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| <b>Title</b>       | <b>Tensile properties of demineralized dentin matrix after 48 months</b>   |
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Confocal examination of *in-vitro* ceramic wear and sub-surface microstructural cracking. METMAN<sup>1</sup>, T.F. WATSON, M.J. WOOLFORD (Conservative Dentistry, Guy's, King's & St Thomas' Dental Institute, KCL, London, UK)

The original ceramic surface finish and its microstructure may have an effect on wear mechanisms as well as enamel loss. Twenty (8x4x2mm) blocks of porcelain, All-ceram (Ducera, Germany) and leucite reinforced glass ceramic Sensation SL (Dilton Com., USA), 10 glazed and 10 polished of each, were fabricated following the manufacturer's instructions. Ten equivalent enamel specimens were also prepared as a control group. Fifty antagonist enamel specimens were made from the labial surfaces of permanent incisors. The abraders were attached to a wear simulating machine so that each enamel specimen presented at 45° to the vertical movement of the abraders; they were immersed in artificial saliva. Wear occurred at 60 cycles/min. with a load of 40N / 2mm horizontal deflection. Silicone impressions were taken at baseline, 5000, 10K, 20K, 40K, & 80K cycles for digitising using a non contact co-ordinate measuring machine, whilst the actual samples were examined for cracks using fluorescence and reflected light confocal microscopy. Wear tracks were rapidly produced (>5000 cycles) on both the glazed and unglazed samples, whilst 20-30 µm deep sub-surface cracking appeared in the leucite reinforced ceramic, with 8-10 µm depth in the All-ceram material. Enamel against enamel showed smooth surface wear, but marked surface roughness against the ceramics; however, no enamel cracking was evident. Neither glazed nor polished ceramic surfaces remained intact during wear. Tooth against tooth contact caused smooth surface wear. The worn ceramic materials showed sub-surface cracking. MRCJREIgrant G9817920

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Shear Bond Strengths of Composite Resin to Porcelain. W. P. KELSEY,\* M. A. LATTA C. M. STANISLAV and W. W. BARKMEIER (Creighton University School of Dentistry, Omaha, Nebraska, USA)

Porcelain restorations have become popular for the restoration of teeth. Repair of fractures with composite resin frequently results in separation of the repaired segment. It was the purpose of this study to evaluate three resin-based products for suitability to repair porcelain as measured by shear bond strengths (SBS) of attachment. Specimens of porcelain were grit-blasted with 50 micron aluminum oxide and divided into four groups (n=20). Composite resin cylinders were then bonded accordingly: Group 1 Control (no further treatment); Group 2 Photobond (Kuraray); Group 3 SE Bond (Kuraray); and Group 4 Singlebond (3M Dental Products). Manufacturer's instructions regarding surface conditioning and adhesive application were strictly followed. Specimens of each group were placed in 37° C deionized water. Half were debonded after 24 hours and the remainder were stored for 30 days and thermocycled 1000 times between water baths maintained at 5° C and 55° C prior to debonding. Mean SBS were determined for each group. Two way ANOVA and Tukey's post-hoc testing were applied to evaluate the differences between the groups. Debonding was accomplished with an Instron Testing Machine operating with a crosshead speed of 5mm/min. The mean 24 hour SBS (MPa) were: Group 1 = 9.0 ± 1.7; Group 2 = 16.8 ± 3.3; Group 3 = 24.0 ± 2.7; and Group 4 = 12.2 ± 1.0. The 30 day mean SBS (MPa) were: Group 1 = 2.4 ± 1.4; Group 2 = 17.4 ± 3.8; Group 3 = 16.6 ± 3.5; and Group 4 = 3.3 ± 2.2. SE Bond and Photobond generated significantly higher SBS of composite resin to porcelain when compared to Singlebond and the controls at either storage period. The mean SBS values observed tended to decrease following extended storage and thermocycling. Clear discrimination among the performance of these materials will require clinical testing. Supported by Kuraray Co., Ltd.

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Bond Strength of Composite To Enamel Using Three Adhesive Conditioners. M.A. LATTA\*, C.M. STANISLAV and W.W. BARKMEIER (Creighton University School of Dentistry, Omaha, Nebraska USA).

While most adhesive systems rely on an acid treatment prior to application of an adhesive primer, some newer systems employ a no-rinse "self-etching" primer. The purpose of this study was to evaluate the shear bond strength (SBS) of composite to both ground and intact enamel using both phosphoric acid and self-etching primer systems. 72 human incisor teeth were prepared either by wet grinding with 600 grit silicon carbide paper or by polishing the surface. Bonded assemblies of Filtek Z-250 (CR) were prepared using a gelatin capsule matrix. 3 groups each containing 12 specimens with intact enamel and 3 groups of 12 of ground enamel were prepared as follows: Group A- 35% phosphoric acid conditioning followed by Singlebond adhesive and CR, Group B-no acid conditioning, Clearfil SE and CR, Group C-no acid conditioning, Prompt LP-2 conditioner and CR. Specimens in each group were debonded after water storage for 24 hours at 37° C using an Instron Model 1123 testing machine with a crosshead speed of 5 mm/min. Statistical analysis included a two-way ANOVA (adhesive system, substrate) and Tukey's post-hoc test for pairwise comparisons. Scanning electron microscopy (SEM) was performed on representative surfaces from each group. Mean SBS (MPa) were:

|               | Group A    | Group B    | Group C    |
|---------------|------------|------------|------------|
| Intact enamel | 21.9 ± 4.1 | 16.6 ± 5.4 | 17.3 ± 5.1 |
| Ground enamel | 37.2 ± 4.3 | 28.1 ± 3.1 | 24.1 ± 4.2 |

SBS in ground enamel was higher (p<0.05) than intact enamel for each group. There was no statistical difference (p>0.05) for SBS among the groups on ground enamel. Group A was statistically equal (p>0.05) to Group C but greater (p<0.05) than Group B on intact enamel. SEM revealed a more uniform and deeper etch pattern in all ground groups compared to intact enamel groups. The no-rinse "self-etching" primers generated similar SBS to ground enamel compared to phosphoric acid. However SBS to intact enamel was lower with the self-etching systems compared to phosphoric acid. The clinical relevance of these findings needs investigation. This study was supported by ESPÉ and the Health Future Foundation.

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Tensile Properties of Demineralized Dentin Matrix After 48 Months. R.M. CARVALHO\*, F. TAY, H. SANO, M. YOSHIYAMA, D.H. PASHLEY (FOB USP, Brazil, U of Hong Kong, Hokkaido U, U of Tokushima, MCG, USA).

Incomplete resin infiltration of the demineralized zone during hybrid layer formation may leave the collagen matrix exposed to the degrading action of water and hydrolytic enzymes. Storage of demineralized dentin in saline for 18 months did not cause any significant decrease of its tensile properties (Carvalho et al. JDR 77: 168, Abst. 501). The purpose of this study was to further investigate the ultimate tensile strength (UTS) and modulus of elasticity (E) of demineralized dentin stored for up to 48 months in saline. Dentin sticks (0.7x0.7x8.0 mm) were obtained from the crowns of extracted human third molars. The ends of the specimens were covered with resin composite and the center section (4.0 mm) was demineralized in 0.5 M EDTA for four days, washed in distilled water and kept in saline at room temperature (25°C) until tested in tension in a Vitrodyne tester after 24 hrs. (control), 18 and 48 months at 0.6 mm/min. Tests were performed while the specimens were immersed in saline. The 48 months failed specimens were examined by TEM. The results were: MPa ± SD (N).

| Tensile Properties | 24 hrs          | 18 months       | 48 months        |
|--------------------|-----------------|-----------------|------------------|
| UTS                | 10.8±0.3 (10)ab | 13.8±4.8 (10)a  | 7.9±3.5 (14)b    |
| E                  | 58.4±16.7 (10)c | 85.1±20.4 (10)d | 68.9±20.3 (14)cd |

The tensile properties of demineralized dentin matrix after 48 months of storage were not statistically different from the values obtained at 24 hrs., (p > 0.05). Storage of demineralized dentin matrix for 48 months in saline did not cause any decrease of its tensile properties. Supported by grant DE 06427 from NIDCR and CNPq 300481/95-0.

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Bonding Mechanism and Micro-Tensile Bond Strength of a 4-MET-based Self-Etching Adhesive. B. VAN MEERBEEK<sup>1</sup>, Y. YOSHIDA<sup>2</sup>, S. INOUE<sup>3</sup>, M. VARGAS<sup>4</sup>, Y. ABÉ<sup>1</sup>, R. FUKUDA<sup>1</sup>, M. OKAZAKI<sup>1</sup>, P. LAMBRECHTS<sup>1</sup>, G. VANHERLE<sup>1</sup> (Catholic University of Leuven, Belgium; <sup>2</sup>Hiroshima and <sup>3</sup>Hokkaido University, Japan; <sup>4</sup>University of Iowa, USA)

Self-etching adhesives are clinically less technique-sensitive because they do not require a rinsing step in their application procedure. The aim of this study was to analyze the mechanism of bonding of a two-step 4-MET-based self-etching adhesive (UniFil Bond, GC) to dentin ultra-morphologically by TEM and AFM, and chemically using XPS. In addition, the micro-tensile bond strength (µTBS) of UniFil Bond was measured to dentin on three depth levels. TEM and AFM showed that the self-etching effect was limited to the formation of a hybrid layer with a thickness of about 0.5 µm. Inside this hybrid layer, individual collagen fibrils were hardly detectable, whereas residual hydroxyapatite (HA) crystals were omnipresent. XPS of HA exposed to a 4-MET solution revealed a significant shift of the peak representing -COO- groups to a lower binding energy. This shift suggests ionic bonding of the carboxyl groups of 4-MET to HA. A relatively high micro-tensile bond strength of 51.4 ± 16.8 MPa to dentin was recorded, which did not vary upon remaining dentin thickness and was not

| µTBS in MPa           | Deep dentin (<2 mm) | Middle dentin (2-3 mm) | Superficial dentin (>3 mm) |
|-----------------------|---------------------|------------------------|----------------------------|
| UniFil Bond           | 44.5 ± 13.2 (n=7)   | 53.5 ± 14.4 (n=7)      | 56.3 ± 21.7 (n=7)          |
| OptiBond FL (control) |                     | 65.5 ± 9.4 (n=7)       |                            |

statistically different from the control (student's t-test: NS at P=0.05). It is concluded that the bonding mechanism of UniFil Bond to dentin is ionic. Micro-mechanical bonding was established by monomer interdiffusion into a shallow partially demineralized dentin layer. Chemical bonding was obtained by ionic interaction of the carboxyl groups of 4-MET with calcium of HA that remained around collagen. How important each of the two mechanisms is in terms of the final bond strength and stability needs to be further investigated. B.V.M. is Postdoctoral Research Fellow with the Fund for Scientific Research of Flanders.

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Microtensile Bond Strength of Glass Ionomer Cement to Dentine. M. TANUMIHARJA, M. F. BURROW, M. J. TYAS\* (Uni of Melbourne, Australia)

Various pretreatments have been recommended prior to the placement of glass ionomer cements (GICs). This study evaluated the effect of Ketac Conditioner (Espc; 25% polyacrylic acid (PAA)), Dentin Conditioner (GC; 10% PAA), Cavity Conditioner (GC; 20% PAA, 3% aluminium chloride), and an experimental conditioner K930 (GC; 12% citric acid, 4% Al chloride) on the microtensile bond strength to human dentine of a self-cure GIC (Fuji IX GP, GC) and two resin-modified GICs (Fuji II LC, GC; Photac Fil Quick, Espc). Specimens were stored in water (24 h/37°C), shaped in an 'hour-glass' form of (1.2 ± 0.2) mm dia. and stressed in tension at a cross-head speed of 1 mm/min.

|                    | Bond strength (SD), MPa (N=10) |             |             |
|--------------------|--------------------------------|-------------|-------------|
|                    | Photac Fil Quick               | Fuji II LC  | Fuji IX GP  |
| Control            | 21.6 (5.2)a                    | 15.0 (2.9)b | 7.5 (1.7)d  |
| Ketac Conditioner  | 20.2 (5.5)a                    | 22.8 (4.8)c | 8.2 (2.6)d  |
| Dentin Conditioner | 17.9 (3.6)a                    | 21.8 (5.9)c | 8.5 (2.9)d  |
| Cavity Conditioner | 19.3 (4.7)a                    | 18.5 (4.0)c | 10.8 (4.0)d |
| K-930              | 19.0 (2.7)a                    | 20.2 (6.0)c | 8.5 (2.5)d  |

Values with the same letter are not significantly different (P > 0.05). Microtensile test values are higher than conventional tensile values. The lack of effect of conditioner on the bond strengths of Photac Fil Quick and Fuji IX GP may be because the free acids in the mixed cement act as self-conditioners. For Fuji II LC, however, a preconditioning step appears necessary. It is concluded that the need for conditioning is variable, and depends on the particular GIC used. Supported by the Australian Dental Research Foundation.

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Fractographic analysis of dentin bonded with a moist or dry technique after microtensile bond testing. CKY YIU<sup>1</sup>, NM KING<sup>2</sup>, FR TAY<sup>3</sup>, DH PASHLEY<sup>1</sup> (University of Hong Kong, Hong Kong SAR; <sup>2</sup>Medical College of Georgia, USA)

This study tested the null hypothesis that application of simplified-step adhesives with a moist or a dry bonding technique produce the same failure modes following a non-rinsing<sup>1</sup> microtensile bond-testing (µTBS) method. Eight extracted, caries-free, human third molars were divided into four groups. The occlusal enamel was removed, leaving a flat dentin surface for bonding. Resin composite buildups were made after the acid-conditioned dentin was bonded with either Single Bond (SB) or One-Step (OS), and using either moist bonding or air-drying for 5s. After stored in water for 24h, the teeth were vertically sectioned into an array of 0.9mm x 0.9mm composite-dentin beams. Two teeth from each group yielded between 42 - 48 beams for bond testing. Following initial classification of the failure modes with a stereoscopic microscope, fractured dentin and composite sides of representative beams from each group were prepared for scanning (SEM) and transmission electron microscopy (TEM). Results: µTBS for SB moist: 60.75 ± 12.03 MPa; SB dry: 25.74 ± 6.66 MPa; OS moist: 57.21 ± 12.30 MPa; OS dry: 27.10 ± 8.45 MPa. A two-way ANOVA showed significant difference in the effect of bonding technique (moist vs dry; p<0.001) but not of the adhesives (SB vs OS; p=0.547) on tensile bond strength, and that the effect of different techniques was independent of the adhesives used (p=0.201). SEM analysis showed that interfacial failure occurred exclusively in both dry groups. Splitting of the incompletely infiltrated hybrid layer occurred, part of which was retained on the composite side of the fractured beams. TEM of the dentin side of the fractured beams further revealed mechanical denaturing of the surface collagen. Interfacial failures above the hybrid layer demonstrated abnormally large interfibrillar spaces within the hybrid layer that suggested plastic deformation of the collagen. In both moist bonding groups, interfacial, mixed and substrate failures could be observed. It is concluded that suboptimal resin infiltration of the demineralized collagen in dry bonding results in a weakened zone in which the incompletely infiltrated collagen are either mechanically denatured or plastically deformed during the application of tensile stress. (Supported, in part, by DE06427 from the NIDCR)

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Adhesion of contemporary glass ionomer cements used in sound dentin. HK YIP<sup>1</sup>, FR TAY<sup>2</sup>, H NGO<sup>3</sup>, RJ Smales<sup>4</sup> and DH Pashley<sup>1</sup> (The University of Hong Kong, Hong Kong SAR; <sup>2</sup>The University of Adelaide, South Australia; <sup>3</sup>Medical College of GA, Augusta, USA)

This work investigated the microtensile bond strength (µTBS) of contemporary glass ionomer cements (GIC) to sound coronal dentin. The coronal enamel of extracted human third molars were removed, leaving flat dentin surfaces for placement of the GICs. Three teeth were prepared for each material tested: ChemFlex (Dentsply), Fuji IX (GC), Ketac-Molar (Espc). GIC buildups were made according to the manufacturer's instructions. After being stored at 100% humidity for 24 h, the teeth were vertically sectioned into an array of 0.9mm x 0.9mm beams. µTBS evaluation was performed using a Bencor Multi-T testing device, in an Instron machine. Following initial classification of the failure modes with optical microscope, representative fractured beams were prepared for scanning (SEM) and transmission microscopic (TEM) examination. Results of the µTBS evaluation: ChemFlex (14.97 ± 9.29 MPa), Fuji IX GP (12.37 ± 8.64 MPa) and Ketac-Molar (11.37 ± 7.67 MPa). One way ANOVA and SNK multiple comparison test showed that ChemFlex has a statistically higher µTBS (p<0.05). Regional mapping revealed significant variation of µTBS within each tooth, but no consistent trend could be found. SEM fractographic analysis revealed that the predominant failure modes were either mixed failures or cohesive within GICs. The GIC side of the fractured beams revealed significant dehydration cracks and numerous voids. In addition, voids with an eggshell-like crust were present, probably representing desiccated, unreacted polyacrylic acid droplets that were dispersed around the periphery as a result of surface tension. TEM of demineralized dentin side of the fractured beams revealed glass particles that were surrounded by an interparticulate matrix. An interaction zone containing resinian layer debris could be seen between the GIC and demineralized dentin collagen. The findings suggested the bonding of GIC to dentin is not weak and that the µTBS values probably represented the weak cohesive strength of investigated GICs under tension. (supported by CRC Grant 10201258, The University of Hong Kong and DE06427, NIDCR)