

**kuraray**



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**COMPILATION  
OF SCIENTIFIC  
ABSTRACTS**

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[Kuraray Europe GmbH](#)

BU Medical Products | Hoechst Industrial Park | Building F 821  
65926 Frankfurt am Main | Germany

Phone: +49 (0) 69 305 35825 | Fax: +49 (0) 69 305 98 35825  
E-mail: [dental@kuraray.eu](mailto:dental@kuraray.eu) | [www.kuraray-dental.eu](http://www.kuraray-dental.eu)

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# COMPANY PROFILE



## More than 80 years of Kuraray

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Kuraray Co. Ltd., established in 1926 in Kurashiki, Japan, was originally involved in the industrial production of fibers out of viscose. Today, thanks to Kuraray's technological strength and comprehensive experience, the company successfully covers the sectors polymer chemistry, chemical synthesis, and chemical engineering developing and producing a broad range of high-quality and innovative products.

In 1973, Kuraray entered the business field of dental materials with the objective to respond to requirements of dental practice precisely and carefully – with products which convince users by their reliability and high quality.

## Inventor of the bonding system

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In 1978, Kuraray introduced the first bonding system to the market: CLEARFIL™ BOND SYSTEM-F, the start of the age of adhesive dentistry. At the same time, the company developed the total-etch technique for enamel and dentin.

Today, Kuraray continues to steadily produce innovative quality products which meet the requirements of a profession that also develops constantly. Its products that make history – such as PANAVIA™ F2.0, CLEARFIL™ PROTECT BOND, CLEARFIL™ SE BOND, CLEARFIL™ AP-X and ESTENIA™ C&B – are proof of Kuraray's capability to develop solutions for practice from the results of their pioneering research.

## Our dedication

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As science and society continue to develop, new questions and challenges also arise for dental materials. Thus, Kuraray has set itself the goal of meeting demands and requirements of dentistry to the very best of its ability, now and in the future.

With this compilation of abstracts presented at IADR, the 88th General Session of the International Association of Dental Research in Barcelona, Spain, Kuraray is delighted to present the most recent and informative scientific information on our clinically tested and evaluated products.

Dedicated to develop and produce high quality products, the external verification of the products' quality is vital for us. Hence, Kuraray expresses its gratitude to the universities for including Kuraray's products in their research.

Please feel invited to contact us in case of questions –  
We are happy to provide even more information.



Dr. Heinz Schuh  
Head of BU Medical Products  
Kuraray Europe GmbH



Dr. Hiroshige Ishino  
Head of Technical Services & Marketing  
Kuraray Europe GmbH



# ADHESIVES

CLEARFIL™ SE BOND

CLEARFIL™ S<sup>3</sup>BOND

CLEARFIL™ PROTECT BOND

CLEARFIL™ LINER BOND II

## CLEARFIL™ SE BOND and CLEARFIL™ S<sup>3</sup> BOND (CLEARFIL™ Tri-S BOND)

### 135282 Influence of Light-irradiation Intensity on Bonding to One-step vs Two-step

N. SEKI<sup>1</sup>, R. KISHIKAWA<sup>1</sup>, K. HOSAKA<sup>1</sup>, M. NAKAJIMA<sup>1</sup>, and J. TAGAMI<sup>2</sup>, <sup>1</sup> Tokyo Medical & Dental University, Tokyo, Japan, <sup>2</sup> GCOE Program, Tokyo, Japan

**Objectives:** This study tested the hypothesis of differences between two-step and one-step self-etch adhesives in dentin bonding by intensity of light irradiated directly and indirectly through resin composite.

**Methods:** Two commercially available one-step self-etch adhesives (Clearfil Tri-S bond (TS, Kuraray Medical, Tokyo, Japan) and Bond Force (BF, Tokuyama Dental, Tokyo, Japan)), and two-step self-etch adhesive (Clearfil SE bond (SE, Kuraray Medical)) were treated to the flat coronal dentin surfaces of the extracted human third molars. Polymerization of the adhesives was performed by photo-irradiation with a light intensity of either 350 or 600 mW/cm<sup>2</sup> for 10s. After filled with two shades of resin

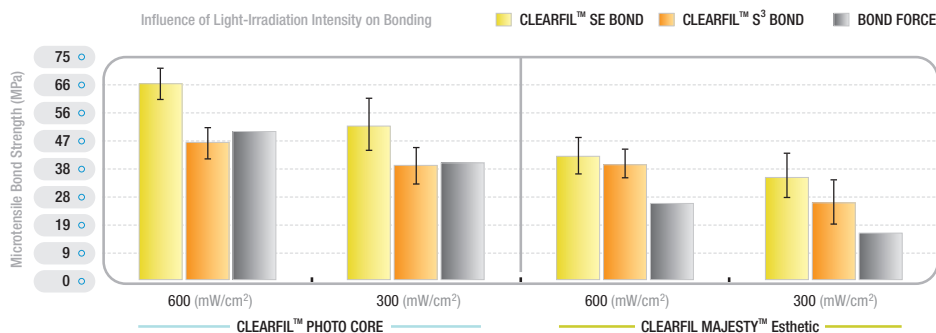
composites; translucent shade (Clearfil Photo Core (PC, Kuraray Medical)) and opaque shade (Clearfil Majesty OA2 shade (MJ, Kuraray Medical)), light-curing was performed with the same light intensities as the bonding procedure for 20 s. The bonded teeth were stored in water at 37°C for 24 h, sectioned perpendicular to the adhesive interface to produce 0.7 x 0.7 mm beam-shaped specimens and subjected to a micro-tensile bond test. The bond strength data were analyzed with two-way ANOVA and Bonferroni test ( $p < 0.05$ ).

**Results:** Micro-tensile bond strength values are in MPa  $\pm$  S.D. (n=18). Groups identified by the different large/small superscript letter within the same row/column are significantly different ( $p < 0.05$ ).

| Results: | PC                              |                                 | MJ                                |                                |
|----------|---------------------------------|---------------------------------|-----------------------------------|--------------------------------|
|          | 600(mW/cm <sup>2</sup> )        | 350(mW/cm <sup>2</sup> )        | 600(mW/cm <sup>2</sup> )          | 350(mW/cm <sup>2</sup> )       |
| SE       | 66.4 $\pm$ 5.5 <sup>A, a</sup>  | 52.5 $\pm$ 9.2 <sup>B, c</sup>  | 42.3 $\pm$ 6.3 <sup>C, e</sup>    | 35.7 $\pm$ 7.7 <sup>C, g</sup> |
| TS       | 46.4 $\pm$ 5.7 <sup>D, b</sup>  | 39.1 $\pm$ 6.8 <sup>E, d</sup>  | 39.7 $\pm$ 5.2 <sup>D, E, e</sup> | 26.6 $\pm$ 7.8 <sup>F, h</sup> |
| BF       | 50.1 $\pm$ 13.0 <sup>G, b</sup> | 39.6 $\pm$ 13.2 <sup>H, d</sup> | 26.1 $\pm$ 7.2 <sup>I, f</sup>    | 16.6 $\pm$ 2.6 <sup>J, i</sup> |

#### ➔ Conclusions:

Each adhesive has its own morphotype, and it is influenced by intensity of light irradiated not only directly but also indirectly through resin composite.



## CLEARFIL™ SE BOND

### 132987 Eight-year clinical evaluation of a two-step self-etch adhesive

*M. PEUMANS, J. DE MUNCK, K. VAN LANDUYT, A. POITEVIN, and B. VAN MEERBEEK,  
Katholieke Universiteit Leuven, Leuven, Belgium*

**Objectives:** The objective of this randomized controlled clinical trial was to evaluate the 8-year clinical performance of a mild two-step self-etch adhesive in non-carious Class-V lesions with and without prior selective phosphoric acid-etching of the enamel cavity margins.

**Methods:** A total of 100 non-carious Class-V lesions in 29 patients were restored with Clearfil AP-X (Kuraray). The composite restorations were bonded following two different approaches: 1. application of Clearfil SE (Kuraray) following a self-etch approach (control group; C-SE non-etch); 2. selective phosphoric acid-etching of the enamel cavity margins before application of Clearfil SE (experimental group; C-SE etch). The restorations were evaluated after 6 months, 1, 2, 3, 5 and 8 years of clinical service regarding retention, marginal integrity and discoloration, caries occurrence, preservation of tooth vitality and post-operative sensitivity.

**Results:** The recall rate at 8 years was 76%. Only two restorations, one of the C-SE non-etch group and one of the C-SE etch group, were clinically unacceptable due to loss of retention leading to a retention rate and a clinical success rate of 97% in both groups. Aging of the restorations was characterized by an increase in the percentage of restorations with a clinically acceptable but small marginal defect (C-SE non-etch: 92%; C-SE etch: 84%) and/or superficial marginal discoloration (C-SE non-etch: 44%; C-SE etch: 28%). At the enamel side, the presence of small marginal defects (C-SE non-etch: 86%; C-SE etch: 65%) and superficial marginal discoloration (C-SE non-etch: 36%; C-SE etch: 11%) was more frequently noticed in the control group than in the experimental group. The difference, however, was only statistically significant for the presence of superficial marginal discoloration (McNemar,  $p=0.01$ ).

#### ➔ Conclusions:

After 8 years of clinical functioning, the clinical effectiveness of CLEARFIL™ SE BOND remained excellent, with selective acid-etching of the enamel cavity margins only having some minor positive effect on marginal integrity and absence of marginal discoloration at enamel.



## CLEARFIL™ SE BOND

### 138074 10-MDP contributes to formation of acid-base resistant zone on enamel

N. LI<sup>1</sup>, T. NIKAIDO<sup>2</sup>, T. TAKAGAKI<sup>2</sup>, P. MAKISHI<sup>3</sup>, J.-H. CHEN<sup>4</sup>, and J. TAGAMI<sup>3</sup>, <sup>1</sup>Fourth Military Medical University, Tokyo Medical & Dental University, Tokyo, Japan, <sup>2</sup>Tokyo Medical & Dental University, Tokyo, Japan, <sup>3</sup>Global COE, International Research Center for Molecular Science in Tooth and Bone Diseases, Tokyo Medical & Dental University, Tokyo, Japan, <sup>4</sup>School of Stomatology, Fourth Military Medical University, Xi'an, China

**Objectives:** To examine the influence of two functional monomers (10-MDP and Phenyl-P) contained in two-step self-etching adhesive systems on the ultrastructure of enamel-adhesive interface after artificial secondary caries formation.

**Methods:** Clearfil SE Bond (Kuraray Medical) and three experimental bonding systems with Phenyl-P were prepared: (1) 10-MDP in both primer and bond (SE-Bond, MM); (2) 10-MDP in primer and Phenyl-P in bond (MP); (3) Phenyl-P in primer and 10-MDP in bond (PM); (4) Phenyl-P in both primer and bond (PP). The buccal ground enamel of human premolars was treated with one of the systems. The bonded interface was exposed to an artificial demineralizing solution (pH4.5) for 4.5h and 5%NaClO with ultrasonic for

30min. Samples were either argon-ion etched (7min) for SEM observation, or ultra-cut for TEM observation.

**Results:** An acid-base resistant zone (ABRZ) was formed on enamel with all the three 10-MDP-containing adhesive systems. However, the ultramorphology and crystallite arrangement of ABRZ were different among groups. PP was the only group devoid of this protective zone.

**Acknowledgements:** This work was supported by the grant from the Chinese Government Graduate Student Overseas Study Program and the Global Center of Excellence (GCOE) Program, International Research Center for Molecular Science in Tooth and Bone Diseases at Tokyo Medical and Dental University.

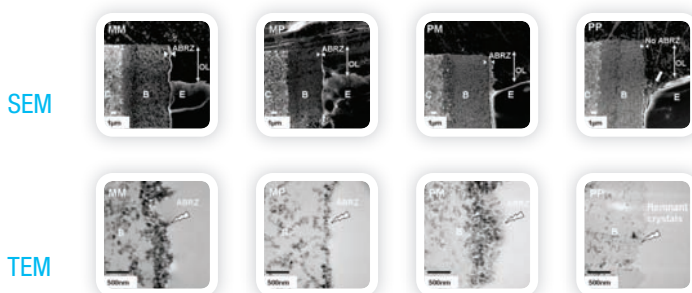


Figure shows SEM (top) and TEM (bottom) micrographs of enamel - adhesive interface after acid-base challenge

#### ➡ Conclusions:

The ABRZ remained at the enamel-adhesive interface after acid-base challenge only with the 10-MDP-containing adhesive systems. The ABRZ may play an important role in bonding performance and prevention of secondary caries in enamel.

## CLEARFIL™ SE BOND and CLEARFIL™ PROTECT BOND

### 136452 The Role of Functional Monomers in Two-step Self-etching Bonding Systems

T. TAKAGAKI<sup>1</sup>, T. NIKAI<sup>1</sup>, H. NURROHMAN<sup>2</sup>, N. LI<sup>3</sup>, and J. TAGAMI<sup>2</sup>, <sup>1</sup>Tokyo Medical & Dental University, Tokyo, Japan, <sup>2</sup>Global COE, International Research Center for Molecular Science in Tooth and Bone Diseases, Tokyo Medical & Dental University, Tokyo, Japan, <sup>3</sup>Fourth Military Medical University; Tokyo Medical & Dental University, Tokyo, Japan

**Objectives:** To investigate the effect of two functional monomers (MDP and phenyl-P) contained in two-step self-etching adhesive systems on bond strength to dentin and secondary-caries inhibition of dentin-adhesive interface in vitro.

**Methods:** Clearfil SE Bond (Kuraray Medical) and three experimental bonding systems were prepared: (1) MDP in both primer and bond (SE-Bond, MM); (2) MDP in primer and Phenyl-P in bond (MP); (3) Phenyl-P in primer and MDP in bond (PM); (4) Phenyl-P in both primer and bond (PP). (Table1). Grounded (#600) dentin of human molars were used for micro-tensile bond test. Sticks were produced with the adhesive area of 1.0mm<sup>2</sup>. The data of  $\mu$ -TBSs (n=20) were analyzed using two-way ANOVA and

t-test with Bonferroni correction ( $\alpha=0.05$ ). For Acid-base resistant zone (ABRZ) observation, the bonded interfaces were exposed to an artificial demineralizing solution (pH4.5) for 90min, 5% NaClO for 20min. Samples were argon-ion etched (7min) before SEM observation.

**Results:** MM and PP showed the highest bond strength among the groups. Although an ABRZ was observed with the adhesive systems containing MDP either in primer or bond, PP group was the only one devoid of this protective zone and showed erosion at interface.

#### ➔ Conclusions:

The functional monomers in two-step self-etching systems not only influence the bonding performance to dentin, but also the formation of ABRZ. Higher bond strength does not always mean the favorable resistance to secondary caries formation.

Table 1 · Means (MPa)  $\pm$  s.d. of micro-tensile bond strength

| Group              | MM (SE bond)                     |      | MP                              |          | PM                              |      | PP                              |          |
|--------------------|----------------------------------|------|---------------------------------|----------|---------------------------------|------|---------------------------------|----------|
|                    | Primer                           | Bond | Primer                          | Bond     | Primer                          | Bond | Primer                          | Bond     |
| Functional Monomer | MDP                              | MDP  | MDP                             | phenyl-P | phenyl-P                        | MDP  | phenyl-P                        | phenyl-P |
| Bond Strength      | 83.50 $\pm$ 11.38 <sup>a,b</sup> |      | 59.31 $\pm$ 8.31 <sup>a,c</sup> |          | 72.09 $\pm$ 8.05 <sup>b,d</sup> |      | 85.07 $\pm$ 8.59 <sup>c,d</sup> |          |

Values with same letters denote statistically significant differences.

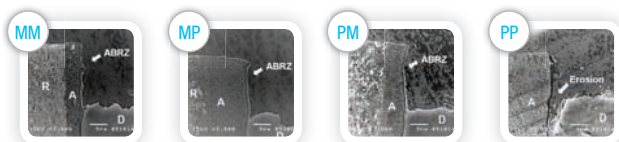


Figure1 SEM images of acid-base resistant zone

## CLEARFIL™ SE BOND and CLEARFIL™ PROTECT BOND

### 135316 SEM observations of the acid-base resistant zone after long-term storage

C. ICHIKAWA<sup>1</sup>, T. NIKAIIDO<sup>1</sup>, G. INOUE<sup>2</sup>, T. NIKAIIDO<sup>1</sup>, and J. TAGAMI<sup>2</sup>, <sup>1</sup>Tokyo Medical & Dental University, Tokyo, Japan, <sup>2</sup>GCOE Program, Tokyo, Japan

**Objectives:** The aim of this study was to evaluate the morphological changes of acid-base resistant zone (ABRZ) at resin-dentin interface after long-term storage in water.

**Methods:** The two-step self-etching adhesive systems, Clearfil SE Bond (SE) and Clearfil Protect Bond (PB) (Kuraray Medical) were used in this study. Each adhesive was applied onto the prepared human dentin disks. A resin composite was put between them and light-cured. After storing in water for 1 day, each specimen was sectioned vertically through the center and embedded in epoxy resin. After storage in water for 1, 3 and 6 months, they were subjected to a demineralizing solution (pH 4.5) for 90 min and then immersed in 5% NaOCl for 20 min. They were sectioned vertically

again and polished. After an argon-ion etching for 7 min, the specimens were observed using a SEM.

**Results:** Outer lesion (OL) at 1 day ranged from 10 to 15  $\mu\text{m}$  for each specimen, however, thickness of the OL increased to approximately 30  $\mu\text{m}$  after 6 months, suggesting the possibility of gradual mineral loss from the dentin surface in water. The ABRZs were observed clearly in all groups. For SE, thickness of the ABRZ was approximately 1  $\mu\text{m}$  after 6 months, which was thinner than that of 1 day (approximately 2  $\mu\text{m}$  thick). For PB, thickness of the ABRZ didn't change after 6 months (2  $\mu\text{m}$ ) at the mid point of the OL. In PB, bottom of the ABRZ was sloped down toward dentin, probably due to fluoride release from the PB adhesive.

#### ➡ Conclusions:

The ABRZs in both SE and PB were clearly observed beneath the hybrid layer after long-term storage in water. The thickness of the ABRZ in SE decreased with storage time, while it was stable in PB, probably due to fluoride release.

## CLEARFIL™ SE BOND and CLEARFIL™ S<sup>3</sup>BOND (CLEARFIL™ Tri-S BOND)

### 133866 Effect of CPN on bond performance of current all-in-one adhesives

S. FUKUDA<sup>1</sup>, K. YASUMOTO<sup>1</sup>, F. NAGANO<sup>2</sup>, S. HOSHIKA<sup>1</sup>, T. TANAKA<sup>1</sup>, T. IKEDA<sup>1</sup>, Y. NAKAOKI<sup>1</sup>, C. WATANABE<sup>1</sup>, D. SELIMOVIC<sup>3</sup>, and H. SANO<sup>1</sup>, <sup>1</sup>Hokkaido University, Sapporo, Japan, <sup>2</sup>Health Science University of Hokkaido, School of Dentistry, Ishikari- Tobetsu, Japan, <sup>3</sup>University of Strasbourg, Strasbourg, France

**Objectives:** The purpose of this study was to examine the effect of Colloidal Platinum Nanoparticles (CPN) on micro-tensile bond strength (MTBS) of current all-in-one adhesives.

**Methods:** Two all-in-one adhesives, Clearfil tri-S bond (S3; Kuraray, Japan) and G-bond plus (GBP; GC, Japan) were used in this study. Clearfil Mega bond (MEGA; Kuraray, Japan) was used as a control. 24 human molars were used in this study. The flat ground surfaces of dentin were polished with 600-grit silicon carbide paper under running water. Application procedures of the adhesives were as follows: control (apply MEGA according to the manufacturer's instructions), dry (apply adhesives according to the manufacturers' instructions), wet (apply adhesives on visibly wet dentin), etch-dry (apply 35% phosphoric acid for 15 seconds, water rinse for 5 seconds and then air dry before the application of

adhesives), etch-wet (apply 35% phosphoric acid for 15 seconds and water rinse for 5 seconds and then apply adhesives on visibly wet dentin), CPN (apply 35% phosphoric acid for 15 seconds and water rinse for 5 seconds, air dry, rewet by 10% CPN for 30 seconds, water rinse for 20 seconds and apply adhesives on visibly wet dentin). Clearfil AP-X (Kuraray, Japan) was built up to approximately 5 mm height on flat surfaces of dentin. They were sectioned into beam specimens (cross-sectional area 1 mm<sup>2</sup>) after storage in 37°C water for 24 hours for MTBS testing and fracture modes were observed by SEM (Hitachi S-4000). One-way ANOVA, Tukey and Games-Howell tests were used for the statistical analysis of the MTBS ( $p < 0.05$ ).

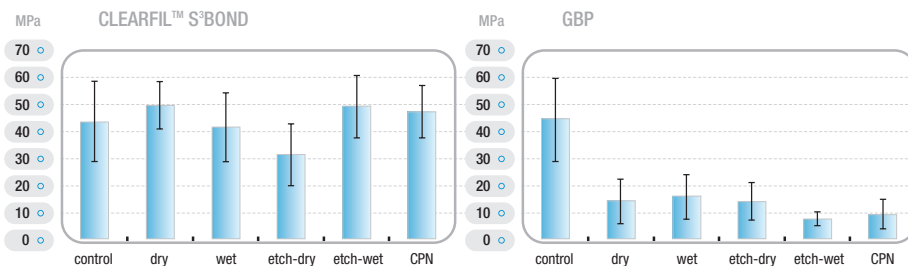
**Results:** The results of the MTBS are listed in Tables and Figures. Identical letters indicate that the values are statistically different ( $p < 0.05$ )

#### ➔ Conclusions:

This study showed that CPN had no effect on MTBS of all-in-one adhesives under the condition of the present study.

|                               | control                     | dry                    | wet                    | etch-dry                   | etch-wet                 | CPN                    |
|-------------------------------|-----------------------------|------------------------|------------------------|----------------------------|--------------------------|------------------------|
| CLEARFIL™ S <sup>3</sup> BOND | 44.4±14.6                   | 50.4±8.66 <sup>a</sup> | 42.3±12.5              | 32.2±11.4 <sup>a,b,c</sup> | 49.9±11.5 <sup>b</sup>   | 47.9±9.45 <sup>c</sup> |
| GBP                           | 44.4±14.6 <sup>a,b,cd</sup> | 14.9±7.91 <sup>a</sup> | 16.6±7.97 <sup>b</sup> | 14.7±6.70 <sup>c</sup>     | 8.59±2.35 <sup>b,c</sup> | 10.5±5.32 <sup>d</sup> |

(n=15)



## CLEARFIL™ SE BOND

### 133917 Bonding performance of contemporary self-etching adhesives

J. FU, K. YASUMOTO, S. HOSHIKA, T. IKEDA, and H. SANO, Hokkaido University, Sapporo, Japan

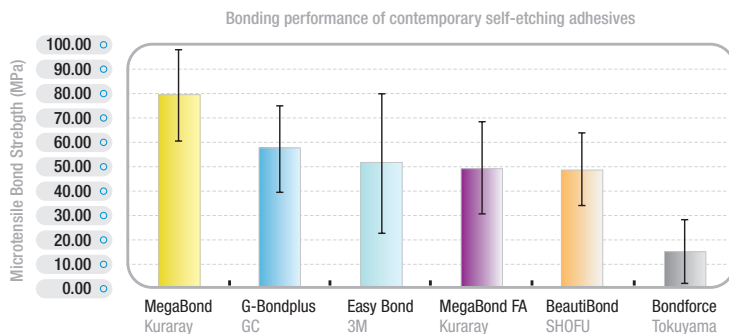
**Objectives:** The objective of the present study was to evaluate the bonding performance of six contemporary self-etching systems (SES). The result of this research would help clinicians to understand the characteristics of bonding performance of current adhesives.

**Methods:** Eighteen human third molars were used in this study and every three teeth were randomly assigned to each system. The adhesives employed were four all-in-one SES, BeautiBond (SHOFU): BB, GBond-plus (GC): GB, BondForce (Tokuyama): BF, Easybond (3M): EB, and two 2-step SES, Megabond (Kuraray): MB and MegabondFA (Kuraray): FA. BB and GB were the HEMA-free adhesives out of these six systems we selected. After removal of crown segment, the 600-grit SiC paper was employed to polish the dentin surface under running water. Then the adhesives

were used under the instruction of each manufacture and followed by the resin composite build-up. After storage in 37° distilled water for 24 hours, the specimen were sectioned into the beam sticks with the cross sectional area 1.0mm<sup>2</sup> for the micro-tensile bond strength test (MTBS) at a crosshead speed of 1mm/min. The obtained data was expressed as MPa and statistically analyzed with one-way ANOVA and Games-Howell test (MB was employed as a control group). **Results:** The mean±SD of MTBS in descending order were: 79.41±19.20 (MB), 57.42±17.77 (GB), 51.26±28.36 (EB), 49.27±19.17 (FA), 49.09±14.95 (BB), and 14.96±13.29 (BF). In statistical analysis, GB, EB, FA, BB and BF showed a significantly lower MTBS (P<0.05) than the control group (MB).

#### ➤ Conclusions:

In this study, MB (two-step SES) could have better bonding performance compared to the all-in-one adhesives and FA. However, the all-in-one SES showed comparable bonding performance compared with the other two-step SES, FA.



## CLEARFIL™ SE BOND and CLEARFIL™ LINER BOND II

### 134260 Ten-Year Degradation of Resin-dentin Bonds in vitro

M. HASHIMOTO, S. FUJITA, F. NAGANO, H. OHNO, and K. ENDO, Health Science University of Hokkaido, School of Dentistry, Ishikari- Tobetsu, Japan

**Objectives:** The purpose of this study was to evaluate the durability of resin-dentin bonds in water storage testing for 10 years.

**Methods:** Resin-dentin bonded bulk specimens were prepared using six resin adhesives. The bulk specimens were stored in water for 24 h (control) and for 10 years (experimental group). After each storage period, specimens were sectioned to produce beams (adhesive area 0.9 mm<sup>2</sup>). Then the specimens were subjected to a microtensile bond test. The bond strength values were statistically evaluated with two-way ANOVA and Fisher's PLSD test (p < 0.05, n = 14 to 18 for each group). After the bond test, fractured surfaces were examined using SEM.

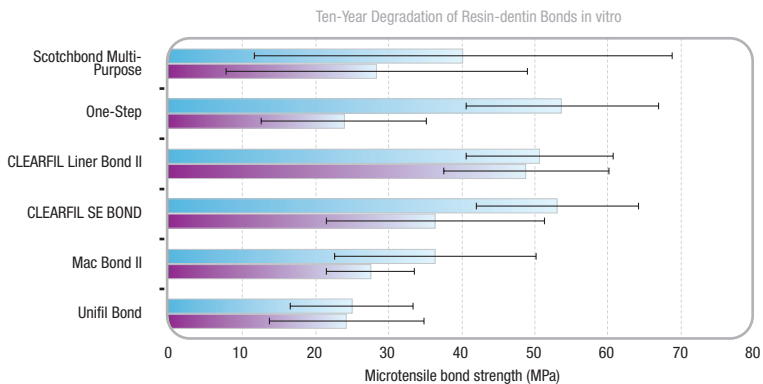
In addition, interfacial observation of silver tracer was performed with SEM.

**Results:** The bond strengths of four adhesives were significantly decreased after 10 years (p < 0.05). However, there was no significant difference in bond strengths between control and 10-year specimens of Clearfil Liner Bond II and Unifil Bond (p > 0.05). The same superscript letters in the table indicate that there were no significant differences (p > 0.05). The extent of nanoleakage was increased within the hybrid layer for all adhesives after 10 years. The water tree propagation was observed in the bonding resin layer after aging.

|          | Scotchbond Multi-Purpose (3M) | OneStep (Bisco) | Clearfil Liner Bond II (Kuraray) | Clearfil SE Bond (Kuraray) | Mac Bond II (Tokuyama) | Unifil Bond (GC)         |
|----------|-------------------------------|-----------------|----------------------------------|----------------------------|------------------------|--------------------------|
| 24 hours | 40.3 ± 15.1                   | 53.8 ± 13.1     | 50.7 ± 10.0 <sup>a</sup>         | 53.2 ± 11.0                | 36.5 ± 13.7            | 25.1 ± 8.4 <sup>b</sup>  |
| 10 years | 28.5 ± 20.5                   | 24.0 ± 11.3     | 48.9 ± 11.2 <sup>a</sup>         | 36.5 ± 14.9                | 27.6 ± 5.9             | 24.4 ± 10.6 <sup>b</sup> |

#### ➡ Conclusions:

Water tree propagation might be a degradation symptom in the bonding resin layer of resin-dentin bonds



## CLEARFIL™ SE BOND and CLEARFIL™ S<sup>3</sup> BOND (CLEARFIL™ Tri-S BOND)

### 136341 Effect of C-factor on Bond Strength to Cavity Floor/Wall

T. YOSHIKAWA, Tokyo Medical & Dental University, Tokyo, Japan, N. WATTANAWONGPITAK, Naresuan University, Phitsanulok, Thailand, and J. TAGAMI, Tokyo Medical & Dental University, GCOE Program, IRCMSTBD, Tokyo, Japan

**Objectives:** The purpose of this study was to evaluate the effect of C-factor and part of dentin on resin composite bond strength to Class I cavity floors and walls using various adhesive systems.

**Methods:** Box-form occlusal cavities, 3x5x2mm (C-factor=3.1), were prepared on flat dentin surfaces of human molars. To assess the relationship between the C-factor and part of dentin, the walls of cavities were removed making a flat surface (deep flat and wall flat) for bonding (C-factor=0.3). Each specimen was restored with one of three adhesives: Clearfil SE Bond (SE, Kuraray Medical), Single Bond (SB, 3M ESPE) or Clearfil Tri-S Bond (TS, Kuraray Medical); followed by bulk filling using Z100 resin composite (3M ESPE). After 600mW/cm<sup>2</sup> for 40s light-curing, the teeth were sectioned mesio-distally obtaining two slabs. One slab was cut perpendicular to cavity floor

and the other was cut parallel to cavity floor obtaining 0.7x0.7 mm beams. Control specimens were also cut obtaining beams. The Micro-tensile bond strength ( $\mu$ TBS, MPa) to flat dentin specimen, cavity floor and cavity wall specimen was determined. Data (n=8) were analyzed using Bonferroni test. **Results:** SB and TS showed significantly lower  $\mu$ TBS to cavity floor and cavity wall compared with that of each deep flat and wall flat group ( $p<0.05$ ). However, there was no significantly different  $\mu$ TBS between deep flat and cavity floor group using SE ( $p>0.05$ ). SB showed significantly lower  $\mu$ TBS to cavity floor compared with that of cavity wall group ( $p<0.05$ ). However, there was no significantly different  $\mu$ TBS between cavity floor and cavity wall group using SE and TS ( $p>0.05$ ).

#### ➔ Conclusions:

Bonding to cavity floor and cavity wall was dominated by C-factor except for CLEARFIL™ SE Bond. Supported by Grant #16591907 from JSPS and GCOE Program, FRMDRTB.

| Adhesive system | Deep flat                   | Wall flat                     | Cavity floor                | Cavity wall                 |
|-----------------|-----------------------------|-------------------------------|-----------------------------|-----------------------------|
| SE              | 54.8 (8.3) <sup>a,A</sup>   | 88.2 (3.9) <sup>a,A,B</sup>   | 48.2 (4.3) <sup>a,B</sup>   | 48.0 (6.6) <sup>a,A</sup>   |
| SB              | 55.0 (9.8) <sup>b,A</sup>   | 74.4 (7.9) <sup>a,A,B</sup>   | 30.9 (6.5) <sup>a,A,B</sup> | 43.6 (7.2) <sup>b,B</sup>   |
| TS              | 35.7 (4.5) <sup>a,b,A</sup> | 52.6 (5.0) <sup>a,A,B,C</sup> | 19.1 (3.1) <sup>a,A,B</sup> | 28.4 (4.1) <sup>a,b,C</sup> |

Intergroup data designated with same superscript small letters each adhesive are significantly different ( $p<0.05$ ). Intergroup data designated with same superscript capital letters each dentin part are significantly different ( $p<0.05$ ).

## CLEARFIL™ SE BOND and CLEARFIL™ S<sup>3</sup> BOND

### 136112 The effect of water-sorption on the mechanical-property of self-etch adhesives

K. HOSAKA<sup>1</sup>, M. NAKAJIMA<sup>1</sup>, M. TAKAHASHI<sup>1</sup>, W. SAKANO<sup>1</sup>, R. KISHIKAWA<sup>1</sup>, D. PASHLEY<sup>2</sup>, and J. TAGAMI<sup>3</sup>, <sup>1</sup>Tokyo Medical & Dental University, Tokyo, Japan, <sup>2</sup>Medical College of Georgia, Augusta, GA, <sup>3</sup>Cariology and Operative Department, GCOE Program at TMDU, Tokyo Medical & Dental University, Tokyo, Japan

**Objectives:** To evaluate the effect of water-sorption on the mechanical-property of self-etch adhesives. **Methods:** Two one-step self-etch adhesives, Clearfil™ S<sup>3</sup>Bond (S<sup>3</sup>; Kuraray Medical Inc.), G Bond Plus (GP; GC Corp.) and a two-self-etch adhesive, Clearfil™ SE Bond (adhesive, SE; Kuraray) were used. Solvents in the one-step adhesives were evaporated using a dental 3-way syringe for 3min, followed by polymerization of all adhesives for 3min. Water sorption (WS) and solubility (SL) values of adhesive polymers were measured using a modification of the ISO 4049 standard. The moduli of elasticity (E) of adhesive polymers were also measured by three-point flexural bending test in initial (24h after

polymerization), wet (when specimens absorbed maximum water) and dry (when specimens were completely dried) states. The data for water sorption and solubility were analyzed using one-way ANOVA and Bonferoni test, respectively (p=0.05). The data of E were statistically analyzed using a two-way ANOVA (materials and experimental conditions) and Bonferoni test (p=0.05).

**Results:** WS and SL varied among adhesives. E of all adhesives fell significantly when water fully diffused into the specimens. E (initial) of S<sup>3</sup> and GP were significantly lower than E (dry), while E (initial) of SE was similar to E (dry). The highest E (wet) was seen in SE.

#### ➔ Conclusions:

WS and SL of one-step self-etch adhesives were higher than in the solvent-free SE adhesive. The results suggested that WS decreased the mechanical property of adhesive polymers, and that residual solvents of one-step adhesives after evaporation may lower their initial mechanical property. Supported by GCOE program at TMDU and #20791382 from MEXT of Japan.

| Materials      | E (initial)              | E (wet)                | E (dry)                  |
|----------------|--------------------------|------------------------|--------------------------|
| S <sup>3</sup> | 3.39±0.38 <sup>A</sup>   | 0.85±0.09 <sup>B</sup> | 4.35± 0.85               |
| GP             | 2.27±0.81                | 1.17±0.13 <sup>B</sup> | 3.08±0.62 <sup>C</sup>   |
| SE             | 3.30±0.35 <sup>A,a</sup> | 2.76±0.36              | 3.70±0.38 <sup>C,a</sup> |

Mean±S.D. (GPa)(n=10) Same superscript letters mean no significant difference (p>0.05).

| Materials      | Water sorption         | Materials      | solubility             |
|----------------|------------------------|----------------|------------------------|
| S <sup>3</sup> | 188.8±6.6 <sup>A</sup> | S <sup>3</sup> | 8.3±13.3 <sup>A</sup>  |
| GP             | 162.2±9.3 <sup>B</sup> | GP             | 31.0±10.0 <sup>B</sup> |
| SE             | 84.4±3.3 <sup>C</sup>  | SE             | 0.0±1.7 <sup>C</sup>   |

Mean±S.D. (mg/mm<sup>3</sup>)(n=10) Different superscript letters mean significant difference (p<0.05).



## CLEARFIL™ SE BOND and CLEARFIL™ S<sup>3</sup> BOND

### 134834 Sealing Abilities of All-in-one Adhesives under Thermo-mechanical Cyclic Stress

T. KIMISHIMA<sup>1</sup>, M. HARA<sup>1</sup>, T. MASEKI<sup>1</sup>, Y. NARA<sup>1</sup>, and I.L. DOGON<sup>2</sup>, <sup>1</sup>Nippon Dental University, Tokyo, Japan, <sup>2</sup>Harvard University, Boston, MA

**Objectives:** The purpose of this study was to examine the sealing abilities of all-in-one adhesives (Bond Force (Tokuyama): BF, Adper Easy Bond (3M ESPE); EB, Clearfil Tri-S Bond (Kuraray): TS, Clearfil SE Bond (Kuraray): SE.) under thermo-mechanical cyclic stress. **Methods:** 80 standardized wedge shaped cavities with occlusal margin on enamel and gingival margin on dentine were prepared in extracted human lower premolars. The adhesives were applied to the cavities according to manufacturer's instructions and resin composites (Estelite P Quick (Tokuyama) for BF, Supreme XT (3M ESPE) for EB, Clearfil AP-X (Kuraray) for TS and SE) were placed and light-cured. The specimens were stored in a moisture box at 37°C for 24hrs, then polished with a series of polishing disc, and randomly divided into two groups, with stress; S+ and without stress; S-. For S+ group, the

specimens were thermocycled (4 and 60°C, 1,250 cycles) and cyclic loaded (12kgf x 10<sup>5</sup>) simultaneously. For S-group specimens, no stress was applied. Dyeing for an hour, both S+ and S-specimens were sectioned vertically and microleakage were evaluated by a graded criterion, and analyzed using Kruskal-Wallis and Wilcoxon tests (n=10). **Results:** At the occlusal wall, microleakage of S+ were significantly greater than S- in BF, EB, TS (p<0.01), while there was no difference in SE. However, at the gingival wall, there were no significant differences between S+ and S- in all adhesives. There were significant differences in microleakage between occlusal and gingival walls in BF, EB and TS of S+ group. There was no significant difference in microleakage at occlusal and gingival wall in the adhesives of S-group.

#### ➡ Conclusions:

Subjected to sever combination stress, gingival margin showed excellent sealing ability compare to occlusal margin. Sealing abilities of all-in-one adhesives were equivalent to the two-step self-etch adhesive SE which is well-known for its high-performance.

## CLEARFIL™ S<sup>3</sup> BOND

### 139403 2-Year Clinical Effectiveness of All-in-One Adhesives in Non-carious Cervical Lesions

*K. HOSAKA<sup>1</sup>, M. NAKAJIMA<sup>1</sup>, M. TAKAHASHI<sup>1</sup>, W. SAKANO<sup>1</sup>, R. KISHIKAWA<sup>1</sup>, D. PASHLEY<sup>2</sup>, and J. TAGAMI<sup>3</sup>, <sup>1</sup>Tokyo Medical & Dental University, Tokyo, Japan, <sup>2</sup>Medical College of Georgia, Augusta, GA, <sup>3</sup>Cariology and Operative Department, GCOE Program at TMDU, Tokyo Medical & Dental University, Tokyo, Japan*

**Objectives:** To evaluate the clinical performance of two one-step self-etch adhesives in a randomized controlled clinical trial. **Methods:** Twenty-nine patients had 172 non-carious cervical lesions restored with composite (Gradia Direct Anterior; GC) using either the HEMA-rich adhesive Clearfil Tri-S Bond (Kuraray) or the HEMA-free adhesive G-Bond (GC). The restorations were evaluated by two calibrated examiners at baseline and after 6, 12 and 24 months of clinical service regarding retention, caries recurrence, marginal integrity and discoloration, preservation of tooth vitality and post-operative sensitivity. Retention loss, caries recurrence, or any severe marginal defects/discoloration requiring clinical intervention (repair or replacement) were considered as clinical failures. The data were statistically analysed using the Mann-Whitney U and Friedman ANOVA tests ( $p < 0.05$ ). **Results:** The recall rate at 6 and

12 months was 100%, and decreased to 93.1% at 24 months. At the last recall, the retention rate was 98.6% for Clearfil Tri-S Bond and 98.7% for G-Bond. No significant differences were observed between the two adhesives for all the parameters evaluated, irrespective of the recall ( $p > 0.05$ ). After 24 months of clinical service, both adhesives presented an increase in the percentage of small, though still clinically acceptable, marginal defects (13.7% for Clearfil Tri-S Bond, and 14.6% for G-Bond) and marginal discoloration (24.7% for Clearfil Tri-S Bond, and 18.7% for G-Bond). For both adhesives, tooth sensitivity was significantly decreased at 6 months as compared to the pre-operative condition. This relative reduction in sensitivity remained stable up to the 24-month recall. **At 24 months, the overall clinical success rate was 95.9% and 96% for Clearfil Tri-S Bond and G-Bond, respectively.**

#### ➔ Conclusions:

Both one-step self-etch adhesives presented an equally favorable clinical effectiveness at 24 months.

## CLEARFIL™ PROTECT BOND

### 140229 In vivo microbiological evaluation of adhesive systems after stepwise excavation

*E. RODRIGUES<sup>1</sup>, DUQUE<sup>2</sup>, and R.M. PUPPIN-RONTANI<sup>1</sup>, <sup>1</sup>University of Campinas, Piracicaba, Brazil, <sup>2</sup>University of Campinas, Piracicaba; Federal Fluminense University, Nova Friburgo, Brazil*

**Objectives:** The purpose of this study was to assess the antimicrobial effect of two self-etching adhesives after stepwise excavation in vivo. **Methods:** After evaluation of specific clinical criteria, 44 children presented caries lesions and 47 primary molars were selected. Treatment consisted of incomplete excavation with burs in low speed, followed by the first collect of carious dentine in RTF (reduced transport fluid) solution, application of one of the self-etching adhesives: Clearfil SE Bond (Kuraray, Japan) – SE group (n=21) or Clearfil Protect Bond (Kuraray, Japan) containing MDPB antimicrobial agent – PB group (n=26) and sealing for 3 months. After this period, the temporary sealing was removed and second collect of remaining carious dentin was performed, following

for the definitive sealing of the cavity. Microbiological samples (first and second collects) were serially diluted and cultivated in specific media for mutans streptococci – MS (Mitis salivarius agar), lactobacilli – LB (Rogosa agar) and total microorganisms – TM (Brain heart infusion agar supplemented with sheep blood) at 37°C for 48h in anaerobic jars. Statistical analyses were conducted using Wilcoxon and Mann-Whitney tests. **Results:** MS, LB and TM counts were compared in the first collect of carious dentin and there was no significant difference ( $p>0.05$ ) among groups, showing similar initial counts. Regardless adhesive system, there was decrease on LB and TM counts between first and second collects. However, PB showed antibacterial effect for MS.

#### ➡ Conclusions:

The antibacterial effect of the adhesive system is dependent on microorganism. Supported by Fapesp 07/08544-9 and 08/05228-1.

# RESIN CEMENT

CLEARFIL™ SA CEMENT  
CLEARFIL™ ESTHETIC CEMENT  
PANAVIA™ F 2.0  
PANAVIA™ 21

## CLEARFIL™ SA CEMENT

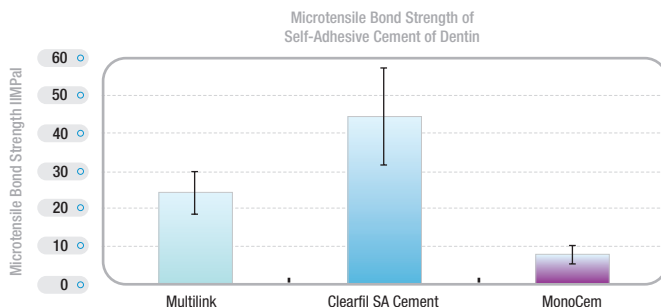
### 138885 Microtensile Bond Strength of self-adhesive resin cements to dentin

A. G. ALMEIDA<sup>1</sup>, E. DAUDT<sup>1</sup>, G. LOPES<sup>2</sup>, and L.N. BARATIERI<sup>2</sup>, <sup>1</sup>Universidade Federal De Santa Catarina, Florianópolis - SC, Brazil, <sup>2</sup>Universidade Federal De Santa Catarina, Florianópolis, Brazil

**Objectives:** This study evaluated the microtensile bond strength ( $\mu$ TBS) of self-adhesive resin cements to dentin. **Methods:** The occlusal enamel of 6 third molars were cut and occlusal dentin were polished with #600 SIC paper to obtain a flat dentin surface. Indirect resin composite (Sinfony/3M ESPE) discs were built using a metallic matrix with 2mm depth and a diameter of 8mm. After the complete polymerization the discs were treated with 50 $\mu$  aluminum oxide air abrasion for 10 seconds. The teeth were randomly assigned to 3 groups according to the resin cement used: Group MU: self-curing resin cement Multilink (Ivoclar Vivadent) with separate self-etching primer step (control group); Group CL: self-adhesive resin cement Clearfil SA Cement (Kuraray); and Group MO: self-adhesive resin cement MonoCem

(Shofu). Each resin cement was applied according to the manufacturer's instruction. After 24h in water, specimens were cut in two perpendicular directions to obtain beams with a low-speed diamond saw (n=30). The specimens were stored in water (37°C, 24h) and then tested ( $\mu$ TBS) with an universal testing machine at a crosshead speed of 0.5mm/min. The results were recorded in Mega Pascals (MPa). Data was analyzed statistically by one-way ANOVA and Duncan's post-hoc ( $p < 0.05$ ).

**Results:** Mean  $\mu$ TBS (MPa  $\pm$  SD) respectively were: MU (24.2  $\pm$  5.7)b; CL (44.5  $\pm$  13.0)a; MO (7.7  $\pm$  2.4)c. There were significant statistical differences between the resin cements. **Conclusion:** The  $\mu$ TBS bond strength of self-adhesive resin cements to dentin is dependent on its specific composition.



#### ➔ Conclusions:

Each adhesive has its own morphotype, and it is influenced by intensity of light irradiated not only directly but also indirectly through resin composite.

## CLEARFIL™ SA CEMENT

### 135908 Long-Term Bond Strength and Fluoride Release Capabilities of Three Cements

L.M. PINZON<sup>1</sup>, L.G. WATANABE<sup>1</sup>, V. USKOKOVIC<sup>1</sup>, S. HABELITZ<sup>1</sup>, J.M. POWERS<sup>2</sup>, S.J. MARSHALL<sup>1</sup>, and G.W. MARSHALL<sup>1</sup>, <sup>1</sup>University of California - San Francisco, San Francisco, CA, <sup>2</sup>University of Texas - Houston/Health Science Center, Houston, TX

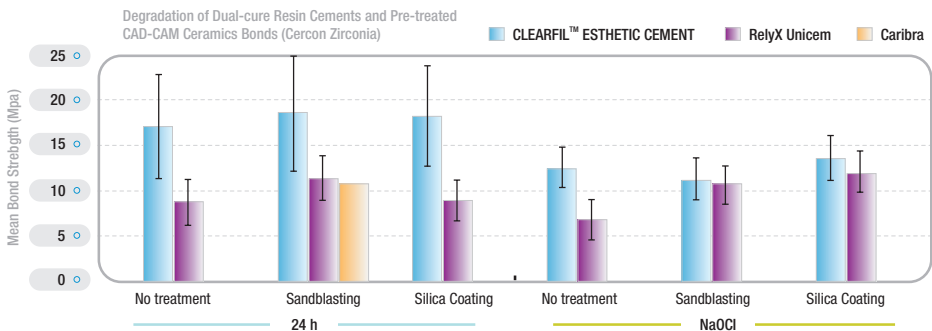
**Purpose:** To evaluate the effects of long-term storage on dentin-cement bond strength and fluoride release of Clearfil SA Cement [(CS)/Kuraray America], FujiCEM [(GC)/GC America], and Maxcem Elite [(ME)/Kerr Corp.] **Methods:** Human third molars were sectioned and polished with 320-grit SiC discs. Composite stubs (5mm height/19.6mm<sup>2</sup> area) (Clearfil AP-X/Kuraray America) were air abraded with Al<sub>2</sub>O<sub>3</sub> particles, ultrasonically cleaned, and bonded to the dentin specimens with the cements. Specimens were stored in water at 37°C for 6 months and were debonded under shear load with an Instron/1122 at a cross-head speed of 5mm/min. Failure modes were determined using 100X magnification. Fluoride-release specimens were disc-shaped, 13.5mm in

diameter and 1mm thick and were polished to 0.25µm. Specimens were conditioned in air for 6 months at 37°C, and then stored in 10ml of a phosphate buffered solution at pH 6.8 in individual containers at 37°C. Fluoride release was measured using direct potentiometry after 7, 15 and 30 days of storage. Data were analyzed using ANOVA with Fisher's PLSD intervals (p<0.05) **Results:** Means and (s.d) of shear bond strengths (MPa/n=8) are listed. Fisher's PLSD interval (p<0.001) among cements was 0.6 MPa. Failures sites were mixed. Values of fluoride release (ppm/n=8) after 7, 15 and 30 days are listed. Fisher's PLSD intervals (p<0.001) between times and among cements were 0.1 and 0.1 ppm.

| Property                                    | ME        | GC        | CS        |
|---|-----------|-----------|-----------|
| Bond strength to dentin (MPa)               | 1.4 (0.4) | 1.4 (0.5) | 3.8 (0.7) |
| Fluoride release from day 2 to day 7 (ppm)  | 1.9 (0.1) | 3.2 (0.0) | 1.3 (0.2) |
| Fluoride release from day 2 to day 15 (ppm) | 2.1 (0.1) | 3.2 (0.1) | 1.5 (0.1) |
| Fluoride release from day 2 to day 30 (ppm) | 2.7 (0.1) | 3.4 (0.1) | 2.0 (0.1) |

#### ➔ Conclusions:

CS had significantly higher bond strength than GC and ME. GC and ME had the highest fluoride release after 7, 15 and 30 days. **Acknowledgment:** Kuraray America.



## CLEARFIL™ ESTHETIC CEMENT

### 135464 Degradation of Dual-cure Resin Cements and Pre-treated CAD-CAM Ceramics Bonds

R.C. OYAGÜE<sup>1</sup>, M. TOLEDANO<sup>2</sup>, F. MONTICELLI<sup>3</sup>, A. ROMEO<sup>4</sup>, E. OSORIO<sup>2</sup>, and R. OSORIO<sup>2</sup>, <sup>1</sup>Complutense University of Madrid, UCM, Madrid, Spain, <sup>2</sup>University of Granada, Granada, Spain, <sup>3</sup>University of Zaragoza, Huesca, Spain, <sup>4</sup>University of Barcelona, Barcelona, Spain

**Objectives:** To evaluate the effect of an in vitro challenge on the bond stability of dual-cure resin cements to zirconium- and aluminum-oxide ceramics.

**Methods:** Surfaces of 18 zirconia blocks (Cercon Zirconia) and 18 glass-infiltrated alumina blanks (Cerec) were conditioned as follows: 1. No treatment; 2. Sandblasting -125µm alumina particles; 3. Silica coating -50µm alumina particles modified by silica. Composite blocks (Tetric Evo Ceram) were bonded to pre-treated ceramic surfaces with three different dual-cure resin cements: 1. CLEARFIL™ ESTHETIC CEMENT (Kuraray); 2. RelyX Unicem (3M ESPE); Cali-

bra (Dentsply Caulk). After 24h, bonded specimens were cut into sticks (bonded area 1mm<sup>2</sup>). Half of the beams were immediately loaded in tension (cross-head speed: 0.5mm/min) until failure. The remaining half of the sticks were tested after 5h of immersion in 10% NaOCl-aqueous solution. Data were analyzed by ANOVA and Tukey's test for multiple comparisons ( $p < 0.05$ ). **Results:** Mean bond strength (MPa) and standard deviation are presented in the table. Letters in rows and numbers in columns indicate differences.

|                | CLEARFIL™ ESTHETIC CEMENT     |                               |                             |                               | RelyX Unicem                  |                               |                               |                                 | Calibra |                                |       |      |
|----------------|-------------------------------|-------------------------------|-----------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|---------------------------------|---------|--------------------------------|-------|------|
|                | 24h                           |                               | NaOCl                       |                               | 24h                           |                               | NaOCl                         |                                 | 24h     |                                | NaOCl |      |
|                | Alum                          | Zirc                          | Alum                        | Zirc                          | Alum                          | Zirc                          | Alum                          | Zirc                            | Alum    | Zirc                           | Alum  | Zirc |
| No treatment   | 13.27<br>(2.43)<br><i>Iac</i> | 17.04<br>(5.70)<br><i>Iab</i> | 6.47<br>(2.06)<br><i>Ie</i> | 12.60<br>(2.24)<br><i>Icd</i> | 11.84<br>(1.32)<br><i>Icd</i> | 8.73<br>(2.50)<br><i>Ide</i>  | 9.36<br>(1.98)<br><i>Icde</i> | 6.77<br>(2.25)<br><i>Ie</i>     | 0.00    | 0.00                           | 0.00  | 0.00 |
| Sandblasting   | 13.42<br>(1.50)<br><i>Iac</i> | 18.63<br>(6.44)<br><i>Ib</i>  | 7.13<br>(1.75)<br><i>Ie</i> | 11.34<br>(2.31)<br><i>Icd</i> | 12.57<br>(1.14)<br><i>Icd</i> | 11.44<br>(2.46)<br><i>Icd</i> | 8.06<br>(2.93)<br><i>Ide</i>  | 10.66<br>(2.13)<br><i>I2cde</i> | 0.00    | 10.84<br>(2.40)<br><i>Icde</i> | 0.00  | 0.00 |
| Silica Coating | 11.60<br>(2.09)<br><i>Iad</i> | 18.19<br>(5.51)<br><i>Ib</i>  | 7.08<br>(2.52)<br><i>Ie</i> | 13.64<br>(2.50)<br><i>Iac</i> | 11.65<br>(1.64)<br><i>Icd</i> | 8.88<br>(2.21)<br><i>Ide</i>  | 6.60<br>(2.52)<br><i>Ie</i>   | 12.09<br>(2.32)<br><i>I2cd</i>  | 0.00    | 0.00                           | 0.00  | 0.00 |

#### ➔ Conclusions:

Resin-cement/ceramic interfaces are prone to degradation. Bonds durability depends on cement selection rather than on surface treatments or ceramic type. **Acknowledgements:** Grants CICYT/FEDER MAT2008-02347/MAT, JA P08-CTS-3944 and P07-CTS-2568.

## PANAVIA™ F 2.0

### 131832 Dual-Curing Cements: Hardness and Elastic Modulus after Different Polymerization Procedures

S. FLURY, A. PEUTZFELDT, and A. LUSSI, University of Bern, Bern, Switzerland

**Objectives:** The aim of this study was to evaluate the Vickers hardness and the elastic modulus of three dual-curing resin cements polymerized according to different curing procedures.

**Methods:** Metal molds were filled with Panavia™ F 2.0 (Kuraray, Japan), SpeedCEM (Ivoclar, Liechtenstein) or SmartCem2 (Dentsply, USA) at 30°C and the resin cements were cured according to one of five curing procedures (n=30 per cement and procedure): 1) 5min light-curing (positive control), 2) 40s light-curing (clinical light-curing), 3) 6min auto-curing (negative control), 4) 6min auto-curing followed by 40s light-curing (efficacy of light-curing after auto-curing), 5) 5s light-curing, 1min auto-curing and 40s light-curing (recommended procedure for removal of excess cement). Light-curing was performed through an IPS Empress CAD ceramic disc (shade: A3, thickness: 2.5mm; Ivoclar) except for the positive control where light-curing was performed through a mylar strip. Vickers hardness (HVN) and elastic modulus

(GPa) were measured simultaneously using a Fisherscope HM2000 hardness indentation device (Fischer, Germany) with an indentation-load of 10mN for 15s. Procedures were compared with pairwise Wilcoxon tests with Bonferroni-Holm adjustment for multiple testing. The level of significance was set at  $\alpha=0.05$ .

**Results:** For all cements, procedure 1 gave the highest Vickers hardness (HVN: Panavia 73.6±13.8; SpeedCEM 46.8±4.5; SmartCem2 62.4±6.0) and elastic modulus (GPa: Panavia 11.7±1.3; SpeedCEM 8.2±0.6; SmartCem2 9.1±0.7). Procedure 2 lowered Vickers hardness and elastic modulus significantly. Procedure 3 showed the lowest Vickers hardness (HVN: Panavia 11.3±6.7; SpeedCEM 10.6±5.9; SmartCem2 10.8±2.9) and elastic modulus (GPa: Panavia 3.0±2.1; SpeedCEM 3.4±1.6; SmartCem2 2.7±0.7) whereas procedures 4 and 5 gave significantly higher Vickers hardness and elastic modulus values compared to procedure 3.

#### ➔ Conclusions:

Auto-curing alone resulted in inferior Vickers hardness and elastic modulus compared to immediate light-curing. However, light-curing the resin cements after the auto-curing restored the Vickers hardness and elastic modulus.



## PANAVIA™ 21

### 132738 Evaluation of Procera Alumina Bridge in anterior regions: preliminary report

*M. FRADEANI, Studio di Odontoiatria Specialistica, Pesaro, Italy, and G. FABBRI, Studio Odontoiatrico Specialistico, Cattolica, Italy*

**Purpose:** The aim of this prospective clinical study was to investigate the clinical performance of Procera Alumina three (3-UB) and four (4-UB) unit bridges, placed in the frontal-lateral areas of the mouth on both natural and implant abutments. **Methods:** Between 2005 and 2010, 12 Procera Alumina bridges were placed in 12 adult patients with good periodontal conditions. 7 supported by natural abutment and 5 by Zirconia abutment. Two restorations (a, 3-UB and a, 4-UB) were placed in the mandible and ten restorations (three, 4-UBs and seven, 3-UBs) were placed in the maxilla. The opposing dentition consisted of natural teeth or ceramic material. Panavia 21 TC (Kuraray) Unicem(3M/ESPE) and Multilink Automix (Ivoclar, Vivadent) were used for the cementation. The internal surfaces were air-abraded with 50 µm aluminum oxide particles or silica coated

with the Rocatec system (3M/ESPE), and silanated (Ceramic Primer, 3M/ESPE) prior to cementation. All patients were evaluated 3, 6 and 12 months after the cementation. Additionally, patients were recalled in 2010 for a clinical assessment: color match, porcelain surface, marginal discoloration, and marginal integrity were clinically examined following modified CDA/Ryge criteria. The survival rate was calculated using Kaplan-Meier life table analysis, taking into account any complications that required a remake of the bridge. **Results:** The survival rate was 100% at 1 year. The longest period of observation was 4 years and no complications were observed. During the observation period chipping occurred on 2 bridges after 6 months. These restorations were smoothed, finished and not considered failed.

#### ➔ Conclusions:

Although limited by the number of patients treated and the short follow up these 1- year results encourage the use of Procera AllCeram bridges as a reliable solution for treatment of anterior areas.

## PANAVIA™ 21

### 134500 Retentive strength of zirconium-oxide crowns to self adhering cements

C.-P. ERNST, G. KAMPF, I. BUSEMANN, E. STENDER, and B. WILLERSHAUSEN, Johannes Gutenberg University, Mainz, Germany

**Objectives:** The retentive strength of one experimental self adhering cement (SFCem44/3M ESPE), six self adhering cements (RelyX Unicem Aplicap, RelyX Unicem Clicker/3M ESPE, iCEM/Heraeus, Maxcem Elite/sds Kerr, Bifix SE/VOCO, SpeedCEM/Ivoclar), two self adhesive cements with self-etch primers (Panavia 21/Kuraray, Secure/Sun Medical) one glass ionomer-cement (Ketac Cem Aplicap/3M ESPE), one resin modified glass ionomer-cement (Meron Plus/VOCO), and a zinc-phosphate cement (Harvard) were examined for luting zircon-oxide ceramic crowns (LAVA, 3M ESPE) on extracted human teeth after thermocycling. **Methods:** Metal molds were filled with Panavia F2.0 (Kuraray, Japan), SpeedCEM (Ivoclar, Liechtenstein) or SmartCem2 (Dentsply, USA) at 30°C and the resin cements were cured according to one of five curing procedures (n=30 per cement and procedure): 1) 5min light-curing (positive control),

2) 40s light-curing (clinical light-curing), 3) 6min auto-curing (negative control), 4) 6min auto-curing followed by 40s light-curing (efficacy of light-curing after auto-curing), 5) 5s light-curing, 1min auto-curing and 40s light-curing (recommended procedure for removal of excess cement). Light-curing was performed through an IPS Empress CAD ceramic disc (shade: A3, thickness: 2.5mm; Ivoclar) except for the positive control where light-curing was performed through a mylar strip. Vickers hardness (HVN) and elastic modulus (GPa) were measured simultaneously using a Fisherscope HM2000 hardness indentation device (Fischer, Germany) with an indentation-load of 10mN for 15s. Procedures were compared with pairwise Wilcoxon tests with Bonferroni-Holm adjustment for multiple testing. The level of significance was set at  $\alpha=0.05$ .

## Results

The retentive strength values [N/mm<sup>2</sup>] were (Min/Q1/Median/Q3/Max):

### ➔ Conclusions:

The performance of the different self adhering cements varied significantly within this group. **A significantly higher retentive strength can be obtained compared to glass ionomer or zinc phosphate cements.** This study was supported by 3M ESPE, Heraeus, Ivoclar Vivadent, VOCO, and Sun Medical.

**SFCem44:** 2.6 / 2.8 / 3.8 / 3.8 / 4.4

**RelyX Unicem Aplicap:** 1.2 / 2.6 / 3.1 / 4.9 / 6.4

**RelyX Unicem Clicker:** 3.2 / 3.9 / 4.1 / 4.4 / 5.9

**iCEM:** 0.8 / 2.2 / 2.3 / 3.0 / 3.3

**MaxcemElite:** 1.4 / 2.5 / 3.0 / 3.6 / 4.5

**Bifix SE:** 1.3 / 1.5 / 1.7 / 2.1 / 2.4

**SpeedCEM:** 0.1 / 1.2 / 1.3 / 1.7 / 2.8

**Panavia™ 21:** 0.2 / 0.6 / 1.7 / 2.1 / 4.4



**Secure:** 1.1 / 2.2 / 3.0 / 3.6 / 4.4

**Ketac Cem:** 0.4 / 1.0 / 1.4 / 1.8 / 3.2

**Meron Plus:** 1.2 / 3.0 / 3.1 / 3.4 / 5.4

**Harvard:** 1.2 / 3.0 / 3.1 / 3.4 / 5.4

# COMPOSITE RESIN

CLEARFIL™ AP-X

## CLEARFIL™ AP-X

### 135039 Study on New Low-shrinkage Resin Composite

*Y. HATSUOKA, H. TANIMOTO, O. TAKEUCHI, and K. YAMAMOTO, Osaka Dental University, Osaka, Japan*

**Purpose:** This study was conducted to evaluate the marginal adaptation of the new low-shrinkage resin composite (CR) in combination with the attached bonding system. **Methods:** The new low-shrinkage CR and the bonding system used were Filtek™ P90 (P90, 3M ESPE) and P90 bonding system (P90S, 3M ESPE). As the control, CLEARFIL™ AP-X (AP-X, Kuraray Medical) with CLEARFIL™ SE Bond (SB, Kuraray Medical) and Filtek™ Supreme DL (DL, 3M ESPE) with Adper™ Easy Bond (EB, 3M ESPE) were used. **Contraction gap (GAP):** A cylindrical cavity was prepared in the extracted bovine dentin. After the bonding systems were used, CR was cured (n=10). GAP was observed using an optical microscope. **Marginal leakage:** An oval-shaped cavity was prepared in the extracted human molars. CR was filled in the cavity and cured (n=10). Specimens were thermal-

cycled for 5.000 times (5-55°C). Marginal leakage was assessed after immersing in fuchsin solution (0-3 scales). The data were analyzed by the Kruskal-Wallis and Mann-Whitney tests (p>0.05). **Results:** GAP: No GAP was found for AP-X+SB. 1µm GAP was detected for 3 specimens of P90+P90S. 1-3µm GAP was found for 7 specimens of DL+EB. GAP of AP-X+SB was significantly smaller than DL+EB. **Marginal leakage:** CR systems and Marginal leakage scores (specimen's number) were as follows; P90+P90S 0(3) 1(7) 2(0), AP-X+SB 0(5) 1(5) 2(0), DL+EB 0(2) 1(7) 2(1) at enamel. P90+P90S 0(8) 1(2) 2(0), AP-X+SB 0(8) 1(2) 2(0), DL+EB 0(4) 1(3) 2(3) at dentin. No significant difference was found, but **marginal leakage of P90+P90S and AP-X+SB tended to be smaller than EB+DL.**

#### ➔ Conclusions:

It was suggested that P90 in combination with P90S may bring about the satisfactory performance in the clinical situations.

## CLEARFIL™ AP-X

### 138620 Longevity of Direct Composites in the Focus of Cyclic Fatigue

U. LOHBAUER, R. BELLI, and A. PETSCHL, University of Erlangen, Erlangen, Germany

**Objectives:** To assess the in vitro cyclic fatigue resistance of modern universal resin composites for posterior indications. **Methods:** Nine commercial composite resins were selected: CeramX Duo, Dentsply, (CX); Clearfil AP-X, Kuraray, (CL); Grandio, VOCO, (GR); Miris II, Coltène, (MS); Filtek Supreme XT, 3M ESPE, (FS); Filtek Silorane, 3M ESPE, (SI); Filtek Z250, 3M ESPE, (FZ); Tetric EvoCeram, Ivoclar, (EV); and Venus Diamond, Heraeus, (VD). Bar specimens with dimensions of 25x2x2 mm were fractured

in an universal testing machine (Z2.5, Zwick) using the four-point-bending test. Flexural strength (FS,  $n = 15$ ) and flexural fatigue limit (FFL,  $n = 25$ , 104 cycles,  $v = 0.5$  Hz) were measured after 14 days water storage at 37°C. Cyclic fatigue was performed according to a “staircase”-approach. The data were analyzed by ANOVA (mod LSD post hoc,  $\alpha=0.05$ ). **Results:** [Mean (SD)], same letters indicate homogenous subsets.

| Material (Manufacturer)    |    | FS [MPa]        | FS [MPa]     | FFL [%] |
|----------------------------|----|-----------------|--------------|---------|
| CeramX Duo (Dentsply)      | CX | 90.1 (6.9) d    | 39.7 (4.9) E | 55.9    |
| Clearfil AP-X (Kuraray)    | CL | 126.6 (13.9) a  | 80.5 (7.9) A | 36.4    |
| Grandio (Voco)             | GR | 114.9 (11.1) b  | 63.0 (4.8) B | 45.2    |
| Filtek Supreme XT (3MESPE) | FS | 106.1 (8.4) c   | 55.5 (4.5) C | 47.7    |
| Filtek Silorane (3MESPE)   | SI | 112.4 (11.7) bc | 62.6 (6.7) B | 44.3    |
| Filtek Z250 (3MESPE)       | FZ | 131.9 (12.6) a  | 55.2 (8.8) C | 58.1    |
| Miris II (Coltene)         | MS | 117.4 (9.2) b   | 44.7 (5.2) D | 61.9    |
| Tetric EvoCeram (Ivoclar)  | TE | 90.6 (4.2) d    | 43.2 (3.8) D | 52.3    |
| Venus Diamond (Heraeus)    | VD | 130.1 (11.1) a  | 44.4 (2.1) D | 65.9    |

#### ➔ Conclusions:

The initial strength level of the composites range between 90 MPa (CX) and 132 MPa (FZ). However, after cyclic fatigue, a decrease in strength between 36% (CL) up to 65.9% (VD) was observed. Cyclic flexural loading significantly reduces strength of composite resins, being this decrease highly material dependent.

## CLEARFIL™ AP-X

### 131272 Surface roughness of two composites after etching with various acids

B. LOOMANS<sup>1</sup>, M. CARDOSO<sup>2</sup>, N. OPDAM<sup>1</sup>, J. ROETERS<sup>1</sup>, J. DE MUNCK<sup>2</sup>, M.-C. HUYSMANS<sup>1</sup>, and B. VAN MEERBEEK<sup>2</sup>, <sup>1</sup>Radboud University Nijmegen Medical Centre, Nijmegen, Netherlands, <sup>2</sup>Catholic University of Leuven, Leuven, Belgium

**Purpose:** To investigate the effect of various etching protocols on the surface roughness of composites with different filler compositions. **Methods:** From a micro-hybrid composite containing barium-glass (Clearfil AP-X, Kuraray) and a nano-hybrid composite containing zirconia/silica nanoclusters (Filtek Supreme XT, 3M ESPE), specimens of 3mm thick with a diameter of 7mm were prepared (n=24). The top surface was polished with 4000-grit abrasive paper and subjected to one of eight surface treatment techniques (n=3): negative control, phosphoric acid for 20sec (H<sub>3</sub>PO<sub>4</sub>), 3% hydrofluoric acid for 20sec (3%HF-20sec), for 120sec (3%HF-120sec), 9.6% hydrofluoric acid for 20sec (9.6%HF-20sec), for 120sec (9.6%HF-120sec), H<sub>3</sub>PO<sub>4</sub> followed by 9.6%HF-120sec (H<sub>3</sub>PO<sub>4</sub>-HF), and 9.6%HF-120sec followed by H<sub>3</sub>PO<sub>4</sub> (HF-H<sub>3</sub>PO<sub>4</sub>). Roughness (Sa) was measured using a 3D-noncontact optical inter-

ferometer (WYKO, Veeco) and SEM evaluation was performed. ANOVA and posthoc Tukey HSD multiple comparisons were used to determine statistical differences in roughness between groups (p<0.05).

**Results:** The micro-hybrid barium-containing composite demonstrated a significant rougher surface than the nano-hybrid zirconium-containing composite (p<0.05). For the micro-hybrid composite, 3%HF-20sec, 3%HF-120sec and 9.6%HF-20sec resulted in a significant increase in roughness (p<0.001). For the nano-composite, only 3%HF-20sec and 9.6%HF-20sec showed an increase in roughness (p<0.02). For both composites, 9.6%HF-120sec, PA-HF and HF-PA resulted in a large increase in roughness compared to control (p<0.001). **From SEM images, it was found that the micro-hybrid composite was much more affected by etching than the nano-hybrid composite.**

|   | Sa [nm] (SD)  |              |
|---|---------------|--------------|
|   | Clearfil AP-X | Supreme XT   |
| Control   | 55.8 (15.6)   | 33.6 (6.31)  |
| H <sub>3</sub> PO <sub>4</sub> 20s                | 71.7 (8.8)    | 35.3 (4.36)  |
| 3% HF 20s   | 107.2 (9.2)   | 32.3 (3.62)  |
| 3% HF 120s  | 138.1 (16.5)  | 44.2 (6.80)  |
| 9.6% HF 20 sec                                    | 172.6 (16.5)  | 46.3 (3.86)  |
| 9.6% HF 120 sec                                   | 558.9 (33.8)  | 277.3 (25.3) |
| H <sub>3</sub> PO <sub>4</sub> 20s + 9.6% HF 120s | 542.2 (35.0)  | 255.9 (17.1) |
| 9.6% HF 120s + H <sub>3</sub> PO <sub>4</sub> 20s | 555.4 (35.4)  | 378.7 (14.2) |

#### ➔ Conclusions:

The surface roughness after etching is largely affected by the composition of the filler particles in the composite.

# ADHESIVE TECHNOLOGY

## BY KURARAY

MDP Monomer



## MDP Monomer

### 131411 The AD-concept revisited as basis for durable tooth bonding

B. VAN MEERBEEK<sup>1</sup>, Y. YOSHIDA<sup>2</sup>, and K. SUZUKI<sup>1</sup>, <sup>1</sup>Catholic University of Leuven, Leuven, Belgium, <sup>2</sup>Okayama University, Okayama, Japan

**The Adhesion-Decalcification concept (Yoshida et al., JDR 2001) dictates how molecules interact with hydroxyapatite-based tissues.**

**Purpose:** To revisit the AD-concept as thought to be fundamental for durable bonding, and to explore why 'mild' self-etch adhesives less effectively bond to enamel than dentin. **Methods:** The interaction of three monomers (4-MET/10-MDP/phenyl-P) with hydroxyapatite (HAp), and for an experimental 10-MDP:EtOH:H<sub>2</sub>O (15:45:40wt%) self-etch primer also with dentin/enamel, was characterized by XRD, and further complemented with TEM-interfacial ultra-structural data upon its reaction with dentin. Finally, the tensile bond strength (TBS) to enamel/dentin of the primer (combined with Clearfil SE 'Bond') with and without 1wt% camphorquinone (CQ) added,

was measured [Clearfil SE (C-SE, Kuraray) served as control]. **Results:** Regarding molecular interaction, the two extremes were phenyl-P that rather 'etched' and 10-MDP that rather 'bonded' to HAp (with 4-MET behaving somewhat in between). 'Etching' was associated with more substantial/rapid DCPD (CaHPO<sub>4</sub> · 2H<sub>2</sub>O) deposition, while 'bonding' was represented by the formation of a Ca-monomer nano-layered structure (confirmed by TEM). XRD revealed a higher crystallite size/crystallinity for enamel, while nano-layering was more pronounced at dentin. Nano-layering was enhanced by actively rubbing the surface with the exp. primer for 20sec (than when enamel/dentin was solely immersed for 30min in 5ml primer). The exp. primer needed CQ to obtain a TBS to dentin comparable to that of C-SE, but not when bonded to enamel.

#### ➡ Conclusions:

Stable ionic-bond formation to HAp competes in time with the deposition of less stable calcium-phosphate salt deposition (DCPD). For durable bonding, Ca-monomer salt formation should precede/exceed DCPD deposition. The lower bonding effectiveness of mild self-etch adhesives to enamel should be ascribed to a lower chemical reactivity (nano-layering) with enamel-HAp, in combination with less potential for micro-mechanical interlocking (as for instance achieved by phosphoric-acid etching). Adequate polymerization (by CQ) is needed to stabilize the formed nano-layer.

# RESEARCH & DEVELOPMENT BY KURARAY

New hydrophilic monomer

## Research & Development

### 133710 New hydrophilic cross-linking monomers for dental adhesive

*N. NISHIGAKI, T. SEKIGUCHI, H. ISHINO, A. HINAMOTO, and K. OKADA, KURARAY MEDICAL INC, Kurashiki, Japan*

**Purpose:** The purpose of this study was to evaluate the performance of new hydrophilic cross-linking monomers with two polymerizable groups and two or more hydroxyl groups, such as erythritol dimethacrylate (EDMA), pentaerythritol dimethacrylate (ErMA) and compared these new monomers with HEMA and TEGDMA in current dental adhesives. **Methods:** Two experimental one-step bonding agents were made from the new hydrophilic cross-linking monomers containing an adhesive phosphate monomer (MDP), Bis-GMA, HEMA, water, ethanol and photo initiators. Tensile bond strength of the experimental one-step bonding agents to bovine dentin cured with a halogen light unit (JETLITE3000/J.Morita) was measured after storing in 37°C water for 24 hours. Further-

more, hydrophilicity and polymerization property of these new monomers themselves were evaluated by partition coefficient (LogP) and degree of conversion respectively. **Results:** Table 1 shows the tensile bond strength of each experimental one-step bonding agent and properties of new hydrophilic cross-linking monomers. Experimental one-step bonding agents, which contain new hydrophilic cross-linking monomers, showed higher bond strength than bonding agents which contain HEMA or TEGDMA as substitutes for the new monomers. New hydrophilic cross-linking monomers showed higher degree of conversion than HEMA and higher hydrophilicity than TEGDMA.

|  | EDMA | ErMA | HEMA | TEGDMA |
|--|------|------|------|--------|
| Bond strength of experimental one-step bonding agent to dentin (MPa) | 16.9 | 15.1 | 14.1 | 11.7   |
| Partition coefficient (LogP)   | 1.1  | 1.5  | 0.3  | 2.2    |
| Degree of conversion (%)   | 42   | 27   | 24   | 25     |

#### ➡ Conclusions:

The evaluated new hydrophilic cross-linking monomers with high hydrophilicity and polymerization property were effective to improve the bond strength of one-step bonding agents.

# FUTURE KURARAY PRODUCTS IN EUROPE

CLEARFIL™ FIBERPOST

## CLEARFIL™ FIBERPOST

### 134955 In-vitro Durability of a New Glass Fiber Post

E. TERAKAWA, H. KITTA, and D. WEERASINGHE, Kuraray Medical Inc, Tokyo, Japan

**Purpose:** The purpose of this in-vitro study was to evaluate the durability of a new glass fiber post, "CLEARFIL™ FIBERPOST" (Kuraray Medical Inc., KM). **Materials and methods:** Durability of CLEARFIL™ FIBERPOST was compared with three other commercially available posts of same diameter (1.2~1.3mm); GC Fiberpost (GC Corp.), Tokuyama FR-Post (Tokuyama Dental Corp.), FibreKor Tapered Post (Pentron Japan Inc.). Five specimens from each of the above groups were checked for three-point flexural strength. Same test was repeated after thermo-cycling (TC, 20.000 cycles 4°C / 60°C, dwell time: 1min, change time: 6s) and flexural strength ratio (post durability) before and after TC was calculated. Similarly thermo-cycled specimens (n>7 for the each group) were pre-treated with approximately 40% phosphoric acid (K-ETCHANT Gel, KM) for 5s, washed and dried. Each post was silane

pre-treated (CLEARFIL™ Ceramic Primer, KM) and cemented at a 5mm depth of a prepared cylindrical copper mold space with dual-cure resin cement (PANAVIA™ F2.0, KM). A 2mm thick rubber spacer was placed on top of the copper mold and core build-up was done (CLEARFIL™ DC Core Automix, KM). After 24h storage in a 37°C incubator, mold was fixed at an angle of 20 degrees to the long-axis of the post and 5N - 50N cyclic-load applied in-contact mode at 1Hz to the core build-up (Figure1) and fracture resistance was measured by the number of cycles lead to fracture. The flexural strength data was statistically analyzed using ANOVA followed by Scheffe's test (p=0.05). Flexural strength ratio and fracture resistance were analyzed by Mann-Whitney test. **Results:** Results are shown in Table 1. Different superscripts show astatistically significant difference.

|                              |          | CLEARFIL™<br>FIBERPOST       | GC<br>Fiberpost            | Tokuyama<br>FR-Post         | FibreKor<br>Tapered Post |
|------------------------------|----------|------------------------------|----------------------------|-----------------------------|--------------------------|
| Flexural strength<br>(MPa)   | None TC  | 1190 (93) <sup>a, b, d</sup> | 1312 (163) <sup>b, d</sup> | 1121 (219) <sup>a, b,</sup> | 1445 (82) <sup>d</sup>   |
|                              | TC 20000 | 981 (83) <sup>a</sup>        | 601 (52) <sup>c</sup>      | 550 (110) <sup>c</sup>      | 970 (83) <sup>a</sup>    |
| Post durability (%)*         |          | 83 (4) <sup>A</sup>          | 46 (3) <sup>B</sup>        | 49 (2) <sup>B</sup>         | 67 (2) <sup>C</sup>      |
| Fracture resistance (cycles) |          | 2952 (910) <sup>α</sup>      | 42 (14) <sup>β</sup>       | 29 (7) <sup>β</sup>         | 488 (278) <sup>γ</sup>   |

\* Flexural strength ratio:  $[TC20000]/[None TC] \times 100$

#### ➡ Conclusions:

Within the limits of this study, in-vitro durability of CLEARFIL™ FIBERPOST was significantly higher than the evaluated commercially available fiber posts.

## CLEARFIL™ FIBERPOST

### 137766 Influence of water immersion on mechanical properties of fiber posts

W. KOMADA, N. KUMAGAE, Y. FUKUI, S. OTAKE, D. OKADA, K. YOSHIDA, and H. MIURA, Tokyo Medical & Dental University, Tokyo, Japan

**Objectives:** The aim of this study was to evaluate the influence of water immersion on the flexural strengths and elastic modulus of three kinds of glass fiber posts. **Methods:** Two commercially available glass fiber posts (FIBER POST, GC, Japan (GP) and FibreKor Tapered Post, Pentron, U.S.A. (PP) and a newly developed [glassfiber post CLEARFIL™ FIBERPOST, Kuraray medical, Japan \(KP\)](#)) were used for this study. Each fiber post was divided into two groups; a control (GPC, PPC and KPC) and a water immersion group (GP30, PP30 and KP30). In the water immersion group, the specimens were stored in deionized water for 30 days at 37°C in darkness. Flexural strengths and elastic modulus were determined using a universal testing machine (Auto Graph AGS-H, Shimadzu, Japan) at crosshead speed of 1.0 mm/min. The data were analyzed by two-way ANOVA

and t-test with Bonferroni correction ( $p < 0.05$ ). After the test of mechanical strength, glassfiber-matrix resin interface of specimens were observed using SEM (FE SEM XL30S, PHILIPS, U.S.A.). **Results:** In the flexural strength, GP30 ( $657.5 \pm 94.7$  MPa) showed significant lower value than GPC ( $885.1 \pm 49.5$  MPa). PP30 ( $775.8 \pm 41.7$  MPa) showed significant lower value than PPC ( $885.7 \pm 37.3$  MPa). KP30 ( $723.1 \pm 53.5$  MPa) and KPC ( $770.5 \pm 51.9$  MPa) showed no significant difference. In the elastic modulus, GP30 ( $10.9 \pm 0.31$  GPa) showed significant lower value than GPC ( $11.7 \pm 0.14$  GPa). PP30 ( $12.3 \pm 0.17$  GPa) showed significant lower value than PPC ( $12.5 \pm 0.27$  GPa). KP30 ( $10.2 \pm 0.20$  GPa) and KPC ( $10.4 \pm 0.23$  GPa) showed no significant difference. In water immersion groups, some glassfiber-matrix resin interfacial gaps were observed by SEM.

#### ➔ Conclusions:

The results suggested that water immersion had little influence on the mechanical properties of KP.

## NOTES

**CLEARFIL™, PANAVIA™ and CLEARFIL MAJESTY™ are trademarks of the company Kuraray Medical Inc..**

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Kuraray Europe GmbH

Kuraray Europe GmbH  
BU Medical Products  
Hoechst Industrial Park | Building F 821  
65926 Frankfurt am Main | Germany

Phone: +49 69 305 35825  
Fax: +49(0)69- 305 98 35825  
[www.kuraray-dental.eu](http://www.kuraray-dental.eu)  
E-mail: [dental@kuraray.eu](mailto:dental@kuraray.eu)

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