bottom/top hardness was found for C3/P0 ( $0.28 \pm 0.06$ ), significantly different from C3/P1 ( $0.95 \pm 0.19$ ) and C3/P2 ( $0.65 \pm 0.34$ ). C4/P1 ( $0.94 \pm 0.06$ ) presented significant difference for C4/P0 ( $0.59 \pm 0.23$ ) and C4/P2 ( $0.64 \pm 0.23$ ). For surface roughness no interaction was observed and post-polymerization indicated significant difference. P0 showed the highest roughness value ( $0.48 \pm 0.54$ ), significantly different from P1 ( $0.26 \pm 0.15$ ) and P2 ( $0.25 \pm 0.12$ ).

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### Irradiance assessment of damaged dental light guides by spectrometer-based methods



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tion, the aperture for measuring the light output is undersized in relation to diameter of the light guide. An innovative in-office spectroradiometer was used (checkMARC™, BlueLight Analytics) as it is capable of measuring the total spectral radiant power output required by the photoinitiators used in light cured composite. To investigate the effects of physical damage and tip contamination on the power output using spectrometer-based methods.

**Methods and materials:** Contaminated and/or damaged light guides (same curved tip design,  $\phi \sim 11$  mm) from different dental curing lights ( $n=9$ ) were assembled. A same design tip without damage or contamination was used as control. Using one curing light (Optilux 501, Kerr), the irradiance was recorded using an integrating sphere, laboratory grade thermopile, and checkMARC™. Laboratory grade integrating sphere spectrometer and thermopile systems were used as reference. The irradiance ( $\text{mW}/\text{cm}^2$ ) was calculated by dividing the output power by the area of the light guide tip. Data were submitted to one-way ANOVA.

**Results:** The mean irradiance values for contaminated tips were 494, 449.6 and 482.5, respectively for integrating sphere, laboratory grade thermopile, and checkMARC™. There was no significant difference among the measurement systems ( $p=0.751$ ).

**Conclusion:** Resin contamination and damage to light guides can reduce the irradiance output and energy delivered to composite restorations. An easy-to-use portable device that can be used in a dental office provides data that is similar to that produced by laboratory grade devices.

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### Tensile bond strength of universal adhesives to repair ceramic restoration



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**Purpose:** Recently, one bottle type of bonding systems to repair ceramic restoration, the universal adhesive systems, have been commercialized. In the present study, we evaluated the bond strength of the universal adhesive systems to repair ceramic restorations.

**Methods and materials:** The bond strength of five types of universal adhesive systems (G-Premio bond: GPB, Scotchbond Universal Adhesive: SU, All-Bond Universal: AU, Universal Primer: UP and BeautiBond Multi: BM) and a conventional adhesive system (C&B Repair Kit: RK) were evaluated by using ceramic blocks (GN Ceram Block, GC) in this study. Surfaces of the ceramic blocks were grinded with SiC paper, grit #600, and then ultrasonic treatment was performed 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. The specimens were stored in distilled water at 37 °C. After 24 h, 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 ( $P < 0.05$ ,  $n = 12$ ).

**Results:** The results of TBS to ceramic block were the followings, GPB:  $18.2 \pm 5.6$  MPa, SU:  $14.9 \pm 3.2$  MPa, AU:  $16.4 \pm 3.4$  MPa, UP:  $15.9 \pm 3.3$  MPa, BM:  $14.9 \pm 4.3$  MPa and RK:  $16.4 \pm 3.1$  MPa. There were no significant differences among all of the adhesives tested. The stereoscopic microscope observations showed cohesive failure mode in the groups of GPB, AU, UP and RK. Several specimens in the groups of SU and BM were shown adhesive failure mode.

**Conclusion:** The results in this study suggested that universal adhesives, consisting from one bottle type, had equal tensile bond strength to the conventional adhesive.

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### Uniaxial and biaxial flexural strengths of resin-composite CAD-CAM blocks



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Dankook University, South Korea

**Purpose:** This study compared the flexural strength of resin-composite blocks for dental CAD-CAM by uniaxial (three-point flexure) and biaxial (ball-on-ring) tests.

**Methods and materials:** Four dental resin-composite blocks for CAD-CAM were evaluated: VE (Vital Enamic, Vita Zahnfabrik), LU (Lava Ultimate, 3M ESPE), MD (Mazic Duro, Vericom), CS (Cerasmart, GC). Rectangular bar specimens ( $4.0 \text{ mm} \times 1.4 \text{ mm} \times 18.0 \text{ mm}$ ) were prepared for uniaxial strength by three-point flexural test. As-received blocks were cut and ground lengthwise and polished with diamond paste ( $6 \mu\text{m}$  and  $1 \mu\text{m}$ ) using a surface grinding machine. The opposing faces were highly flat and parallel within 0.01 mm and edges were rounded ( $n=30$ ), except for MD ( $n=24$ ). For biaxial strength test, the blocks first were machined to a cylindrical form by bench lathe, and then were cut in parallel to produce disk specimens ( $14 \text{ mm}$  in diameter  $\times 1.5 \text{ mm}$  in thick) by a high-speed cutting machine (Accutom 50), except for VE ( $12\text{-mm}$  in diameter). The disk specimens were finished by final polishing with  $1\text{-}\mu\text{m}$  diamond paste ( $n=20$ ). The specimens were stored in 37 °C distilled water for 2 days before testing. Three-point flexural strengths were determined with a 14-mm span. Ball-on-ring (BOR) biaxial strength was tested on a 10-mm circular ball-bearing race as support ring by central loading with a ball (3.2 mm). The biaxial flexural strength was calculated by Shetty et al.'s equations (1980). Both tests were conducted with a constant loading rate of 1.0 mm/min using Instron 3344. Uniaxial flexural strength (UFS) and biaxial flexural strength (BFS) between groups was analyzed by ANOVA and Scheffe's test. Strength data for each material were analyzed by 2-parameter Weibull distribution function.

**Results:** Mean UFS  $\pm$  SD and Weibull modulus of specimen groups were as follow: VE ( $140.1 \pm 7.0$ , 24.2), LU ( $159.1 \pm 6.3$ , 30.7), MD ( $144.9 \pm 13.13$ , 13.6), CS ( $165.4 \pm 16.9$ , 11.5). Mean BFS and Weibull modulus of specimen groups were as follow: VE ( $102.0 \pm 7.0$ , 17.5), LU ( $151.9 \pm 19.6$ , 9.2), MD ( $98.7 \pm 16.0$ , 7.4), CS ( $166.8 \pm 15.4$ , 13.0). Statistical analyses showed that UFS or BFS

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**Methods and materials:** The bond strength of five types of universal adhesive systems (G-Premio bond: GPB, Scotchbond Universal Adhesive: SU, All-Bond Universal: AU, Universal Primer: UP and BeautiBond Multi: BM) and a conventional adhesive system (C&B Repair Kit: RK) were evaluated by using ceramic blocks (GN Ceram Block, GC) in this study. Surfaces of the ceramic blocks were grinded with SiC paper, grit #600, and then ultrasonic treatment was performed 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. The specimens were stored in distilled water at 37 °C. After 24 h, tensile bond strength, TBS, was measured by universal testing machine (cross head speed at

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of CS and LU was significantly higher than those of VE and MD ( $P < 0.05$ ). Groups LU/SC or VE/MD showed no significant differences in both strengths. There was significant decrease in Weibull modulus of flexural strength for VE, LU, and MD, when determined by BFS test.

**Conclusion:** There was a high correlation between UFS and BFS of the resin-composite blocks ( $R^2 = 0.96$ ). However, the reliability of flexural strength of resin-composite materials was affected by the testing method applied.

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### Antibacterial effects of self-adhesive resin cements containing chlorhexidine



R. Kitagawa<sup>1,\*</sup>, H. Kitagawa<sup>1</sup>, N. Hirose<sup>1</sup>, S. Yamaguchi<sup>1</sup>, I.M. Mehdawi<sup>2</sup>, M. Hayashi<sup>1</sup>, S. Imazato<sup>1</sup>

<sup>1</sup> Osaka University, Japan

<sup>2</sup> University of Benghazi, Libya

**Purpose:** Since residual bacteria beneath restorations and plaque accumulation around restoration margins may cause secondary caries, luting cements which show antibacterial effects are advantageous. However, commercially available luting cements do not have any antibacterial activities. In this study, the experimental self-adhesive resin cements containing different concentrations of chlorhexidine diacetate (CHX) were prepared, and their antibacterial effects against oral bacteria and CHX-release profile were evaluated.

**Methods and materials:** CHX was added to the commercial dual-cure self-adhesive resin cement (G-CEM LinkAce, GC, Tokyo) to give 0.5, 1, 2, 5, 10 or 15 (wt)% after mixing of two pastes. For testing cured cements, disc-shaped specimens (10 mm diameter, 2 mm thickness) were prepared. Inhibitory effects of uncured and cured cements, as well as after aging by storage in water for 14 days, against *Streptococcus mutans* NCTC10449 and *Enterococcus faecalis* SS497 were examined by agar diffusion tests. Concentrations of CHX released into deionized water from cured specimens were measured using high performance liquid chromatography up to 14 days. The minimum inhibitory/bactericidal concentrations (MIC/MBC) of CHX against two bacteria were determined by micro-dilution methods.

**Results:** Uncured and cured cements containing CHX at 1% or more inhibited both bacteria, showing significantly greater inhibition at 5% or greater than 2% or less (ANOVA, Tukey's HSD test,  $p < 0.05$ ). After aging for 14 days, the experimental cements containing CHX at 5% or more exhibited inhibition while the specimens with 0.5–2% CHX lost antibacterial activity. There were no significant differences in the size of inhibition zones among the specimens with 5–15% CHX except for the case of cured and aged cements against *S. mutans* ( $p > 0.05$ ). All specimens exhibited continuous release of CHX for 14 days, and concentrations of CHX released were increased as the amount of CHX incorporated increased. Release of CHX above the MIC values determined for both bacteria (3.2 µg/ml) was found for the specimens with 5% or greater throughout 14 days period.

**Conclusion:** Incorporation of CHX at 5% or more was effective to provide the self-adhesive resin cement G-CEM LinkAce with antibacterial activity against *S. mutans* and *E. faecalis*. Cavity disinfection and inhibition of bacterial growth around cements for certain period can be expected by the usage of the experimental cements containing CHX.

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### Clinical result of 23% hydrogen peroxide tooth bleaching agent



H. Shiga<sup>\*</sup>, N. Okada, T. Hasebe, R. Yagi, A. Asano, M. Noda

School of Dentistry, Iwate Medical University, Iwate, Japan

**Purpose:** Recently, vital tooth bleaching has been reported good result. However, it is still difficult to predict the relapse of tooth color after the bleaching because there are several differences of the bleaching effect among individual cases. In the present study, we evaluated the bleaching effect and relapse of tooth color when TiON in Office<sup>®</sup> (GC "Ti"), mainly consists of 23% hydrogen peroxide office bleaching agent, was applied to a vital incisor tooth.

**Methods and materials:** This study was performed at Iwate Medical University Hospital with permission by the research ethics committee. Ten patients obtained informed consent attended this study. Maxillary central incisor tooth were bleached using Ti according to the manufacturer's instructions and repeatedly bleached after a week. Tooth color was measured 7 times: before treatment, after bleaching immediately, 2 weeks, 7 days, 3 month, 6 month and 12 month thereafter. The center of labial surface was measured with a dental spectrophotometer Crystaleye<sup>®</sup> (CE100-DC/JP, Olympus). The color changes of the tooth surface were calculated  $\Delta E^*ab$  value on the basis of before treatment. The data were analyzed by One-way ANOVA and Student-Newman-Keuls test at  $P < 0.05$ .

**Results:** At before treatment, the tooth color of ( $L^*$ ,  $a^*$ ,  $b^*$ ) were  $72.5 \pm 3.7$ ,  $1.4 \pm 0.9$ ,  $18.2 \pm 2.9$ ). After the 2nd bleaching, it changed to ( $74.8 \pm 3.0$ ,  $1.0 \pm 0.7$ ,  $13.8 \pm 3.1$ ). There were significant differences in all parameters. After 2 weeks,  $L^*$  was decreased and  $b^*$  was increased slightly. Those changes continued until 12 month and  $b^*$  was significantly difference from 12 month after treatment.  $\Delta E^*ab$  value was  $5.4 \pm 2.6$  after the 2nd bleaching. After 12 month, it was  $4.2 \pm 1.8$ . There was no significant difference between after bleaching and 12 month.

**Conclusion:** The results in the present study suggested that Ti, a 23% hydrogen peroxide office bleaching agent, has enough bleaching effects. Though relapse of color occurred relatively early after treatment, it was very gently and significant bleaching effect continued after a year.

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**Conclusion:** There was a high correlation between UFS and BFS of the resin-composite blocks ( $R^2 = 0.96$ ). However, the reliability of flexural strength of resin-composite materials was affected by the testing method applied.

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### Dental materials impact in the scientific technological development of dentistry

G.S. Lima\*, W.L.O. Rosa, A.F. Silva, E. Piva

Federal University of Pelotas, Brazil



**Purpose:** A systematic review was conducted to analyze the impact of dental materials within the scientific production in dentistry in the last 50 years.

**Methods and materials:** This report followed the Preferred Reporting Items for Systematic Reviews and Meta-Analysis (Prisma) statement. Searches were performed until December 2014 in six databases: MedLine (PubMed), Scopus, Lilacs, Ibecs, BBO and Cochrane Library. Additionally, the Brazilian patent database (INPI, Instituto Nacional de Propriedade Industrial) was screened in order to get an overview of the Brazilian technological development in dental materials field. Two reviewers independently analyzed the documents, in which only studies and patents related to dental materials were included. Data regarding material category, dental specialty, number of documents and patents, filiation countries, number of citations were tabulated and analyzed in Microsoft Office Excel (Microsoft Corporation, Redmond, WA, USA).

**Results:** A total of 111,590 studies and 53 patents were related to dental materials and were included in this review. The majority of dental materials studies were from United States (18%); and Brazil had 8% of affiliation. Most papers published in the last 50 years were related to dental implants, dental alloys, dental porcelain and composite resins. Considering all papers with Brazil as filiation country, patents deposited by this country represent about 0.6% of the scientific production in dental material field.

**Conclusion:** Dental materials science has a substantial impact on the scientific and technological development of dentistry; however it is necessary to improve the relationship between academia and industry to expand the technological development in countries such as Brazil.

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### Resin-modified glass ionomer cements and demineralization progress in vitro

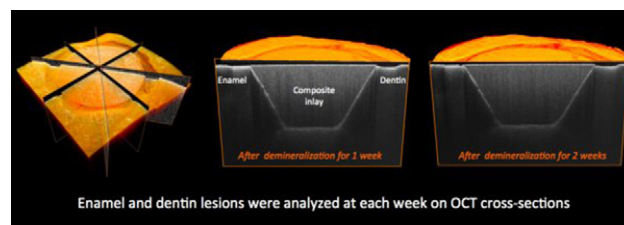
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**Purpose:** To investigate *in vitro* the effect of luting cements in terms of composition and release of calcium and fluoride on demineralization progress around composite resin inlays using optical coherence tomography (OCT).

**Methods and materials:** Round class-I cavities (4 mm diameter; 2 mm depth) were prepared on flat cervical surfaces of bovine incisors. Resin inlays (CLEARFIL MAJESTY Posterior, MP, Kuraray Noritake Dental) were cemented to the cavities using resin-modified glass ionomer cement (RM-GIC) Vitremer Luting Cement (VT, 3M ESPE), POs-Ca-containing RM-GIC Adshield RM (AD, Kuraray Noritake Dental) or self-adhesive resin cement RelyXUnicem 2 (RX, 3M ESPE). Control group was restored with MP only ( $n=3$ ). After 3-day storage in artificial saliva and 10,000 thermal cycles, specimens were demineralized using acidic gel (pH = 4.5) for 2 weeks. After each week, fixed four cross-sectional images were obtained from each restoration using swept-source OCT (Santec) at 1310 nm wavelength. Cross-sectional areas of tissue loss formed due to demineralization at enamel and dentin margins were measured at 8 locations on cross-sectional images. Lesion size was compared by one-way ANOVA with Bonferroni correction at significance level of 0.05.

**Results:** Lesion formation was significantly different among materials ( $p < 0.05$ ) for both enamel and dentin. In both substrates, the largest lesions were found in Control group followed by the resin cement, RX. The lesion progress around VT and AD was significantly slower than the other two groups ( $p < 0.05$ ).

**Conclusion:** In addition to improved interfacial sealing, calcium and fluoride released from luting cements influence lesion formation and progress around indirect restorations increasing their durability. OCT would be a promising clinical tool for evaluation and monitoring of resin inlay margins.



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### Surface morphology and corrosion evaluation of anodized titanium oxides

R.S. Williamson\*, S. Jain, M.D. Roach

University of Mississippi Medical Center, Biomedical Materials Science, Jackson, USA



**Purpose:** Anatase (A) and Rutile (R) phases of titanium oxide have been reported to increase antimicrobial effects and enhance bioactivity levels. However, an A/R phase ratio which may have the greatest effects has not been specified. A previous study in our laboratories produced specific A/R phase ratios through the use of different acid electrolyte mixtures and a stepped potentiostatic forming voltage. The purpose of this study was to evaluate the crystalline formation, surface morphology, wetting angle and corrosion resistance of anodized layers at each voltage step in acid electrolyte mixtures.

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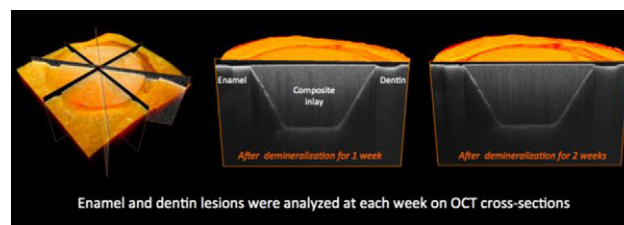
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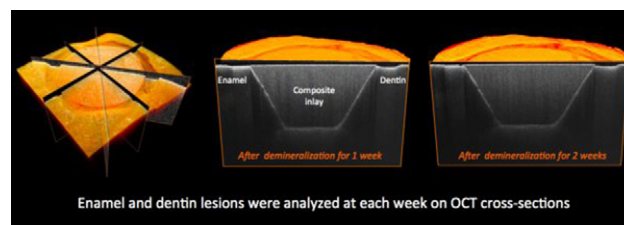
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**Results:** Lesion formation was significantly different among materials ( $p < 0.05$ ) for both enamel and dentin. In both substrates, the largest lesions were found in Control group followed by the resin cement, RX. The lesion progress around VT and AD was significantly slower than the other two groups ( $p < 0.05$ ).

**Conclusion:** In addition to improved interfacial sealing, calcium and fluoride released from luting cements influence lesion formation and progress around indirect restorations increasing their durability. OCT would be a promising clinical tool for evaluation and monitoring of resin inlay margins.



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### Surface morphology and corrosion evaluation of anodized titanium oxides

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**Purpose:** Anatase (A) and Rutile (R) phases of titanium oxide have been reported to increase antimicrobial effects and enhance bioactivity levels. However, an A/R phase ratio which may have the greatest effects has not been specified. A previous study in our laboratories produced specific A/R phase ratios through the use of different acid electrolyte mixtures and a stepped potentiostatic forming voltage. The purpose of this study was to evaluate the crystalline formation, surface morphology, wetting angle and corrosion resistance of anodized layers at each voltage step in acid electrolyte mixtures.

**Methods and materials:** Commercially pure titanium Grade 4 samples were cut from 2.00-mm sheet to 1 in 2 samples and ultrasonically cleaned in alcohol. Samples were dipped in a nitric-hydrofluoric acid solution for a period of 30 s, rinsed with distilled water, and anodized using potentiostatic 12 V 10 s steps. The anodization electrolytes were mixtures of sulfuric acid, phosphoric acid, oxalic acid, and hydrogen peroxide. Samples were analyzed for crystalline phases using thin film X-ray diffraction (XRD). Oxide thickness and surface morphology were examined using scanning electron microscopy (SEM) and image analysis. Wetting angles of both UVA (1 h) and non-UV treated samples were measured using a digital microscope. Corrosion resistance was evaluated using electrochemical impedance spectroscopy (EIS) at 37 °C in Ringer's solution.

**Results:** XRD results showed anatase and rutile formation to be electrolyte dependent with anatase forming first and rutile at higher forming voltages. Anodized layer thickness showed exponential growth with increasing voltage steps. The surface pore density was shown to have a bimodal distribution while all but one electrolyte showed increasing pore size with increasing forming voltage. The wetting angle results showed only a small effect of the UVA treatment with some samples becoming more hydrophilic and others becoming more hydrophobic. EIS results showed a trend of increasing corrosion resistance with increasing barrier thickness until a critical pore size was reached.

**Conclusion:** The surface oxide crystallinity, thickness, morphology, and corrosion resistance for each anodized layer were found to be dependent on both the electrolyte mixture and the applied voltage. The wetting angles showed little variation between forming voltages and UVA exposure for each electrolyte. The corrosion resistance was found to be superior for anodized layers with compact barrier layers and smaller pores. The detailed understanding of the surface porosity and corrosion resistance of each of these anodized layers is anticipated to provide valuable insight for future antimicrobial, bioactivity, and osseointegration testing of implant materials.

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### Evaluation of the ion-releasing property of tailored Sr–Ca-containing inorganic cement



W. Kiba<sup>1,\*</sup>, H. Imai<sup>2</sup>, K. Kondoh<sup>2</sup>, S. Imazato<sup>1</sup>

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**Purpose:** It is known that Sr<sup>2+</sup> promotes bone remodeling and Ca<sup>2+</sup> induces osteoblast differentiation, which is expected to act as a bio-active component. The purpose of this study was to develop a Sr<sup>2+</sup> and Ca<sup>2+</sup> containing inorganic cement using bioactive glass based on 45S5 and to investigate its ion-releasing property *in vitro*.

**Methods and materials:** Bioactive glass (45SiO<sub>2</sub>–6P<sub>2</sub>O<sub>5</sub>–24.5Na<sub>2</sub>O–(24.5–x)CaO–xSrO) (wt%), where x = 0 (Sr0), 6 (Sr<sub>6</sub>), 12 (Sr<sub>12</sub>), were made by melting in a plat-

inum crucible at 1200 °C for 2 h and immediately quenched in deionized water. The bioactive glass was ground and sieved with a planetary ball mill to obtain <10 μm powder. The chemical composition of glasses was analyzed by an X-ray diffractometer (XRD) and X-ray Fluorescence Analyzer (XRF). The experimental cement was made by mixing the bioactive glass with fluoroaluminosilicate glass and polyacrylic/tricarboxylic acids. The glass powder and cement discs were immersed in MilliQ water, and the concentration of Sr and Ca released after storage at 37 °C for up to 21 days was measured by an ICP optical emission spectroscopy. The results were analyzed by ANOVA and Fisher's PLSD tests at p < 0.05 level.

**Results:** Strontium was successfully incorporated in the glass resulting in XRD and XRF. When strontium substitution increases, both glass powder and cements, show significantly greater Sr-release (p < 0.05). Regarding Ca liberation, increasing strontium substitution show significantly lower release (p < 0.05) until 3 days, and after 7–21 days there was no correlation between release profile and glass composition. Similarly, there were no significant differences in Ca-release among all the cements. This may be because hydroxyapatite was formed in the water. In general, continuous release of both Sr (Sr<sub>6</sub> and Sr<sub>12</sub>) and Ca was observed for 14 days.

**Conclusion:** Sr–Ca-containing cement was successfully fabricated. This cement continuously releases both Sr<sup>2+</sup> and Ca<sup>2+</sup> and the amount of Sr released can be controlled. Supported by a Grant-in-Aid for Scientific Research (268615940) from the JSPS.

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### Effect of natural polyphenols on resin–dentin bond strength



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**Purpose:** The aim of this study was to evaluate the effect of dentin pre-treatment with natural polyphenols such as Quercetin (Q) and Resveratrol (R) on microtensile bonding strength of the dentin/resin interface.

**Methods and materials:** Extracted human permanent sound third molars were prepared to expose the dentin surface that was etched using 35% phosphoric acid for 15 s, followed by rinsing (30 s) and slight drying. On etched dentin 5 μl of one of the following solutions was applied: Distilled water; Quercetin (0.02%, 0.1%, 0.5%, 1.0%); Resveratrol (0.02%, 0.1%, 0.5%, 1.0%); Quercetin + Resveratrol 3:1, 1:1, 1:3 (0.02%, 0.1%, 0.5%, 1.0%); Chlorhexidine digluconate 2%. Then Single Bond Universal Adhesive (3M ESPE) was applied and a subsequent Z350XT (3M ESPE) resin crown was built with 2 increments of 2-mm-thick. After 24 h bonded teeth were sectioned in slabs and measurements of resin/dentin microtensile bond strength were taken. Data were analyzed by a one-way ANOVA and Tukey post hoc test (p < 0.05).

**Methods and materials:** Commercially pure titanium Grade 4 samples were cut from 2.00-mm sheet to 1 in 2 samples and ultrasonically cleaned in alcohol. Samples were dipped in a nitric-hydrofluoric acid solution for a period of 30 s, rinsed with distilled water, and anodized using potentiostatic 12 V 10 s steps. The anodization electrolytes were mixtures of sulfuric acid, phosphoric acid, oxalic acid, and hydrogen peroxide. Samples were analyzed for crystalline phases using thin film X-ray diffraction (XRD). Oxide thickness and surface morphology were examined using scanning electron microscopy (SEM) and image analysis. Wetting angles of both UVA (1 h) and non-UV treated samples were measured using a digital microscope. Corrosion resistance was evaluated using electrochemical impedance spectroscopy (EIS) at 37 °C in Ringer's solution.

**Results:** XRD results showed anatase and rutile formation to be electrolyte dependent with anatase forming first and rutile at higher forming voltages. Anodized layer thickness showed exponential growth with increasing voltage steps. The surface pore density was shown to have a bimodal distribution while all but one electrolyte showed increasing pore size with increasing forming voltage. The wetting angle results showed only a small effect of the UVA treatment with some samples becoming more hydrophilic and others becoming more hydrophobic. EIS results showed a trend of increasing corrosion resistance with increasing barrier thickness until a critical pore size was reached.

**Conclusion:** The surface oxide crystallinity, thickness, morphology, and corrosion resistance for each anodized layer were found to be dependent on both the electrolyte mixture and the applied voltage. The wetting angles showed little variation between forming voltages and UVA exposure for each electrolyte. The corrosion resistance was found to be superior for anodized layers with compact barrier layers and smaller pores. The detailed understanding of the surface porosity and corrosion resistance of each of these anodized layers is anticipated to provide valuable insight for future antimicrobial, bioactivity, and osseointegration testing of implant materials.

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### Evaluation of the ion-releasing property of tailored Sr–Ca-containing inorganic cement



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**Methods and materials:** Bioactive glass (45SiO<sub>2</sub>–6P<sub>2</sub>O<sub>5</sub>–24.5Na<sub>2</sub>O–(24.5–x)CaO–xSrO) (wt%), where x = 0 (Sr0), 6 (Sr<sub>6</sub>), 12 (Sr<sub>12</sub>), were made by melting in a plat-

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**Results:** Strontium was successfully incorporated in the glass resulting in XRD and XRF. When strontium substitution increases, both glass powder and cements, show significantly greater Sr-release (*p* < 0.05). Regarding Ca liberation, increasing strontium substitution show significantly lower release (*p* < 0.05) until 3 days, and after 7–21 days there was no correlation between release profile and glass composition. Similarly, there were no significant differences in Ca-release among all the cements. This may be because hydroxyapatite was formed in the water. In general, continuous release of both Sr (Sr<sub>6</sub> and Sr<sub>12</sub>) and Ca was observed for 14 days.

**Conclusion:** Sr–Ca-containing cement was successfully fabricated. This cement continuously releases both Sr<sup>2+</sup> and Ca<sup>2+</sup> and the amount of Sr released can be controlled. Supported by a Grant-in-Aid for Scientific Research (268615940) from the JSPS.

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### Effect of natural polyphenols on resin–dentin bond strength



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**Purpose:** The aim of this study was to evaluate the effect of dentin pre-treatment with natural polyphenols such as Quercetin (Q) and Resveratrol (R) on microtensile bonding strength of the dentin/resin interface.

**Methods and materials:** Extracted human permanent sound third molars were prepared to expose the dentin surface that was etched using 35% phosphoric acid for 15 s, followed by rinsing (30 s) and slight drying. On etched dentin 5 μl of one of the following solutions was applied: Distilled water; Quercetin (0.02%, 0.1%, 0.5%, 1.0%); Resveratrol (0.02%, 0.1%, 0.5%, 1.0%); Quercetin + Resveratrol 3:1, 1:1, 1:3 (0.02%, 0.1%, 0.5%, 1.0%); Chlorhexidine digluconate 2%. Then Single Bond Universal Adhesive (3M ESPE) was applied and a subsequent Z350XT (3M ESPE) resin crown was built with 2 increments of 2-mm-thick. After 24 h bonded teeth were sectioned in slabs and measurements of resin/dentin microtensile bond strength were taken. Data were analyzed by a one-way ANOVA and Tukey post hoc test (*p* < 0.05).

**Methods and materials:** Commercially pure titanium Grade 4 samples were cut from 2.00-mm sheet to 1 in 2 samples and ultrasonically cleaned in alcohol. Samples were dipped in a nitric-hydrofluoric acid solution for a period of 30 s, rinsed with distilled water, and anodized using potentiostatic 12 V 10 s steps. The anodization electrolytes were mixtures of sulfuric acid, phosphoric acid, oxalic acid, and hydrogen peroxide. Samples were analyzed for crystalline phases using thin film X-ray diffraction (XRD). Oxide thickness and surface morphology were examined using scanning electron microscopy (SEM) and image analysis. Wetting angles of both UVA (1 h) and non-UV treated samples were measured using a digital microscope. Corrosion resistance was evaluated using electrochemical impedance spectroscopy (EIS) at 37 °C in Ringer's solution.

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### Effect of natural polyphenols on resin–dentin bond strength



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**Purpose:** The aim of this study was to evaluate the effect of dentin pre-treatment with natural polyphenols such as Quercetin (Q) and Resveratrol (R) on microtensile bonding strength of the dentin/resin interface.

**Methods and materials:** Extracted human permanent sound third molars were prepared to expose the dentin surface that was etched using 35% phosphoric acid for 15 s, followed by rinsing (30 s) and slight drying. On etched dentin 5 μl of one of the following solutions was applied: Distilled water; Quercetin (0.02%, 0.1%, 0.5%, 1.0%); Resveratrol (0.02%, 0.1%, 0.5%, 1.0%); Quercetin + Resveratrol 3:1, 1:1, 1:3 (0.02%, 0.1%, 0.5%, 1.0%); Chlorhexidine digluconate 2%. Then Single Bond Universal Adhesive (3M ESPE) was applied and a subsequent Z350XT (3M ESPE) resin crown was built with 2 increments of 2-mm-thick. After 24 h bonded teeth were sectioned in slabs and measurements of resin/dentin microtensile bond strength were taken. Data were analyzed by a one-way ANOVA and Tukey post hoc test (p < 0.05).



**Table 1**

Concentration	Means (MPa) and standard deviation			
	0.02%	0.1%	0.5%	1%
Quercetin	26.4 (8.9) <sup>bcd</sup>	32.1 (8.9) <sup>ef</sup>	25.6 (6.8) <sup>abcde</sup>	25.4 (6.2) <sup>ab</sup>
Resveratrol	19.8 (5.9) <sup>abe</sup>	18.8 (6.1) <sup>a</sup>	23.7 (5.9) <sup>c</sup>	20.1 (5.3) <sup>abcde</sup>
Q + R (3:1)	34.8 (8.7) <sup>f</sup>	24.3 (7.3) <sup>abcd</sup>	25.1 (4.6) <sup>abcde</sup>	31.4 (8.5) <sup>def</sup>
Q + R (1:1)	23.7 (7.0) <sup>abc</sup>	18.8 (3.6) <sup>a</sup>	23.3 (5.2) <sup>abc</sup>	19.1 (5.5) <sup>ab</sup>
Q + R (1:3)	20.7 (2.9) <sup>abc</sup>	22.7 (6.4) <sup>abc</sup>	25.9 (7.9) <sup>abcde</sup>	23.8 (5.8) <sup>abc</sup>

**Results:** Table 1 displays the means and standard deviation of the obtained results. Quercetin at 0.02%, 0.1%, 0.5% produced significant change in resin–dentin bond strength compared to Resveratrol. There was not significant difference between Q 1% and R 1%. Both Q + R solutions (3:1–0.02% and 1%) improved significantly the bond strength, similar to chlorhexidine.

**Conclusion:** With the limitations of these results, it was noticed that Quercetin was more effective than Resveratrol in improving resin–dentin bond strength.

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#### Push-out bond strength of fiberglass posts with two resin cements



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**Purpose:** The aim of this study was to evaluate the push-out bond strength between fiberglass posts to root canal dentin using two different resin cements, a conventional etch-and-rinse and a self-adhesive resin cement.

**Methods and materials:** 12 single-rooted premolars recently extracted for orthodontics or periodontal reasons were endodontically treated and stored in distilled water for 24 h (37 °C). Specimens were then distributed into 2 groups ( $n=6$ ) according to the cementation strategy: G1 – a self-adhesive dual-cure resin cement (Relyx U200, 3M ESPE, ST. Paul, MN, USA) and G2 – a conventional, dual-cure, etch-and-rinse resin cement (Relyx ARC, 3M ESPE, ST. Paul, MN, USA) associated with one bottle total etch adhesive system (Single Bond2, 3M ESPE, ST. Paul, MN, USA), the adhesive system was applied according to the manufacturer's instructions and photoactivated for 20s before cementation. All posts (Exacto # 1, Angelus, Londrina, Brazil) were cleaned with 96% alcohol and silanized (Angelus, Londrina, Brazil) before luting procedures. Both cements were applied according to the manufacturer's instructions. After storage in water at 37 °C during 24 h, the samples were sectioned perpendicular to the long axis of the root into four slices (thickness of 2 mm). Push-out test was performed at a crosshead speed of 0.5 mm/min. The bond strength (MPa) data were submitted to Mann–Whitney non-parametric test at 5% of significance.

**Results:** There was no statistically significant difference between the auto-adhesive resin cement and the conventional resin cement ( $p=0.78$ ) by Mann–Whitney non-parametric test

for the push-out bond strength. The mean bond strength (MPa) and standard deviation were G1 = 15.85 ( $\pm 6.54$ ) and G2 = 12.55 ( $\pm 4.75$ ).

**Conclusion:** The self-adhesive and the conventional etch-and-rinse resin cements presented similar performance when used to lute fiberglass posts to root canal dentin.

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#### Total MMP activity of dentin matrices pretreated with natural crosslinkers



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**Purpose:** Enzymatic degradation of resin-infiltrated dentin matrices by host-derived endogenous proteinase activity results in the loss of stability of resin–dentin bonds. Matrix metalloproteinases (MMP's) found in dentin were shown to be responsible for the degradation. The aim of this study was to investigate the effects of one or five minute application of natural crosslinkers on matrix-bound total MMP activity.

**Methods and materials:** Dentin specimens (0.4 mm  $\times$  4 mm  $\times$  4 mm,  $n=10$ /group) were demineralized in 10% phosphoric acid for 24 h and baseline activity of each sample was measured using generic MMP activity assay (Sensolyte Generic MMP assay, Anaspec, San Jose, CA). After baseline measurements, dentin specimens were dipped in 300  $\mu$ l aqueous solution of 1 or 5 wt% grape seed extract (GS), 10 wt% sumac extract (S), 20  $\mu$ M or 200  $\mu$ M curcumin (CR) for 1 or 5 min, rinsed and incubated in fresh chromogenic substrate and assay buffer in the 96-well plate for 60 min and total MMP activity was reassessed. 5% glutaraldehyde (GA) treatment and no crosslinker treatment groups served as control. The total MMP activity was expressed as a percentage of the untreated baseline level of each sample to determine the percent inhibition or activation. Another set of demineralized dentin beams ( $n=10$ /group) were treated with respective crosslinkers and incubated in extraction buffer for 72 h at 4 °C. Extracted proteins were measured with total protein assay and MMP-2, -8, -9 were measured by using multiplex bead technology. Data were analyzed with ANOVA at  $\alpha=0.05$ .

**Results:** The results of the study showed that MMP activity in crosslinker pretreated samples, decreased significantly between 27% and 70%, ( $p<0.05$ ), whereas untreated control samples' activity increased up to 84%. Total protein assay showed a decrease of 30–50% in the amount of extractable total protein after 1 min of crosslinking and 70–80% decrease after 5 min crosslinking. Multiplex analysis of extracts showed a significant reduction in the MMP-8, MMP-2 and MMP-9 release after crosslinker treatment ( $p<0.05$ ). MMP-9 extractability from all groups was significantly lower compared to MMP-2 or MMP-8 levels ( $p<0.05$ ). After 5 min pre-treatment, none of the pre-treatment groups showed any extractable MMP-9.

**Table 1**

Concentration	Means (MPa) and standard deviation			
	0.02%	0.1%	0.5%	1%
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**Methods and materials:** 12 single-rooted premolars recently extracted for orthodontics or periodontal reasons were endodontically treated and stored in distilled water for 24 h (37 °C). Specimens were then distributed into 2 groups ( $n=6$ ) according to the cementation strategy: G1 – a self-adhesive dual-cure resin cement (Relyx U200, 3M ESPE, ST. Paul, MN, USA) and G2 – a conventional, dual-cure, etch-and-rinse resin cement (Relyx ARC, 3M ESPE, ST. Paul, MN, USA) associated with one bottle total etch adhesive system (Single Bond2, 3M ESPE, ST. Paul, MN, USA), the adhesive system was applied according to the manufacturer's instructions and photoactivated for 20s before cementation. All posts (Exacto # 1, Angelus, Londrina, Brazil) were cleaned with 96% alcohol and silanized (Angelus, Londrina, Brazil) before luting procedures. Both cements were applied according to the manufacturer's instructions. After storage in water at 37 °C during 24 h, the samples were sectioned perpendicular to the long axis of the root into four slices (thickness of 2 mm). Push-out test was performed at a crosshead speed of 0.5 mm/min. The bond strength (MPa) data were submitted to Mann–Whitney non-parametric test at 5% of significance.

**Results:** There was no statistically significant difference between the auto-adhesive resin cement and the conventional resin cement ( $p=0.78$ ) by Mann–Whitney non-parametric test

for the push-out bond strength. The mean bond strength (MPa) and standard deviation were  $G1=15.85 (\pm 6.54)$  and  $G2=12.55 (\pm 4.75)$ .

**Conclusion:** The self-adhesive and the conventional etch-and-rinse resin cements presented similar performance when used to lute fiberglass posts to root canal dentin.

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### Total MMP activity of dentin matrices pretreated with natural crosslinkers



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**Purpose:** Enzymatic degradation of resin-infiltrated dentin matrices by host-derived endogenous proteinase activity results in the loss of stability of resin–dentin bonds. Matrix metalloproteinases (MMP's) found in dentin were shown to be responsible for the degradation. The aim of this study was to investigate the effects of one or five minute application of natural crosslinkers on matrix-bound total MMP activity.

**Methods and materials:** Dentin specimens (0.4 mm × 4 mm × 4 mm,  $n=10$ /group) were demineralized in 10% phosphoric acid for 24 h and baseline activity of each sample was measured using generic MMP activity assay (Sensolyte Generic MMP assay, Anaspec, San Jose, CA). After baseline measurements, dentin specimens were dipped in 300  $\mu$ l aqueous solution of 1 or 5 wt% grape seed extract (GS), 10 wt% sumac extract (S), 20  $\mu$ M or 200  $\mu$ M curcumin (CR) for 1 or 5 min, rinsed and incubated in fresh chromogenic substrate and assay buffer in the 96-well plate for 60 min and total MMP activity was reassessed. 5% glutaraldehyde (GA) treatment and no crosslinker treatment groups served as control. The total MMP activity was expressed as a percentage of the untreated baseline level of each sample to determine the percent inhibition or activation. Another set of demineralized dentin beams ( $n=10$ /group) were treated with respective crosslinkers and incubated in extraction buffer for 72 h at 4 °C. Extracted proteins were measured with total protein assay and MMP-2, -8, -9 were measured by using multiplex bead technology. Data were analyzed with ANOVA at  $\alpha=0.05$ .

**Results:** The results of the study showed that MMP activity in crosslinker pretreated samples, decreased significantly between 27% and 70%, ( $p<0.05$ ), whereas untreated control samples' activity increased up to 84%. Total protein assay showed a decrease of 30–50% in the amount of extractable total protein after 1 min of crosslinking and 70–80% decrease after 5 min crosslinking. Multiplex analysis of extracts showed a significant reduction in the MMP-8, MMP-2 and MMP-9 release after crosslinker treatment ( $p<0.05$ ). MMP-9 extractability from all groups was significantly lower compared to MMP-2 or MMP-8 levels ( $p<0.05$ ). After 5 min pre-treatment, none of the pre-treatment groups showed any extractable MMP-9.

**Table 1**

Concentration	Means (MPa) and standard deviation			
	0.02%	0.1%	0.5%	1%
Quercetin	26.4 (8.9) <sup>bcd</sup>	32.1 (8.9) <sup>ef</sup>	25.6 (6.8) <sup>abcde</sup>	25.4 (6.2) <sup>ab</sup>
Resveratrol	19.8 (5.9) <sup>abe</sup>	18.8 (6.1) <sup>a</sup>	23.7 (5.9) <sup>c</sup>	20.1 (5.3) <sup>abcde</sup>
Q + R (3:1)	34.8 (8.7) <sup>f</sup>	24.3 (7.3) <sup>abcd</sup>	25.1 (4.6) <sup>abcde</sup>	31.4 (8.5) <sup>def</sup>
Q + R (1:1)	23.7 (7.0) <sup>abc</sup>	18.8 (3.6) <sup>a</sup>	23.3 (5.2) <sup>abc</sup>	19.1 (5.5) <sup>ab</sup>
Q + R (1:3)	20.7 (2.9) <sup>abc</sup>	22.7 (6.4) <sup>abc</sup>	25.9 (7.9) <sup>abcde</sup>	23.8 (5.8) <sup>abc</sup>

**Results:** Table 1 displays the means and standard deviation of the obtained results. Quercetin at 0.02%, 0.1%, 0.5% produced significant change in resin–dentin bond strength compared to Resveratrol. There was not significant difference between Q 1% and R 1%. Both Q + R solutions (3:1–0.02% and 1%) improved significantly the bond strength, similar to chlorhexidine.

**Conclusion:** With the limitations of these results, it was noticed that Quercetin was more effective than Resveratrol in improving resin–dentin bond strength.

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#### Push-out bond strength of fiberglass posts with two resin cements



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Federal University of Santa Catarina, Department of Operative Dentistry, Florianópolis, Brazil

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**Conclusion:** The result of this work suggests that the effect of the crosslinkers is source-dependent. The use of crosslinkers for as little as 1 min on demineralized dentin can inactivate the endogenous protease activity of dentin matrices by crosslinking dentin MMPs as well as crosslinking collagen matrix and non-collagenous proteins.

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### Effect of thickness of indirect resins in microhardness luting systems



L.P. Moraes, V.C. Ruschel, S. Monteiro Júnior, C. Deucher\*, C.P. Gré, R.C. De Ré Silveira

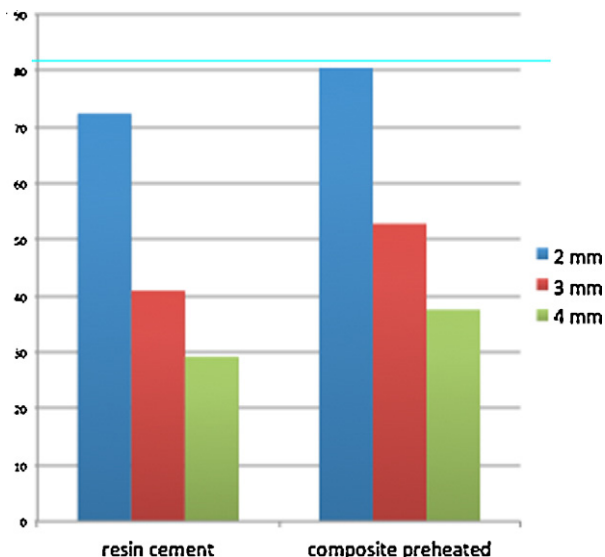
Federal University of Santa Catarina, Brazil

**Purpose:** This study aimed to assess the hardness of a pre-heated composite resin and a photoactivated self-adhesive cement using indirect resin discs with different thicknesses.

**Methods and materials:** 30 nanoparticle composite discs (Filtek Z350 XT – 3M ESPE) were manufactured and pre-heated to CalSet device (68 °C for 15 min) and 30 self-adhesive resin cement discs (RelyX U200, 3M ESPE). Three additional disks of indirect microparticulate resin (SR Love – Ivoclar-Vivadent) of different thicknesses (2 mm, 3 mm, 4 mm) were also prepared, which served as a barrier during polymerization (for 120 s) luting material. The discs were divided into 6 groups (n=10) according to the thickness of the disc and the luting material: GR2 – preheated resin + 2 mm disk; GR3 – preheated resin + 3 mm disc; GR4 – preheated resin + 4 mm disc; GC2 – resin cement + 2 mm disk; GC3 – resin cement + 3 mm disc; GC4 – resin cement + 4 mm disc. Then there was the Vickers hardness test (HMV 2 Version 1.23 Shimadzu, Japan), performing the averages of five indentations in each sample.

**Results:** Microhardness values were obtained: GR2 ( $80.50 \pm 8.55$ ) > GR3 ( $52.87 \pm 7.52$ ) > GR4 ( $37.58 \pm 5.06$ ) ( $p < 0.05$ ). For resin cement, the hardness was found: GC2 ( $72.38 \pm 25.73$ ) was statistically similar to GC3 ( $40.95 \pm 3.81$ ), and significantly higher than GC4 ( $29.20 \pm 8.16$ ). The GC3 group showed statistically similar values to GC4 ( $p > 0.05$ ). The preheated composite resin ( $56.98 \pm 19.35$ ) showed higher hardness compared to the resin cement ( $47.51 \pm 23.96$ ) ( $p < 0.05$ ), regardless of the thickness of the restoration. The 2 mm thick restorer material showed higher microhardness values ( $76.44 \pm 19.12$ ) compared to 3 mm ( $46.91 \pm 8.43$ ) and 4 mm ( $33.39 \pm 7.88$ ) ( $p < 0.05$ ), regardless of the employee cementing agent.

**Conclusion:** There is indirect influence of the thickness of the resin in hardness conversely, whether the cementing agent, the greater the thickness the lower hardness. Also, the preheated composite has higher hardness values compared to the self-adhesive cement, independent of the thickness.



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### Zymographic analysis of experimental MMP inhibitors containing adhesives



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**Purpose:** Recent studies supported the use of MMP inhibitors during bonding procedures to inactivate endogenous dentin proteases, preventing dentin collagen degradation thus improving bond durability. This study aimed at evaluating the effect of experimental adhesives containing MMP inhibitors: clorexidine or galardin.

**Methods and materials:** For this study, an experimental etch-and-rinse adhesive system (EXP) was developed (composition in weight: acetone 15%, HEMA 25%, TEGDMA 25%, 4-META 30%, H<sub>2</sub>O 4%, canphoroquinone 0.5%, EDMAB 0.5%). Chlorhexidine diacetate (CHX) or galardin (GAL) were blended within the adhesive formulation producing the following adhesive formulations: EXP + 5 μM GAL; EXP + 2% in weight CHX. Zymographic analyses on protein extracts from demineralized dentin powder were performed in accordance with Mazzoni et al., 2014. Demineralized dentin powder was treated as follow: (1) untreated as control; (2) treated with EXP; (3) treated with GAL-containing EXP for 30 min; (4) treated with CHX-containing EXP for 30 min.

**Results:** The results of the zymographic analysis showed that the use of EXP induced MMP2 and MMP9 activation, while both MMP-inhibitors containing adhesives resulted in an incomplete inhibition of MMP-9 activity, and a complete inhibition of MMP-2.

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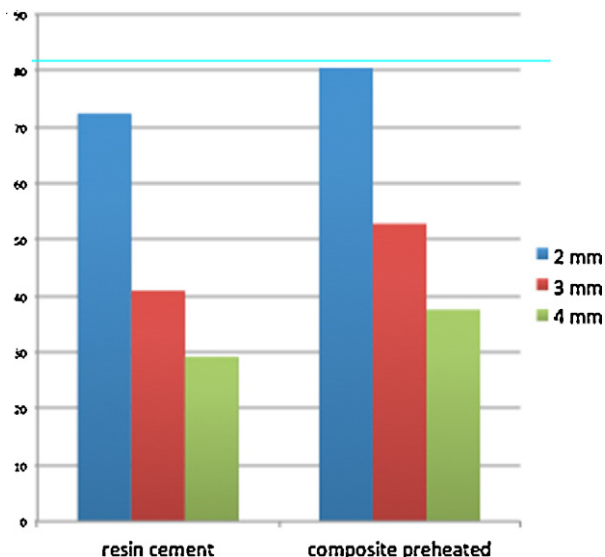
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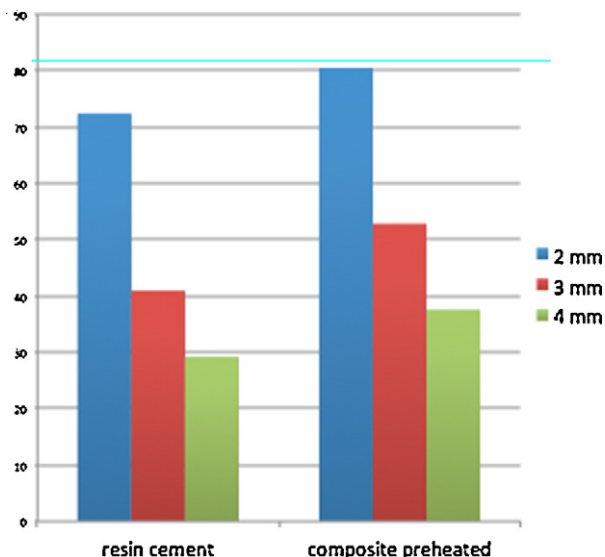
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needed to validate the use of these experimental MMPs-inhibitor containing adhesives to stabilize the bond over time.

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### Silanization effect on bond strength between glass-ceramic and resin cement



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Federal University of Santa Catarina, Brazil

**Purpose:** To evaluate the influence of a silane-coupling agent on the microtensile bond strength of etch-and-rinse cement to a lithium disilicate reinforced glass-ceramic.

**Methods and materials:** 4 ceramic blocks – 11 mm long × 9 mm wide × 4 mm thick- (IPS e.max Press, Ivoclar-Vivadent AG, Schann, Liechtenstein) were fabricated and divided into 2 groups (n=2), in Group 1 the ceramic surface was etched with hydrofluoric acid 10% (Condac, FGM, SC, Brazil) for 20 s, rinsed with water spray and air-dried. One layer of a silane-coupling agent (Monobond S, IvoclarVivadent AG, Schann, Liechtenstein) was applied onto all ceramic specimens for 60 s and air-dried for 30 s. In Group 2, the ceramic surface was etched with hydrofluoric acid 10% (Condac Porcelana, FGM, Joinville, Brazil) for 20 s, rinsed with water spray and air-dried without application of the silane-coupling agent. The ceramic blocks were bonded to composite resin blocks (Filtek Z350 XT, 3M ESPE, St. Paul, MN), previously fabricated with the same dimensions of the ceramic blocks, with etch-and-rinse resin cement (Variolink II, IvoclarVivadent AG, Schann, Liechtenstein) according to the manufacturer's instructions. After storage for 24 h in distilled water at 37 °C, the specimens were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of 0.8 mm<sup>2</sup> and submitted to a microtensile bond strength test (n=30). Data were statistically analyzed by Student's T test (p=0.05).

**Results:** Microtensile bond strength was significantly affected by the application of the silane-coupling agent (p=0.01). Silanized group presented higher bond strength values (21.93 ± 3.45 MPa) than the group without silanization of the ceramic surface (17.99 ± 4.31 MPa).

**Conclusion:** Applying a silane-coupling agent on the lithium disilicate reinforced glass-ceramic may improve the bond strength when etch-and-rinse resin cement is used for cementation.

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### Evaluation of universal bonding agent containing MDP on zirconia bonding



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Gangnam Severance Hospital, College of Dentistry, Yonsei University, South Korea

**Purpose:** Use of Yttria-tetragonal zirconia (Y-TZP) has increased because of its strength. However, cementation is one of the critical issues because it is well known that Y-TZP cannot be etched with HF. To solve this problem, the effect of silica coated sandblasting, zirconia primer, glass infiltration and MDP containing resin cement had been studied. Recently many universal bonding agents were introduced in the market, and most of them contain MDP. If they are effective for zirconia bonding, the cementation procedure can be simplified with no need to use other instruments or materials. The purpose of this study was to evaluate the MDP-containing universal bonding agents for the zirconia bonding.

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**Results:** Mean bond strength values are shown in Table 1. SBU showed higher MSB than CSE, GCU and OFL. OFL, which does not contain MDP showed the lowest MSB. There was no significant difference between CSE, ABU and GCU.

**Table 1 – Microshear bond strength of experimental groups (MPa, n = 15).**

Materials	Mean ± S.D.
Optibond FL (OFL)	23.49 ± 7.60 <sup>c</sup>
Clearfil SE Bond (CSE)	26.60 ± 9.42 <sup>b</sup>
Single Bond Universal (SBU)	35.38 ± 6.24 <sup>a</sup>
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needed to validate the use of these experimental MMPs-inhibitor containing adhesives to stabilize the bond over time.

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### Silanization effect on bond strength between glass-ceramic and resin cement



R. de Re Silveira\*, C.P. Gré, C.M. Velho, C. Deucher, L.P. Moraes, L.C.C. Vieira

Federal University of Santa Catarina, Brazil

**Purpose:** To evaluate the influence of a silane-coupling agent on the microtensile bond strength of etch-and-rinse cement to a lithium disilicate reinforced glass-ceramic.

**Methods and materials:** 4 ceramic blocks – 11 mm long × 9 mm wide × 4 mm thick- (IPS e.max Press, Ivoclar-Vivadent AG, Schann, Liechtenstein) were fabricated and divided into 2 groups ( $n=2$ ), in Group 1 the ceramic surface was etched with hydrofluoric acid 10% (Condac, FGM, SC, Brazil) for 20 s, rinsed with water spray and air-dried. One layer of a silane-coupling agent (Monobond S, IvoclarVivadent AG, Schann, Liechtenstein) was applied onto all ceramic specimens for 60 s and air-dried for 30 s. In Group 2, the ceramic surface was etched with hydrofluoric acid 10% (Condac Porcelana, FGM, Joinville, Brazil) for 20 s, rinsed with water spray and air-dried without application of the silane-coupling agent. The ceramic blocks were bonded to composite resin blocks (Filtek Z350 XT, 3M ESPE, St. Paul, MN), previously fabricated with the same dimensions of the ceramic blocks, with etch-and-rinse resin cement (Variolink II, IvoclarVivadent AG, Schann, Liechtenstein) according to the manufacturer's instructions. After storage for 24 h in distilled water at 37 °C, the specimens were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of 0.8 mm<sup>2</sup> and submitted to a microtensile bond strength test ( $n=30$ ). Data were statistically analyzed by Student's T test ( $p=0.05$ ).

**Results:** Microtensile bond strength was significantly affected by the application of the silane-coupling agent ( $p=0.01$ ). Silanized group presented higher bond strength values ( $21.93 \pm 3.45$  MPa) than the group without silanization of the ceramic surface ( $17.99 \pm 4.31$  MPa).

**Conclusion:** Applying a silane-coupling agent on the lithium disilicate reinforced glass-ceramic may improve the bond strength when etch-and-rinse resin cement is used for cementation.

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### Evaluation of universal bonding agent containing MDP on zirconia bonding



T.W. Kim, D.K. Yang\*, S.I. Kim, S.J. Shin, J.W. Park

Gangnam Severance Hospital, College of Dentistry, Yonsei University, South Korea

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### Can cementation strategy influence the fatigue resistance of zirconia crowns?



F. Campos<sup>1</sup>, M.A. Bottino<sup>1,\*</sup>, L.F. Valandro<sup>2</sup>, C.J. Kleverlaan<sup>3</sup>, A.J. Feilzer<sup>3</sup>

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**Purpose:** The aim of this study was to investigate the influence of the cementation strategy on the fatigue resistance of zirconia crowns. The null hypothesis was that the cementation strategy will not affect the fatigue resistance of the crowns.

**Methods and materials:** Seventy five posterior full-crown simplified abutments (height=6mm, flat top, axial walls' convergence=12 degrees,) were machined in glass-fiber-filled epoxy resin. Zirconia crowns were designed (thickness=0.7 mm), milled by CAD/CAM and sintered, according to manufacturer's instructions. These crowns were cemented on the resin abutments using five cementation strategies: ZP – zinc phosphate cement, PN – resin cement, AL – air-particles abrasion with alumina particles (125 μm)+resin cement, CJ – air-particle abrasion with alumina coated by silica particles (30 μm) + silane + resin cement, GL – application of a glaze layer + etching with hydrofluoric acid + silane + resin cement. The resin cement was activated for 30 s each face. The specimens were tested until fracture in a stepwise stress fatigue test (10,000 cycles in each step, 600–1400 N, frequency of 1.4 Hz). Data were analyzed by Kaplan–Meier and Mantel–Cox (Log Rank) tests and a pairwise comparison ( $P < 0.05$ ), and by a two-parameter Weibull analysis.

**Results:** For the load to fracture, a difference was detected among the cementation strategies (Mantel–Cox Log-Rank test,  $X^2 = 56.50$ ,  $df = 4$ ,  $P = 0.000 < 0.05$ ). Besides, for the number of cycles to fracture, this difference was also detected (Mantel–Cox Log-Rank test,  $X^2 = 92.34$ ,  $df = 4$ ,  $P = 0.000 < 0.05$ ). The crowns cemented adhesively with silicatization (CJ) showed higher fatigue resistance when compared to the others crowns. The Weibull modulus and characteristic strength were higher for the CJ group.

**Conclusion:** It can be concluded that the cementation strategy influenced the fatigue resistance of zirconia crowns. Besides, the cementation with silicatization and resin cement presented better results of fatigue resistance.

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### In silico multi-scale analysis of composite resin block for CAD/CAM



S. Yamaguchi\*, S. Inoue, T. Sakai, T. Abe, S. Imazato

Osaka University Graduate School of Dentistry, Suita, Japan

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**Methods and materials:** The models of CAD/CAM CR block in nano-scale were designed by CAD software (Solidworks Simulation 2011). They were consisted of silica nano-filler particles (20, 40, 60, 80, and 100 nm) and matrix (Bis-GMA/TEGDMA), with volume contents of filler being 55.2%. Young's moduli and Poisson's ratios of silica and Bis-GMA/TEGDMA were set as 72 GPa, 0.16 and 2 GPa, 0.45, respectively. Calculation of Young's moduli and Poisson's ratios of the block were conducted by homogenization analysis in CAE software (VOXELCON2015). Compressive strengths were defined when the fracture loads exceeded 6075 N in case of macro-scale model (3 mm × 3 mm × 3 mm). Finally, localization analysis was performed to compare maximum principal strain (MPS) related to threshold of fracture initiation in nano-scale.

**Results:** Table 1 shows Young's moduli, Poisson's ratios, and compressive strengths of macro-scale model, and MPS of nano-scale model. As the filler size increased, Young's moduli and compressive strength were decreased while Poisson's ratios and MPS were increased. All those parameters significantly correlated with diameters of filler particles (Pearson's correlation test,  $r = -0.949, 0.943, -0.951, 0.976$ ,  $p < 0.05$ ).

**Table 1**

Size of nano-filler particles (nm)	20	40	60	80	100
Young's moduli (GPa)	16.7	15.1	13.8	13.5	13.1
Poisson's ratios	0.30	0.320	0.33	0.33	0.34
Compressive strength (MPa)	752	675	611	597	574
Maximum principal strain	2.5e−3	4.9e−3	7.1e−3	7.340e−3	9.3e−3

**Conclusion:** Within the limitation of this experiment, MDP-containing universal bonding agents showed possibility for the bonding of the zirconia restoration with a simplified clinical procedure.

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### Topical antibacterial gel for treatment of periodontal disease



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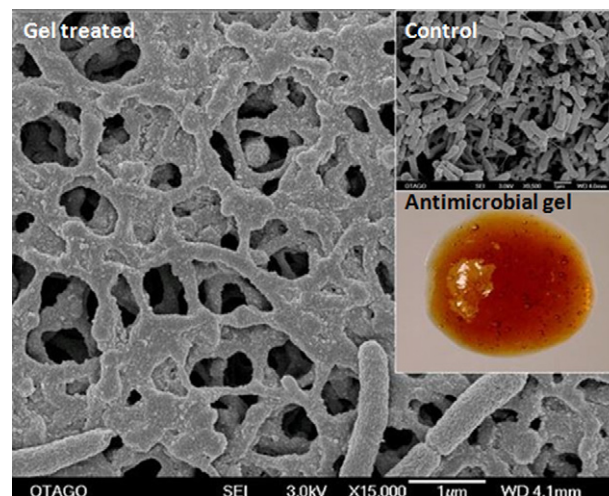
<sup>2</sup> University of Otago, Sir John Walsh Research Institute, Dunedin, New Zealand

**Purpose:** Despite intensive research exploring the treatment of periodontal disease there have been few significant advances leading to improved outcomes. Current management strategies involve chemo-mechanical disruption of biofilms which has limited success. Existing antimicrobials are less effective against biofilm cells; therefore extrapolation of planktonic data is not representative. We aim to develop robust biofilm models for the assessment of antimicrobials for pre-clinical *in vitro* approval. By establishing the efficacy of our novel antimicrobial gel we aim to demonstrate the potential for improved clinical outcomes in the management of chronic periodontal disease.

**Methods and materials:** Viability of planktonic bacterial cultures, when exposed to our novel formulation, was assessed with a Baclight™ Live/Dead assay. The "membrane sandwich" technique was developed for evaluating the viability of monoculture biofilms exposed to the formulation constituted as a gel. Morphology and architecture of treated biofilms were evaluated by scanning electron microscopy and compared to controls. Confocal laser scanning microscopy was used in conjunction with Comstat (Matlab) for quantitative analysis. Specific interactions of antimicrobial agent and bacteria were assessed by transmission electron microscopy. Gels were prepared containing a range of silver concentrations and tested against oral representative bacteria, including; *E. Coli*, *S. Oxford*, *P. Seudomonas*, *S. Mutan*, *S. Mitis*, *E. Faecalis* and *S. Gordonii*. Bacterial enumeration and biofilm imaging was conducted using Baclight™ Live/dead. Interaction of the antimicrobial with dental tissues in a sheep jaw was assessed histologically.

**Results:** Bactericidal activity was recorded within 3h of exposure of planktonic cultures. Immediate exposure resulted in >20% reduction in viability and in some cases complete inhibition. Disruption to bacterial morphology and biofilm structure was evident microscopically. In contrast, extracellular polymeric substance (EPS) increased when exposed to a control gel. 3-Dimensional analysis of biofilm geometrics indicated that treated biofilms had higher proportions of dead bacteria throughout the strata.

**Conclusion:** The novel silver gel formulation developed was effective against both planktonic and biofilm cultures. Where other formulations possess a limited antimicrobial spectrum, we have demonstrated this formulation against a range of bacteria, with no risk of resistance developing and a formulation that allows the sustained release of active agent.



<http://dx.doi.org/10.1016/j.dental.2015.08.087>

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### Re-examining the role of ferric chloride in resin-dentin bonding



R.V. Rodrigues<sup>1</sup>, M. Giannini<sup>1</sup>, R.M. Puppim-Rontani<sup>2</sup>, F.M. Pascon<sup>2</sup>, A.P. Manso<sup>3,\*</sup>, R.M. Carvalho<sup>3</sup>

<sup>1</sup> University of Campinas, Piracicaba Dental School, Piracicaba, Brazil

<sup>2</sup> University of Campinas, Pediatric Dentistry Department, Piracicaba, Brazil

<sup>3</sup> Department of Oral Biological and Medical Sciences, Faculty of Dentistry, University of British Columbia, Vancouver, Canada

**Purpose:** Etchants based on ferric chloride have long been abandoned in adhesive systems. Iron has recently been identified as the element responsible for preserving soft tissues in fossils (Microsc. Microanal. 2014;20:1430) Therefore, iron may play a role to preserve collagen in resin-dentin bonding. The objective of this preliminary study was to evaluate the bond strength to dentin treated with different acid solutions.

**Methods and materials:** 21 molars were randomly divided into 7 groups ( $n=3$ ), according to 5 different combinations of citric acid and ferric chloride. These acids were applied to dentin for 15 s before bonding with Adper Scotchbond MP (3M ESPE). Phosphoric acid (PA) treatments for 5 s and 15 s (control) were also included. Composite blocks (5 mm thick) were built-up and bonded teeth stored in PBS for 24 h at 37 °C. The teeth were sectioned into beams (1 mm<sup>2</sup>) and tested in tension (microtensile) @0.5 mm/min. The failure mode was observed under a stereomicroscope. Data were submitted to ANOVA and Tukey's test ( $\alpha=5\%$ ).

**Conclusion:** The *in silico* analytical model established in this study demonstrated that the Young's moduli, Poisson's ratios, compressive strengths, and MPS of CAD/CAM CR blocks could be improved by loading silica nano-filler particles with smaller diameter. It is possible to increase fracture resistance of CAD/CAM CR blocks by using smaller size silica nano-filler.

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86

### Topical antibacterial gel for treatment of periodontal disease



G.C. Cotton<sup>1,\*</sup>, C.J. Meledandri<sup>1</sup>, D.R. Schwass<sup>2</sup>, G. Tompkins<sup>2</sup>

<sup>1</sup> University of Otago, Department of Chemistry, Dunedin, New Zealand

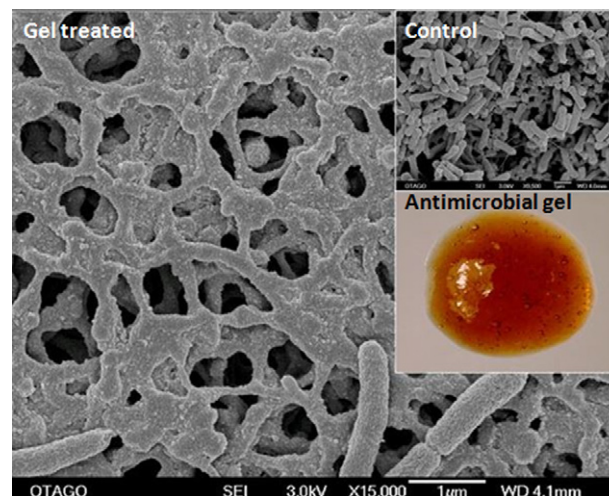
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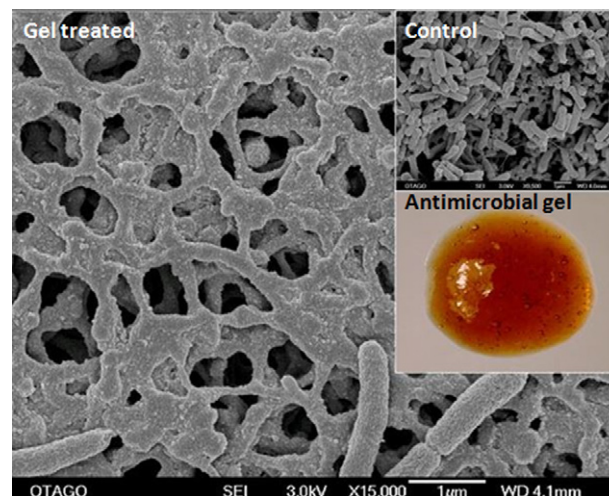
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**Results:** Different combinations of citric acid and ferric chloride resulted in bond strengths that were not different from the control PA/15s and also from the experimental PA/5s, except for 10% citric acid and 3% ferric chloride solution that resulted in the lowest bond strength. There were no differences between PA/5s and PA/15s (Table 1).

**Conclusion:** Different combinations of CA and ferric chloride resulted in bond strengths that were similar to current etching methods. There might be an optimal CA/FC ratio for improved bond strength and pursuit of that is warranted.

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### Evaluation of bonding performance using new adhesive and composite resin



H. Kato\*, A. Arita, T. Kumagai

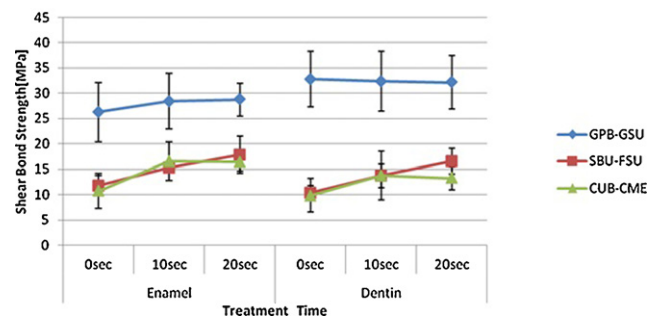
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### Cytotoxicity of adhesives and dental filling materials



J. Ruodan\*, L. Hong, Z. Gang

Peking University School of Stomatology, Beijing, China

**Purpose:** Apply a dentin barrier test using human dentin disks to evaluate the cytotoxicity of two dentin adhesives and two dental filling materials. Compare the results with those in a conventional filter diffusion test in order to investigate the advantages of the dentin barrier test.

**Methods and materials:** The cytotoxicity of adhesives [XP BOND(XP) and Xeno (XE)], composite resin [Clearfil Majesty ES-2(CF)] and calcium silicate pulp liner [TheraCal LC(TC)] were analyzed by both of the dentin barrier test and filter diffusion test. In the dentin barrier test, L929 mouse fibroblasts were three-dimensional cultured in polystyrene meshes. The 0.5 mm dentin disks were cut from the human third molars, near the pulp and in parallel with the occlusal surface, which mimic deep cavities *in vivo*. Their permeability within the measurement area was evaluated by a hydraulic permeability device. The dentin disks having the similar permeability were selected and used in the dentin barrier test. A slice of mesh with the cells was placed in the “pulp cavity” of the chamber in the dentin barrier test device and one dentin disk was put over the cell mesh and its “pulp side” was downward. The test materials and controls were in contact with the “occlusal side” of the dentine disks for 24 h. Cell survival rate (a percentage of control material) was evaluated using a MTT assay. The data was analyzed by the Mann-Whitney U test. In the filter diffusion test, after a 24 h contact between the test materials and the filters with monolayer cells, the grades of cytotoxicity were decided according to the result of cell staining.



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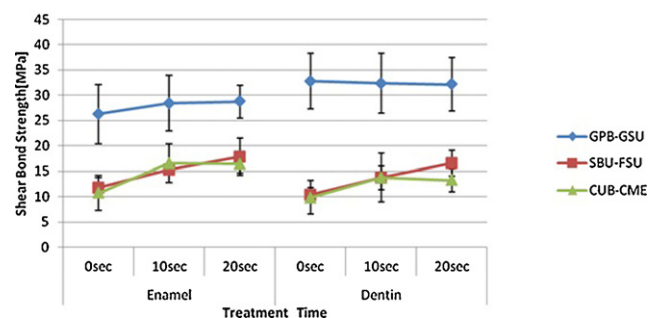
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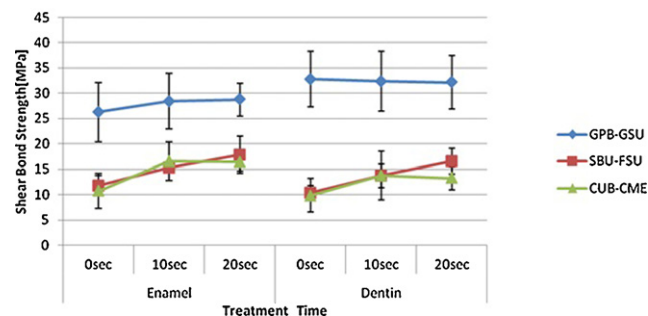
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**Table 1 – The cytotoxicity results for the dentin barrier test and the filter diffusion test.**

Test material	The dentin barrier test (cell survival)	The filter diffusion test (cytotoxicity grade)
XP	91%	Severe cytotoxicity
XE	97%	Moderate cytotoxicity
CF	91%	Mild cytotoxicity
TC	96%	Moderate cytotoxicity
Negative control	100%	No cytotoxicity

**Results:** In the dentin barrier test, XP, XE, CF and TC decreased cell viability non-significantly ( $p > 0.05$ ). In the filter diffusion test, XP showed severe cytotoxicity. XE and TC were moderately cytotoxic. CF was mildly cytotoxic. All the four materials in the dentin barrier test led to lower cytotoxicity than in the filter diffusion test.

**Conclusion:** The cytotoxicity of the test materials using the dentin barrier test with the human dentin disks having the similar permeability is lower than that in the filter diffusion test, which has good correlation with the clinical situation.

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#### Odontogenic differentiation of human dental pulp cells using mesoporous-bioactive-nanoparticles



J. Lee\*, H. Lee, H. Kim

Dankook University, South Korea

**Purpose:** Mesoporous bioactive nanoparticles (MBN) has been widely used in bone and dentin regeneration as carrier of drug and nucleonic acid; however, reports on the biological effects of MBN itself on dental pulp stem cells are rarely studied. This study aims to investigate the effects of MBG on odontogenic differentiation of dental pulp stem cells.

**Methods and materials:** After MBN was fabricated, rat dental pulp stem cells (rDPSC) were primarily cultured directly with MBN *in vitro*. Internalization of MBN was investigated by confocal images and FACS analysis. qPCR was performed to reveal Col1, BSP, OCN, DSPP and DMP-1 gene expression up to 21 days. Alkaline phosphate assay and Alizarin red staining were performed to detect mineralization of rDPSC.

**Results:** The cell viability did not change up to 50  $\mu\text{g/ml}$  MBG. The internalization of fluorescein-isothiocyanate (FITC) conjugated MBG was observed by confocal microscope images at 1 h incubation condition and FITC conjugated MBN was observed in ~95% of cells at 4 h incubation condition by flow cytometry experiments. Results from decrease of uptake after 1 h pretreatment of rDPSC with inhibitor of endocytosis showed internalization of MBN was performed by ATP dependent manner and majorly macropinocytosis. The mineralization capacity and expression of odontogenic-related proteins and genes (osteocalcin, dentin sialophosphoprotein, dentin matrix protein 1 and Collagen, type I, alpha 1) of rDPSC were significantly up-regulated with MBG, which was

explained by odontogenic ion release such as  $\text{Ca}^{2+}$  and  $\text{Si}^{4+}$  inside the cell.

**Conclusion:** This study reveals that MBG induces the odontogenic differentiation of dental pulp stem cells and may serve as a potential material for dentin regeneration.

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#### Effect of immediate or delayed dental sealing on bond strength



N. Scotti\*, F. Lerda, A. Comba, A. Boaglio, C. Ghiberto, E. Berutti

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**Purpose:** The aim of this *in vitro* study was to evaluate the influence of immediate or delayed adhesive system application on coronal dentin bond-strength. The null hypothesis is that immediate dental sealing (IDS) do not provide higher bond strength than delayed dental sealing (DDS), independently of the adhesive technique.

**Methods and materials:** 30 molars crowns were flattened and standardized smear layer was created with 600 grit paper. Samples were randomly divided in 6 groups according to adhesive treatment: G1: IDS with 3 step etch-and-rinse (Optibond FL, Kerr), G2: IDS with EDTA 10% for 1 min, TeethMate Desensitizer (Kuraray), water rinse for 10 s, 2 step self-etch adhesive application (Clearfil SE Bond 2, Kuraray). G3: IDS with 2 step self-etch adhesive application (Clearfil SE Bond 2). G4: DDS (after 8 weeks of storage in artificial saliva at 37 °C) with G1 procedure, G5: DDS with G2 procedure, G6: DDS with G3 procedure. Then 4 mm resin composite was applied and cured with LED lamp. Specimens were serially sectioned to obtain 1-mm-thick beams in accordance with the  $\mu\text{TBS}$  test technique. Beams were stressed to failure after 24 h. Two-way Anova was performed to evaluate the effects of immediate/delayed sealing and adhesive procedure on coronal bond strength.

**Results:** Two-way Anova showed that adhesive technique did not influenced immediate bond strength, while delayed bond strength was significantly worst when etch-and-rinse technique was employed ( $p = 0.0001$ ).

**Conclusion:** Null hypothesis was partially rejected since the IDS provided higher bond strength than DDS only when etch-and-rinse adhesive system was employed.

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### OCT evaluation of marginal seal of bulk-fill composites in dentin



A. Comba<sup>1,\*</sup>, G. Ventura<sup>1</sup>, A. Lugini<sup>3</sup>, M. Alovisi<sup>1</sup>, C. Alovisi<sup>2</sup>, E. Berutti<sup>1</sup>, N. Scotti<sup>1</sup>

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<sup>3</sup> Department of Life Sciences and Systems Biology, University of Turin, Italy

**Purpose:** The aim of this *in vitro* study was a non-invasive evaluation of the marginal sealing to dentin cervical margin of second class cavities after thermal aging using the optical coherence tomography (OCT). The null hypothesis is that there is no difference in marginal sealing between composites of different viscosity.

**Methods and materials:** 8 intact upper premolars, 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. Restoration was performed as follows: a 1 mm horizontal layer of bulk-fill composite (Venus Bulk-Fill, Heraeus Kulzer) was placed over only the mesio-cervical cavity floor and cured for 20 s. Then composite restoration of the cavity was performed with oblique layering of nanohybrid resin composite (Clearfill ES-2, Kuraray). OCT ('Spectralis', Heidelberg-Engineering, Germany) imaging was performed after 24 h and 2000 thermocycles. Selected samples were sectioned for interfacial observation by confocal laser scanning microscope (CLSM). Marginal adaptation (percentage) was analyzed on 20 B-scans through each sample. Images were analyzed with the software ImageJ to assess the percentage of marginal gap between composite and cervical margin. Data were statistically analyzed with ANOVA test and significance was set for  $p < 0.05$ .

**Results:** Statistical analysis showed any differences in marginal adaptation obtained with Venus Bulk-Fill and Clearfill. Thermal cycling significantly affected marginal integrity of tested composites ( $p = 0.001$ ). CLSM closely confirmed OCT findings in all samples.

**Conclusion:** The null hypothesis is accepted. Thermal aging significantly affected sealing of composites on dentin margin.

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### Histologic analysis of two dental implants extracted after osseointegration



V. Checchi<sup>1,\*</sup>, L. Checchi<sup>2</sup>, A. Mazzoni<sup>2</sup>, L. Breschi<sup>2</sup>, P. Felice<sup>2</sup>

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<sup>2</sup> University of Bologna, Italy

**Purpose:** The aim of this case report was to analyze and observe the bone tissue formation around two dental implants after their osseointegration.

**Methods and materials:** A 58 y.o. woman was referred to the Division of Periodontology and Implantology, Department of Biomedical and Neuromotor Sciences (University of Bologna, Italy) due to severe pain and discomfort caused by two of the four dental implants placed in the upper jaw (sites #13 and #23) in a private practice 8 months before. Implants were made of titanium, sandblasted with zirconium oxide and etched with mineral acids (Sweden & Martina, Due Carrare, Italy). At clinical and radiographic analysis, implants appeared properly osseointegrated but placed tilted way too buccally, with their emergence profile in non-keratinized oral mucosa. For the above-mentioned reasons, the patient started having discomfort immediately after the prosthetic load occurred four months after implants placement. The patient was rehabilitated with an implant supported removable prosthesis. The patient explains difficulties to perform adequate oral hygiene and declares pain every time she had to wear the implant-supported removable prosthesis. The implants were removed, preserving the bone around implant threads, and replaced with two new dental implants, placed in a prosthetic-guided correct inclination. The removed implants were processed for histological analysis in accordance with Checchi et al.

**Results:** The histological analysis revealed proper osseointegration of the implant fixture after eight months. Histological views of biopsies show good bone-implant contact recorded from the coronal side to the apical implant section. Presence of direct connecting bridges between the peri-implant bone trabeculae and the implant surface were recorded.

**Conclusion:** This case report clearly shows how after 8 months from implant placement, the fixtures coated with a rough surface were properly osseointegrated with good bone-implant contact recorded along their entire surface.

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### Discoloration of root canal by photosensitizers curcumin and methylene blue

G. Sivieri-Araujo<sup>1,\*</sup>, P.H. Santos<sup>2</sup>, L.T.A. Cintra<sup>1</sup>, J.E. Gomes-Filho<sup>1</sup>, E. Dezan-Junior<sup>1</sup>, R.C. Jacinto<sup>1</sup>, I.O.A. Queiroz<sup>1</sup>, D. Jacomassi<sup>3</sup>, C. Kurachi<sup>3</sup>, V.S. Bagnato<sup>3</sup>

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**Purpose:** Photodynamic therapy (PDT) is based on a set of physical, chemical, and biological procedures, which occur after the use of a photosensitizer activated by visible light of a specific wavelength, with the intention of destroying the target cell. The aim of this study was to evaluate *in vitro* the influence of methylene blue and curcumin, used as photosensitizers, in the capacity of discoloration of the root dentin.

**Methods and materials:** The dentin portion used was in the internal region of the cervical third of the root canal of human incisors (0.5 cm × 0.5 cm wide and 2 mm thick). After baseline color measurements, randomly selected specimens were immersed in photosensitizers and divided into 3 groups ( $n=20$ ): G1 – curcumin 1000 mg/ml; G2 – curcumin 1500 mg/ml; G3 – methylene blue 0.025 mg/ml, which were put into individual amber vials for 5 min, and immersed in 2 ml of the respective photosensitizer to be tested. The fragments were washed with distilled water, dried with absorbent paper and sealed with two layers of epoxy adhesive. The staining analysis of the fragments on the inner surface was accomplished through the use of reflectance spectrophotometer. Two color measurements were taken for each of the fragments with the aim of determining color stability, both in the initial and final periods. The difference of color values between the initial and final periods ( $\Delta E^*ab$ ) was established. The collected data were submitted to statistical analysis, ANOVA and Tukey test, in 5% of significance.

**Results:** G2 (curcumin 1500 mg/ml) showed the highest degree of color change of the root structures, showing Delta  $E^*$  index, followed by G3 (methylene blue 0.025 mg/ml).

**Conclusion:** The root canal structures can suffer discoloration according to the concentration and type of photosensitizer used.

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### Marginal quality of direct composite veneering system for cervical fillings

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**Purpose:** To evaluate the marginal adaptation of pre-fabricated nano-hybrid-composite enamel shells for direct veneering of class V cavities before and after thermo-mechanical loading compared to conventional composites. The null hypothesis tested was that marginal adaptation of the composite shells was not significantly different if compared to conventional composites before and after thermo-mechanical loading.

**Methods and materials:** Standardized Class V cavities (width 2.0 mm, length 3.0 mm, depth 1.5 mm) were prepared in 40 human premolars and molars at the cement–enamel junction. Cavity dimensions were standardized using a digital caliper. Teeth were divided in four groups ( $N=10$ ): Group 1: One Coat Self etching Bond (adhesive system, Coltene) with Synergy D6 (composite, Coltene) + composite shells (Componeer Class V, Coltene); Group 2: One coat Bond Self Etching with Synergy D6 flow (flowable composite, Coltene) + Componeer Class V; Group 3: Scotchbond Universal (adhesive system, 3M ESPE) with Filtek Supreme XTE (composite, 3M ESPE); Group 4: Scotchbond Universal with Filtek Supreme XTE flow (flowable composite, 3M ESPE). Restorations were placed according to manufacturers' instructions. Marginal quality was analyzed under a SEM using epoxy resin replicas before and after thermo-mechanical loading (240,000 mechanical cycles × 50 N with a frequency of 1HZ and 7800 thermo-cycles between 5° and 55 °C with chewing simulator CS4-Mechatronik). Data were statistically analyzed with two-way ANOVA and Tukey's post-hoc test ( $\alpha=0.05$ ).

**Results:** The null hypothesis was accepted since no significant statistical differences were found in marginal adaptation of Componeer Class V compared to margins obtained with conventional composites both before and after thermo-mechanical cycling ( $p>0.05$ ).

**Conclusion:** The marginal quality of prefabricated composite shells for direct veneering of class V cavities was comparable to that of conventional composites, both before and after thermo-mechanical cycling. Further clinical research is essential to confirm that the prefabricated composite shells for direct veneering can be a valid procedure to restore cervical lesions.

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<sup>3</sup> Physics Institute of São Carlos, University of São Paulo-USP, São Carlos, Brazil

**Purpose:** Photodynamic therapy (PDT) is based on a set of physical, chemical, and biological procedures, which occur after the use of a photosensitizer activated by visible light of a specific wavelength, with the intention of destroying the target cell. The aim of this study was to evaluate *in vitro* the influence of methylene blue and curcumin, used as photosensitizers, in the capacity of discoloration of the root dentin.

**Methods and materials:** The dentin portion used was in the internal region of the cervical third of the root canal of human incisors (0.5 cm × 0.5 cm wide and 2 mm thick). After baseline color measurements, randomly selected specimens were immersed in photosensitizers and divided into 3 groups ( $n=20$ ): G1 – curcumin 1000 mg/ml; G2 – curcumin 1500 mg/ml; G3 – methylene blue 0.025 mg/ml, which were put into individual amber vials for 5 min, and immersed in 2 ml of the respective photosensitizer to be tested. The fragments were washed with distilled water, dried with absorbent paper and sealed with two layers of epoxy adhesive. The staining analysis of the fragments on the inner surface was accomplished through the use of reflectance spectrophotometer. Two color measurements were taken for each of the fragments with the aim of determining color stability, both in the initial and final periods. The difference of color values between the initial and final periods ( $\Delta E^*ab$ ) was established. The collected data were submitted to statistical analysis, ANOVA and Tukey test, in 5% of significance.

**Results:** G2 (curcumin 1500 mg/ml) showed the highest degree of color change of the root structures, showing Delta  $E^*$  index, followed by G3 (methylene blue 0.025 mg/ml).

**Conclusion:** The root canal structures can suffer discoloration according to the concentration and type of photosensitizer used.

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### Marginal quality of direct composite veneering system for cervical fillings

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**Purpose:** To evaluate the marginal adaptation of pre-fabricated nano-hybrid-composite enamel shells for direct veneering of class V cavities before and after thermo-mechanical loading compared to conventional composites. The null hypothesis tested was that marginal adaptation of the composite shells was not significantly different if compared to conventional composites before and after thermo-mechanical loading.

**Methods and materials:** Standardized Class V cavities (width 2.0 mm, length 3.0 mm, depth 1.5 mm) were prepared in 40 human premolars and molars at the cement–enamel junction. Cavity dimensions were standardized using a digital caliper. Teeth were divided in four groups ( $N=10$ ): Group 1: One Coat Self etching Bond (adhesive system, Coltene) with Synergy D6 (composite, Coltene) + composite shells (Componeer Class V, Coltene); Group 2: One coat Bond Self Etching with Synergy D6 flow (flowable composite, Coltene) + Componeer Class V; Group 3: Scotchbond Universal (adhesive system, 3M ESPE) with Filtek Supreme XTE (composite, 3M ESPE); Group 4: Scotchbond Universal with Filtek Supreme XTE flow (flowable composite, 3M ESPE). Restorations were placed according to manufacturers' instructions. Marginal quality was analyzed under a SEM using epoxy resin replicas before and after thermo-mechanical loading (240,000 mechanical cycles × 50 N with a frequency of 1HZ and 7800 thermo-cycles between 5° and 55 °C with chewing simulator CS4-Mechatronik). Data were statistically analyzed with two-way ANOVA and Tukey's post-hoc test ( $\alpha=0.05$ ).

**Results:** The null hypothesis was accepted since no significant statistical differences were found in marginal adaptation of Componeer Class V compared to margins obtained with conventional composites both before and after thermo-mechanical cycling ( $p>0.05$ ).

**Conclusion:** The marginal quality of prefabricated composite shells for direct veneering of class V cavities was comparable to that of conventional composites, both before and after thermo-mechanical cycling. Further clinical research is essential to confirm that the prefabricated composite shells for direct veneering can be a valid procedure to restore cervical lesions.

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### Effectiveness of silane coupling agent incorporated in universal adhesive



K. Yoshihara<sup>1,\*</sup>, N. Nagaoka<sup>1</sup>, M. Irie<sup>1</sup>, A. Sonoda<sup>2</sup>, Y. Makita<sup>2</sup>, Y. Yoshida<sup>3</sup>, B. Van Meerbeek<sup>4</sup>

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**Purpose:** 'Universal' adhesives can be employed following the adhesive procedure of preference ('etch-and-rinse'; 'self-etch') for both direct and indirect indications. Regarding its indirect application, Scotchbond Universal (3M ESPE) incorporates a silane coupling agent in order to chemically bond to glass-rich ceramics without the use of a separate ceramic primer. Silane bifunctional monomers are known to be sensitive to hydrolysis and dehydration condensation. With this work, the effectiveness/stability of the silane coupling function of Scotchbond Universal using a shear bond-strength test protocol was investigated.

**Methods and materials:** The experimental groups involved the application of (1) Scotchbond Universal (3M ESPE), (2) a conventional self-etch adhesive Clearfil S3 ND Quick (Kuraray Noritake), (3) Scotchbond Universal following the application of the water-free, non-acidic silane coupling agent Porcelain Activator (Kuraray Noritake), (4) Clearfil S3 ND Quick following Porcelain Activator, (5) Scotchbond Universal to which 2% 3-methacryloxypropyltrimethoxysilane (3-MPTS, Sigma-Aldrich) was added, and (6) Clearfil S3 ND Quick containing 2% 3-MPTS. Silica glass plates were treated following the abovementioned experimental protocols, followed by air-drying. Zirconia blocks were bonded using the composite cement Clearfil Esthetic Cement (Kuraray Noritake) to the treated glass plates, prior to being subjected to a shear bond-strength test. The two 3-MPTS containing adhesives, prepared following the experimental protocols 5 and 6, were applied immediately upon mixing, but also 1 day, 3 and 7 days after mixing. One-way ANOVA and Tukey-Kramer test were used to statistically analyze the bond-strength data ( $\alpha=0.05$ ).

**Results:** The shear bond strength of Scotchbond Universal was not different from that of Clearfil S3 ND quick. When Porcelain activator was beforehand used, significantly higher bond strength was recorded. The two 3-MPTS containing adhesives revealed significantly higher bond strength only when applied immediately after mixing; delayed application (after 1, 3 or 7 days) resulted in significantly lower bond strength.

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### Dentin substituting material for in vitro biomechanical trials: Preliminary results



P. Baldissara<sup>1,\*</sup>, A. Casamenti<sup>1</sup>, V.F. Wandscher<sup>2</sup>, A.M.E. Marchionatti<sup>2</sup>, C. Parisi<sup>1</sup>

<sup>1</sup> University of Bologna, Italy

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**Purpose:** To develop a material that has the mechanical and adhesive properties of dentin and that can be milled with CAD/CAM technology in order to use it as substrate for laboratory testing of CAD/CAM fabricated prostheses.

**Methods and materials:** An epoxy matrix was tested for its mechanical properties and filled with reinforcement fibers to reach dentin Young's modulus ( $E_f=18$  GPa) and flexural strength ( $s_f=300$  MPa). The fillers used were woven carbon fiber, woven glass fiber and hydroxyapatite sub-micron particles. Two groups were performed: (1) w/o-Hap: hybrid glass-carbon reinforced composite without hydroxyapatite and (2) w-HAP: hybrid glass-carbon reinforced composite with hydroxyapatite. Three-point-bending flexural test was conducted for each specimen, and the modulus of elasticity  $E_f$  and flexural strength  $s_f$  were calculated. Shear bond strength (SBS) with self-adhesive luting cement (RelyX Unicem, 3M-ESPE) was evaluated after dry and wet (short-term, 24 h) storage.

**Results:** t-Student test showed that group (1) presented higher flexural strength and Young's modulus values ( $447.7 \pm 35.4$  and  $17.8 \pm 1.25$  respectively) compared to group (2):  $391.6 \pm 22.9$  and  $16.1 \pm 0.7$ , respectively. t-Student test showed statistically significant difference between paired comparisons: group (1) dry vs. group (2) dry ( $8.96 \pm 1.86$  and  $14.65 \pm 4.80$ , respectively); group 1) wet vs. group 2) wet ( $10.90 \pm 3.22$  and  $17.28 \pm 3.31$  respectively); Group (1) dry vs. wet ( $8.96 \pm 1.86$  and  $10.90 \pm 3.22$  respectively) and group (2) dry vs. wet ( $14.65 \pm 4.80$  and  $17.28 \pm 3.31$ , respectively) showed no statistical significant differences ( $\alpha=0.05$ ).

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### Chemical-physical properties of innovative polysiloxane-guttapercha calcium silicate-containing systems



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<sup>1</sup> Dental School, University of Bologna, Italy

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**Purpose:** Calcium silicate cements (CaSi) showed remarkable clinical outcomes when used as filling-sealing materials in root-resection and root-perforation repair in relationship to their ability to seal in fluid-contaminated environments and to form apatite in situ. Root canal filling

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**Purpose:** Calcium silicate cements (CaSi) showed remarkable clinical outcomes when used as filling-sealing materials in root-resection and root-perforation repair in relationship to their ability to seal in fluid-contaminated environments and to form apatite in situ. Root canal filling

system based on polydimethylpolymethylhydrogensiloxane (RoekoSeal) or polydimethylpolymethylhydrogensiloxane guttapercha (GuttaFlow) are in the market and showed advantages in handiness and clinical application. Experimental calcium silicate sealers, have been innovatively proposed by Gandolfi et al. (2010), and now some calcium silicate-based sealer is present on the market (MTA Fillapex).

**Methods and materials:** Two experimental formulations of PGCaSi sealers were examined and compared with GuttaFlow2 (Coltène/Whaledent, Switzerland). RoekoSeal (Coltène/Whaledent, Switzerland) and MTA Fillapex (Angelus dental solutions, Brazil) represented guttapercha-free comparison materials. Calcium release, alkalizing activity, setting time, water sorption, open and impervious porosity and apparent porosity, and weight loss were evaluated. ESEM/EDX analyses of fresh materials and after soaking in simulated a body fluid were also performed.

**Results:** The materials showed evident differences in calcium release and alkalizing activity, as highest calcium release for MTA Fillapex and no alkalizing activity for RoekoSeal (see Table 1). Huge differences were obtained in initial (range from 25 to 130 min) and final (range from 45 to 270 min) setting times; namely Fillapex showed the highest values and the experimental PGCaSi the shortest. Wide differences in porosity, water sorption and solubility were obtained. ESEM-EDX of fresh materials showed the constitutive elements of the materials whilst different amount of calcium phosphate deposits were detected on the surface; higher deposits were displayed by the experimental PGCaSi.

**Conclusion:** CaSi particles may co-operate with polysiloxane (Si-O-Si) groups and improve the biointeractive properties of the filling system. The incorporation of CaSi into polydimethylpolymethylhydrogensiloxane guttapercha sealers may represent a new frontier for endodontic therapy to obtain bioactive biointeractive flowable guttapercha, likely helpful to favour the regeneration of the damaged apical tissues such as periodontal ligament, cementum and periapical bone when applied in the root canal and/or extruded.

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### AGF(NH<sub>3</sub>)<sub>2</sub>F treated dentin/epoxy-based cement bond strength and Ag remaining evaluations

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**Purpose:** To evaluate the bond strength of an epoxy base cement/treated dentin with different concentrations of AgF(NH<sub>3</sub>)<sub>2</sub>F and time, with a push-out evaluation and MEV/EDX of remaining Ag

**Methods and materials:** 60 extracted human premolars instrumented endodontically until F5 (PtoTaper), irrigated with 2.5% NaOCl and in the end with 17% EDTA. The premolars were divided in 6 groups (n=10) according to Ag(NH<sub>3</sub>)<sub>2</sub>F

concentration and time (24 and 72 h): G1 – saline/24 h; G2 – Ag(NH<sub>3</sub>)<sub>2</sub>F 12%/72 h; G3 – Ag(NH<sub>3</sub>)<sub>2</sub>F 12%/24 h; G4 – Ag(NH<sub>3</sub>)<sub>2</sub>F 12%/72 h; G5 – Ag(NH<sub>3</sub>)<sub>2</sub>F 3,8%/24 h; G6 – Ag(NH<sub>3</sub>)<sub>2</sub>F 3,8%/72 h. Ag(NH<sub>3</sub>)<sub>2</sub>F was applied to F5 absorbent paper cone. In the 72 h Groups, the medication was changed every 24 h. Then, the roots were cleaved in BL sense and a specimen was analyzed in EDX and the other in SEM. Images of each specimen were obtained at 500×, middle-cervical segment and the mid-apical, to assess the presence Ag, being evaluated by scores. Another 60 teeth were prepared the same way and F5 gutta percha cones were cemented with epoxy base cement and roots sectioned transversely into thirds and submitted to the push-out test, with a load of 5 kN and speed of 0.5 mm/min. The types of fractures were classified into loupe (30× increase). The scores obtained were submitted to the Kruskal-Wallis test and the push-out the ANOVA (*p* < 0.05).

**Results:** Minor amounts of Ag residues were found in Groups 2–6. Ca and P values were similar for all groups. The bond strength of different groups did not differ statistically.

**Conclusion:** The treatment with Ag(NH<sub>3</sub>)<sub>2</sub>F did not influence the bond strength and the amount of residual silver was concentration/time dependent.

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### Synthesis of electrospun bioactive carbon nanotube based dental resin-based composites



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Interdisciplinary Research Centre in Biomedical Materials, COMSATS Institute of Information Technology, Lahore, Pakistan

**Purpose:** The aim of this study was to synthesize electrospun carbon nanotube/nano-hydroxyapatite (CNT/nHA) reinforced dental composites and the objective was to increase the mechanical properties of the restorative material.

**Methods and materials:** Commercial multi-walled CNTs (MWCNT) were heat treated to remove the amorphous CNTs, later functionalized with nitric acid (HNO<sub>3</sub>) and refluxed the mixture in microwave oven for 10 min (30 s on: 30 s off). For further oxidative treatment 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) was mixed with HNO<sub>3</sub> treated CNT. In situ precipitation of nHA was performed with MWCNTs. The precursors for nHA were calcium nitrate tetrahydrate and diammonium hydrogen phosphate. MWCNT were mixed in calcium precursor and the mixture was refluxed in microwave oven at 700 W for 10 min (15 s on: 15 s off). After ageing at 80 °C for 24 h, the resulting material was heat treated at 1100 °C. Microwaves augment the synthesis, enhance the reaction rate, and institute energy savings. The ceramic composite was characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Scanning Electron Microscopy (SEM). Cylindrical discs were made for mechanical testing. The resulting material was silanized with 3-methacryloxypropyl-trimethoxysilane (MPS) and mixed with polyvinyl alcohol (PVA) with the ratio of 10, 20, and 40 wt/vol%. The solution was electrospun at 20 kV with



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**Methods and materials:** Two experimental formulations of PGCaSi sealers were examined and compared with GuttaFlow2 (Coltène/Whaledent, Switzerland). RoekoSeal (Coltène/Whaledent, Switzerland) and MTA Fillapex (Angelus dental solutions, Brazil) represented guttapercha-free comparison materials. Calcium release, alkalizing activity, setting time, water sorption, open and impervious porosity and apparent porosity, and weight loss were evaluated. ESEM/EDX analyses of fresh materials and after soaking in simulated a body fluid were also performed.

**Results:** The materials showed evident differences in calcium release and alkalizing activity, as highest calcium release for MTA Fillapex and no alkalizing activity for RoekoSeal (see Table 1). Huge differences were obtained in initial (range from 25 to 130 min) and final (range from 45 to 270 min) setting times; namely Fillapex showed the highest values and the experimental PGCaSi the shortest. Wide differences in porosity, water sorption and solubility were obtained. ESEM-EDX of fresh materials showed the constitutive elements of the materials whilst different amount of calcium phosphate deposits were detected on the surface; higher deposits were displayed by the experimental PGCaSi.

**Conclusion:** CaSi particles may co-operate with polysiloxane (Si-O-Si) groups and improve the biointeractive properties of the filling system. The incorporation of CaSi into polydimethylpolymethylhydrogensiloxane guttapercha sealers may represent a new frontier for endodontic therapy to obtain bioactive biointeractive flowable guttapercha, likely helpful to favour the regeneration of the damaged apical tissues such as periodontal ligament, cementum and periapical bone when applied in the root canal and/or extruded.

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### AGF(NH<sub>3</sub>)<sub>2</sub>F treated dentin/epoxy-based cement bond strength and Ag remaining evaluations

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**Purpose:** To evaluate the bond strength of an epoxy base cement/treated dentin with different concentrations of AgF(NH<sub>3</sub>)<sub>2</sub>F and time, with a push-out evaluation and MEV/EDX of remaining Ag

**Methods and materials:** 60 extracted human premolars instrumented endodontically until F5 (PtoTaper), irrigated with 2.5% NaOCl and in the end with 17% EDTA. The premolars were divided in 6 groups (n=10) according to Ag(NH<sub>3</sub>)<sub>2</sub>F

concentration and time (24 and 72 h): G1 – saline/24 h; G2 – Ag(NH<sub>3</sub>)<sub>2</sub>F 12%/72 h; G3 – Ag(NH<sub>3</sub>)<sub>2</sub>F 12%/24 h; G4 – Ag(NH<sub>3</sub>)<sub>2</sub>F 12%/72 h; G5 – Ag(NH<sub>3</sub>)<sub>2</sub>F 3,8%/24 h; G6 – Ag(NH<sub>3</sub>)<sub>2</sub>F 3,8%/72 h. Ag(NH<sub>3</sub>)<sub>2</sub>F was applied to F5 absorbent paper cone. In the 72 h Groups, the medication was changed every 24 h. Then, the roots were cleaved in BL sense and a specimen was analyzed in EDX and the other in SEM. Images of each specimen were obtained at 500×, middle-cervical segment and the mid-apical, to assess the presence Ag, being evaluated by scores. Another 60 teeth were prepared the same way and F5 gutta percha cones were cemented with epoxy base cement and roots sectioned transversely into thirds and submitted to the push-out test, with a load of 5 kN and speed of 0.5 mm/min. The types of fractures were classified into loupe (30× increase). The scores obtained were submitted to the Kruskal-Wallis test and the push-out the ANOVA (*p* < 0.05).

**Results:** Minor amounts of Ag residues were found in Groups 2–6. Ca and P values were similar for all groups. The bond strength of different groups did not differ statistically.

**Conclusion:** The treatment with Ag(NH<sub>3</sub>)<sub>2</sub>F did not influence the bond strength and the amount of residual silver was concentration/time dependent.

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### Synthesis of electrospun bioactive carbon nanotube based dental resin-based composites



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**Purpose:** The aim of this study was to synthesize electrospun carbon nanotube/nano-hydroxyapatite (CNT/nHA) reinforced dental composites and the objective was to increase the mechanical properties of the restorative material.

**Methods and materials:** Commercial multi-walled CNTs (MWCNT) were heat treated to remove the amorphous CNTs, later functionalized with nitric acid (HNO<sub>3</sub>) and refluxed the mixture in microwave oven for 10 min (30 s on: 30 s off). For further oxidative treatment 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) was mixed with HNO<sub>3</sub> treated CNT. In situ precipitation of nHA was performed with MWCNTs. The precursors for nHA were calcium nitrate tetrahydrate and diammonium hydrogen phosphate. MWCNT were mixed in calcium precursor and the mixture was refluxed in microwave oven at 700 W for 10 min (15 s on: 15 s off). After ageing at 80 °C for 24 h, the resulting material was heat treated at 1100 °C. Microwaves augment the synthesis, enhance the reaction rate, and institute energy savings. The ceramic composite was characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Scanning Electron Microscopy (SEM). Cylindrical discs were made for mechanical testing. The resulting material was silanized with 3-methacryloxypropyl-trimethoxysilane (MPS) and mixed with polyvinyl alcohol (PVA) with the ratio of 10, 20, and 40 wt/vol%. The solution was electrospun at 20 kV with

