

**kuraray**



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COMPILATION  
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ABSTRACTS

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# COMPANY PROFILE



## More than 85 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.

This leads to the development and production of a broad range of high-quality and innovative products.

## Inventor of the bonding system

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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.

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 MAJESTY™ and ESTENIA™ C&B – are proof of Kuraray's capability to develop unique solutions for the dental professionals as results of pioneering research.

## Entering the era of synergy

Kuraray Noritake Dental Inc. was born on 1 April 2012 from Kuraray Medical Inc. and Noritake Dental Supply Co., Limited.

Kuraray Noritake Dental Inc. will promote the development of materials that possess new functions by integrating technologies from the area of organic dental materials (including bonding agents, resin cements and composites) and the area of inorganic dental materials (including ceramics for crowns and plaster). The future of dentistry looks bright for materials born out of the integration of our technologies.

## The real test starts with the usage of our products

We are delighted to present with this compilation of abstracts 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, we express our gratitude to the Universities for including our products in their research.

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



Satoshi Yamaguchi  
Head of BU Medical  
Kuraray Europe GmbH



Naofumi Murata  
Head of Technical Services & Marketing  
Kuraray Europe GmbH



# ADHESIVES

CLEARFIL™ S<sup>3</sup> BOND PLUS  
CLEARFIL™ S<sup>3</sup> BOND  
CLEARFIL™ SE BOND  
CLEARFIL™ PROTECT BOND

## CLEARFIL™ S<sup>3</sup> BOND PLUS<sup>1</sup> · CLEARFIL™ S<sup>3</sup> BOND

### IADR 3052 Effect of a pulpal-pressure on dentin-bonding of single-bottle, one-step self-etching adhesives

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**Objectives:** To evaluate the effect of simulated pulpal pressure on microtensile bond strengths of 3 single-bottle, one-step self-etching adhesives to dentin, and the quality of resin-dentin interfaces.

**Methods:** Ninety-six non-carious human third molars were divided into 3 groups: experimental (MTB-200; Kuraray Medical Inc.); Clearfil S3 Bond (C3S; Kuraray) and iBond Self-Etch (IB; Heraeus-Kulzer). Each group was subdivided into two subgroups (N=10). Specimens in subgroup A were bonded under zero pulpal pressure, while subgroup B specimens were bonded under 15 cm H<sub>2</sub>O pressure (PP). Each tooth was bonded to mid-coronal dentin, followed by incremental placement of a light-cured nano-hybrid resin composite. Each bonded tooth was sectioned into 0.9 x 0.9 mm sticks and stressed to failure using a universal testing machine at a cross-head speed of 1 mm/min. Data were analyzed using two-way ANOVA and Tukey-Kramer multiple-comparison procedures, to examine the effects of “adhesive-type” and “pulpal-pressure”, and their interactions on bond strength ( $\alpha=0.05$ ). Remaining teeth in each subgroup (N=6) were used for microscopy, using rhodamine as a tracer dye for CLSM, and ammoniacal silver nitrate as a tracer dye for SEM and TEM.

**Results:** Microtensile bond strengths (mean $\pm$ SD; in MPA) were 33.4 $\pm$ 6.9 (MTB) 33.2 $\pm$ 4.7 (MTB-PP), 35.0 $\pm$ 8.6 (C3S), 25.5 $\pm$ 7.3 (C3S-PP), 18.4 $\pm$ 4.0 (IB), 16.5 $\pm$ 6.9 (IB-PP). “Adhesive-type” (P<0.001), “pulpal-pressure” (P<0.001), and their interactions (P<0.001) significantly affected bond strength results. Without PP, bond strengths were, in descending order: MTB=C3S>IB. With PP, bond strengths were, in descending order: MTB-PP>C3S-PP>IB-PP. No difference between no-PP and PP subgroups were found for MTB and IB (P>0.05). CLSM, SEM and TEM showed extensive water droplets along the resin-dentin interface for IB, IB-PP and C3S-PP.

#### ➔ Conclusion:

IB exhibits water sensitivity when bonding is performed with/without pulpal pressure. C3S exhibits water sensitivity when bonding is performed with pulpal pressure. **MTB does not exhibit water sensitivity** when bonding is performed with/without pulpal pressure.

# CLEARFIL™ S<sup>3</sup> BOND PLUS · CLEARFIL™ SE BOND CLEARFIL™ PROTECT BOND<sup>2</sup>

## AADR 794 Bonding Performance of a New Adhesive »CLEARFIL TRI-S BOND PLUS«

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Kuraray Medical Inc. has developed a new self-etching adhesive “CLEARFIL TRI-S BOND PLUS (TSP)”. TSP is a single-step fluoride-releasing bond system comprised of an adhesive phosphate monomer (MDP), methacrylate monomers, water, ethanol, initiators, sodium fluoride and filler. It contains an original hydrophilic dimethacrylate in combination with HEMA to achieve optimal balance between curing property and hydrophilicity.

**Objectives:** The purpose of this study was to compare the tensile bond strength (TBS) of TSP to bovine enamel and dentin with other commercial 1-step and 2-steps self-etching adhesives; Opti-Bond All-In-One (AIO)/ Kerr (1-step) and OptiBond XTR (XTR)/ Kerr (2-steps) which contains GDMP as an adhesive monomer, CLEARFIL SE BOND (SEB)/

Kuraray Medical (2-steps) and CLEARFIL SE PROTECT (SEP)/ Kuraray Medical (2-steps) which contains MDP as well as TSP. **Methods:** Each adhesive was applied to bovine enamel or dentin according to the manufacturer’s instructions. TBS after 24 hrs immersion and thermo cycling (4°C-60°C, 1 min. each, 4,000 cycles: TC4000) was measured using a universal testing instrument (Shimadzu).

**Results:** The TBS data are shown in Table 1. **TSP, MDP-based optimal 1-step formulation, showed higher bond strength to bovine enamel than AIO and XTR both after 24 hrs and TC4000.** Regarding dentin bonding, TBS of TSP after TC4000 was higher than that of AIO, and it was comparable to 2-steps system.

Table 1 / MPa (SD)

		Bovine Enamel		Bovine Dentin	
		37°C, 24 hrs	TC4000	37°C, 24 hrs	TC4000
1-step	TSP	20.4(4.5)	20.5(6.1)	18.8(4.3)	21.7(2.9)
	AIO	13.0(2.5)	16.3(2.1)	13.9(2.4)	16.4(2.1)
2-steps	SEB	22.1(6.3)	21.8(7.4)	22.9(6.0)	22.5(3.8)
	SEP	22.6(4.5)	21.3(3.9)	22.7(4.0)	21.9(4.2)
	XTR	16.8(7.0)	16.2(6.5)	22.6(5.4)	19.9(3.7)

### ➔ Conclusion:

This result indicated that “CLEARFIL TRI-S BOND PLUS” might exhibits reliable clinical performance equal or superior to 1-step and 2-steps adhesives used in this study.



# CLEARFIL™ S<sup>3</sup> BOND PLUS<sup>1</sup>

## CLEARFIL™ S<sup>3</sup> BOND

### PER/IADR 488 Bond durability of contemporary and experimental all-in-one adhesives

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**Objectives:** The objective of the present study was to evaluate the long-term bond durability of all-in-one adhesives using a PCR thermal cycler.

**Methods:** Human third molars were used, removing upper half of crown segment and were ground to reveal its dentin surface (#600SiC paper, water). Resin composite (CLEARFIL AP-X, Kuraray) was bonded to dentin using either a commercial adhesive CLEARFIL TRI-S BOND (Kuraray) or an experimental adhesive MTB200 (Kuraray). Both of adhesives contained HEMA, but experimental one included hydrophobic monomers and improved initiation system. Specimens were sectioned into beams with the

cross-sectional area of 1.0 mm<sup>2</sup>. Group 1: Half of beams were performed microtensile bond strength test, after 1day storage in water. Group 2: Other beams were performed microtensile bond strength test, after being provided for PCR thermal cycle of 20,000 times taking approximately 2 months. The obtained data were expressed as MPa and statistically analyzed (one-way ANOVA, Duncan). Fracture surfaces were observed (SEM, TEM). **Results:** Mean microtensile bond strength values (MPa±SD) are summarized in the table. The same letters mean that there are no significant differences among the groups ( $p>0.05$ ). There was no pretesting failure.

Adhesive	1day	20,000 thermal cycles
CLEARFIL TRI-S BOND	64.78±14.19ab (n=28)	58.71±12.86b (n=29)
MTB200	72.52±17.17a (n=28)	69.70±19.30a (n=29)

#### ➔ Conclusion:

**MTB200 showed comparable or better bonding performance compared with CLEARFIL TRI-S BOND over time.**

## CLEARFIL™ S<sup>3</sup> BOND

### IADR 2340 Quality of adhesion with self-etch systems after prolonged exposure times

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**Objectives:** This study evaluated the effect of prolonged exposure times on immediate (IM) and 6-month (6M) adhesive properties: degree of conversion within hybrid layer (DC), silver nitrate uptake (SNU) and resin-dentin bond strength ( $\mu$ TBS) for three one-step self-etch adhesive systems (Adper Easy Bond [EB], Clearfil S3 Bond [CS3] and GO [GO]). **Methods:** Ninety caries-free extracted molars were included in this study being 45 for  $\mu$ TBS and SNU (n=5) and 45 for DC (n=5). The dentin surfaces were bonded according to the manufacturer's instructions, but they were light-cured for 10, 20, and 40 s at 600mW/cm<sup>2</sup>. Bonded teeth for the  $\mu$ TBS test were sectioned in order to obtain stick-like specimens (0.8mm<sup>2</sup>) and tested under tensile stress (0.5mm/min). Two bonded sticks from each tooth were analyzed by scanning electron microscopy for SNU. The DC within the hybrid layer were evaluated with micro-Raman spectroscopy. All tests were performed IM and at 6M. Data were analyzed by a three-way ANOVA and Tukey's test ( $\alpha=0.05$ ).

**Results:** The prolonged exposure times increased

the DC (%) [10s- 67 $\pm$ 19; 20s- 85  $\pm$  12 and 40s- 83  $\pm$  13] for all adhesives and decreased the SNU (%) [10s- 25 $\pm$  13; 20s- 13  $\pm$  7 and 40s- 13  $\pm$  9] significantly for all adhesives; **however significant improvements in the  $\mu$ TBS and stable bond strengths after water storage were observed only for CS3.** EB [IM- 36  $\pm$  5; 6M- 28  $\pm$  6] and GO [IM- 26  $\pm$  5; 6M- 14  $\pm$  4] decreased the  $\mu$ TBS after 6 months. EB

#### ➔ Conclusion:

The prolonged exposure times improved the DC within the hybrid layer and reduced the SNU for all adhesives, however this did not prevent the degradation of the adhesive interface.

## CLEARFIL™ SE BOND

### PER/ADR 109 Shear-bond-strength of a New Single-bottle-adhesive to Pulp-chamber-dentin Pretreated with Various-irrigants

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**Objectives:** The aim of this in vitro study was to evaluate the shear-bond-strength(SBS) of a new single-bottle adhesive (Single-Bond Universal, 3M ESPE-Germany)) in comparison with a well known self-etch adhesive system (Clearfil SE-Bond, Kuraray-Japan) to dentin pretreated with 2% chlorhexidine (CHX-Drogsan/Turkey), 2.5% sodium hypochlorite (NaOCL-ÇağlayanKimya/Turkey) and octenisept dihydrochloride (OCT-Schülke&Mayr/Germany) solutions. **Methods:** One hundred-twenty human mandibular third molars were sectioned 3 mm below the occlusal level and were then separated longitudinally in a mesiodistal direction to produce a total of 240 specimens having pulp-chamber-dentin. The specimens were grounded flat with silicon carbide abrasive papers (#600-1200), embedded in auto-polymerizing acrylic resin and randomly divided into four groups (n=60) according to the irrigants used; G1(control), distilled water; G2, 2.5% NaOCl; G3, 2% CHX; G4, OCT for 30 minutes. Groups were then divided into 2 subgroups according to the restoration material used (n=15) and restored either with the Single Bond Universal together with Filtek™ Supreme Ultra Universal Restorative Composite (3M ESPE, USA) or Clearfil SE-Bond together with Clearfil AP-X- Composite (Kuraray/Japan) using Ultradent's device(USA). The specimens were then submitted to the SBS testing in a Universal Testing Machine at a crosshead speed of 1 mm min-1until fracture. Data were statistically analysed (Two-way ANOVA and Tukey HSD tests).

**Results:** Clearfil SE-Bond demonstrated significantly better SBS values on all pretreated surfaces than the Single-Bond Universal-adhesive ( $P<0.05$ ). Statistical ranking of SBS values of Single-Bond Universal on all pretreated surfaces was as follows; Control  $>$ OCT  $\geq$  CHX  $\geq$  NaOCL. Clearfil SE-Bond demonstrated equal SBS on CHX affected dentin as controls. SBS of Clearfil SE-Bond decreased when dentin-surfaces exposed to NaOCL and OCT ( $P<0.05$ ).

#### ➔ Conclusion:

**Clearfil SE-Bond has better SBS values on irrigant-pretreated dentin than the new single-bottle adhesive Single-Bond Universal.** CHX could be safely-used as a final root-canal irrigant in endodontic cases when pulp-chamber-wall restored with Clearfil SE-Bond/composites.

## CLEARFIL™ SE BOND

### PER/IADR 378 Clinical Evaluation of Self-etch Adhesives in Class V Carious Lesions

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**Objectives:** The purpose of this randomized, controlled clinical study was to evaluate the three years clinical performance of four self-etch adhesive systems in combination with a nano-composite for the restoration of Class V carious lesions.

**Methods:** Thirty-three patients with at least 2 equivalent carious cervical lesions were enrolled in the study. One hundred and twenty lesions were restored with a nano-composite (Grandio SO, VOCO, Germany) (n=30/per adhesive system) according to the manufacturer's instructions by one operator. The tested adhesives were; Futurabond DC (VOCO), Clearfil SE Bond (Kuraray, Japan), Adper SE Plus (3M-Espe); G-Bond (GC- Europe). Two other independent examiners clinically evaluated the restorations for retention, colour match, marginal adaptation, anatomic form, marginal discoloration, recurrent caries, post-operative sensitivity and surface texture at baseline, one year, two-year and three years according to the modified USPHS criteria. Statistical analysis was completed using the Pearson Chi-square and Fisher's Exact Test ( $p < 0.05$ ).

**Results:** After 3 years, Twenty-six patients were available for recall and 99 restorations were evaluated (recall rate of 82 %). Three restorations of Adper SE and three restorations of G Bond were lost after three years. Futurabond Dc and SE Bond showed no loss of restorations after three years. There was statistically significant difference between the tested adhesives after three years ( $p > 0.05$ )

#### ➔ Conclusion:

Considering all the clinical evaluation criteria, Futurabond DC and Clearfil SE Bond showed superior performance than Adper SE and G bond after three years.

## CLEARFIL™ SE BOND

### AADR 715 Three-Dimensional Characterization of Nanoleakage Using Serial Ion-Ablation Scanning Electron Microscopy

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**Objectives:** The aim of this study was to evaluate the application of serial ion-ablation scanning electron microscopy (SIA-SEM) to evaluate the nanoleakage of resin-bonded dental interfaces. **Methods:** Eight recently extracted molars were selected and the middle dentin exposed. Hypersensitive dentin was created in half of the specimen by immersing middle dentin into EDTA solution (pH=8.0) for 2 minutes, while the other half middle dentin were left intact (H: hypersensitive, I: intact). All specimens were subject to a two-step etch-and-rinse ethanol- and water-based dentin adhesive (Adper Single Bond Plus - SB) and a two-step self-etch dentin adhesive (Clearfil SE Bond, CSE). All teeth were restored with a microhybrid composite (Filtek Z250). The specimens were immersed in 50wt% ammoniacal silver nitrate, and processed for electron microscopy. Serial ion-ablation scanning electron microscopy (SIA-SEM) specimen analysis was carried out using a dual beam focused ion beam (FIB). Using the electron imaging, a specific area of the hybrid layer displaying nanoleakage was identified and selected for investigation. Stacks of several hundred sections, 20 nm thick were imaged using 2kV 0.2 nA electron beam current. Imaging registration, 3D reconstruction and image analysis was done using Image J and Amira 5.2.2 software. Quantitative analysis of volumetric silver-uptake ( $\text{Ag}/\mu\text{m}^3$ ) was performed and the data submitted to t-Test ( $\alpha=.05$ ).

**Results:** The shape, direction, location, and amount of silver uptake changed drastically depending upon the location evaluated within the same specimen. Quantitative analysis of **3D images confirm more overall silver uptake for etch-and-rinse adhesive** (SBI:  $1835.7 \pm 214.2 \text{ Ag}/\mu\text{m}^3$ , SBH:  $718.5 \pm 40.9 \text{ Ag}/\mu\text{m}^3$ ) **than that of the self-etch adhesive** (CSEI:  $153.8 \pm 4.5 \text{ Ag}/\mu\text{m}^3$ , CSEH:  $38.9 \pm 2.06 \text{ Ag}/\mu\text{m}^3$ ), irrespective of dentin surface.

#### ➔ Conclusion:

Nanoleakage is a dynamic and deleterious process that affects the sealing efficacy of bonded restorations. 3D reconstruction of the bonded interfaces allows better understanding and interpretation of nanoleakage.

## CLEARFIL™ SE BOND

### IADR 2291 Effect of Desensitizing Agents on Teeth with Hypersensitive Dentine

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**Objectives:** The objective of this study was to evaluate the effects of desensitizing agents on pain sensation to air blast and cold stimulation and the micro-morphology of exposed hypersensitive dentine in human subjects.

**Methods:** The experiments were carried out on 8 buccal cervical hypersensitive dentine of premolars in 7 subjects (aged 18-60 yr). The study was approved by the university ethic committee. After identified the exposed hypersensitive dentine area, air-blast and cold (water 4°C) stimulation were applied for 3 s. The subject indicated the intensity of any pain produced by placing a mark on a 100 mm visual analog scale (VAS). Then, silicone rubber impressions (Xantopren®, Heraeus, Germany) were taken of the cervical surface of the tooth. This method of testing dentine sensitivity and the impression were repeated again after application of Colgate® Sensitive Pro-relief™ for 5 min then washing with water and after application of SE bond (Kuraray, Japan). Epoxy resin replicas were made from the impressions and examined in a scanning electron microscope.

**Results:** For air blast stimulus, both Colgate® Sensitive Pro-relief™ and SE bond, the mean VAS scores decreased significantly ( $P < 0.05$ , 1way RM ANOVA, Tukey test) from  $50.0 \pm 22.7$  (s.d.) to  $36.3 \pm 18.5$  and to  $6.3 \pm 17.7$  respectively. Cold stimulus, treatment with Colgate® Sensitive Pro-relief™ the mean VAS scores were not significantly different from the baseline value (baseline  $63.8 \pm 20.7$ ; after Colgate® Sensitive Pro-relief™  $58.6 \pm 22.3$ ). SE bond significantly decreased the mean VAS scores to  $40.0 \pm 31.6$  ( $P < 0.05$ , Tukey test). SEM micrographs after SE bond showed the resin layer covering the exposed dentine.

#### ➔ Conclusion:

**SE bond reduces the sensitivity to air blast and cold stimuli in hypersensitive dentine patients.**

Colgate® Sensitive Pro-relief™ could do only air blast but not cold stimulus. This abstract is based on research that was funded entirely or partially by an outside source: Thailand Research Fund

## CLEARFIL™ SE BOND

### IADR 1675 Dentin Bonding of SR Adhesive after Two Years Water Storage

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**Objectives:** Self-reinforcing (SR) adhesive has been introduced as a technology to contribute high bond efficiency with the multi-functional interaction to enamel and dentin. The purpose of this study was to evaluate the micro-tensile bond strength (mTBS) to dentin after two-years water storage and to characterize its interfacial interaction with dentin using TEM. **Methods:** Five adhesive groups: Bond Force (BF, self-etch 1step (SE1) with SR, Tokuyama), Bond Force no-SR (NSR, experimental SE1, Tokuyama), Optibond FL (OBFL, total-etch 3step Kerr), Clearfil SE (CSE, self-etch 2step, Kuraray) and Optibond All-in-One (OBA, SE1, Kerr) were tested. 150 Extracted human molars were randomly distributed into five adhesive groups. Dentine surfaces were prepared using CNC Specimen Former (University of Iowa). The adhesives were applied according to manufacturer's instructions. Resin-based composite (Z100, 3M-ESPE) 'crowns', approximately 5mm in height,

were incrementally formed and light-cured. Each restored tooth was sectioned into 2x2mm square sticks with diamond blades then into dumbbell-shaped specimens with the CNC (0.5mm<sup>2</sup> round cross-sectional area, 1mm gage-length, 0.6mm radius of curvature). The specimens were stored for two-years in artificial saliva containing sodium azide (changed monthly) at 37°C. Specimens were tensile-tested at a crosshead speed of 1mm/min in a calibrated testing machine (Z2.5, Zwick). The data was analyzed by two-way ANOVA with post-hoc Turkey's test (alpha=0.05). Additional specimens per each group were subjected to a silver impregnation protocol and nanoleakage patterns were observed under TEM. **Results:** The mTBS means and standard deviations are presented in the table below. Pre-test failures were included as 1MPa. Means followed by same letters (number for rows, character for columns) are not significantly different.

Material (n=30)	24hr	24mo
Bond Force	58.2(10.5) A,1	31.8(7.6) B,2
Bond Force no SR	38.3(17.5) B,1	18.8(11.6) C,2
Optibond FL	56.4(8.8) A,1	43.2(11.3) A,2
<b>Clearfil SE</b>	55.9(12.7) A,1	44.7(13.8) A,2
Optibond All-in-one	55.0(11.8) A,1	33.7(12.8) B,2

#### ➔ Conclusion:

The bonding effectiveness of SR technology applied to a one bottled adhesive system for dentin bonding was confirmed. Mr. Yamagawa was employed by Tokuyama Dental Corp. while a visiting scientist at the University of Iowa.

## CLEARFIL™ SE BOND

### IADR 2016 Clinical evaluation of chlorhexidine incorporation in two-step self-etching adhesives

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**Objectives:** This clinical study aims to evaluate the effect of incorporation of chlorhexidine in self-etch primers in restorations of non-cariou cervical lesions. **Methods:** This study was approved by Federal University of Ceará #95/10. Twenty-three patients with similarly sized non-cariou cervical lesions were selected to participate of this study. A total of 126 restorations were placed according to the groups: G1 - Clearfil SE (Kuraray Medical, Japan); G2 - Clearfil SE + Chlorhexidine; G3 - AdheSE Bond (Ivoclar Vivadent, Germany); G4 - AdheSE + Chlorhexidine. The incorporation of chlorhexidine on primers was done by directly addition of a 2% solution of chlorhexidine digluconate to prepare a mixture with 1.0% concentration of chlorhexidine. All lesions were restored using micro-hybrid composite resin Filtek Z250 (3M-ESPE, Germany). Evaluation of the restorations was performed at baseline and after six months of clinical service in terms of retention, marginal discoloration, marginal adaptation, postoperative sensitivity and secondary caries using the modified Ryge criteria.

**Results:** Twenty-two patients were available for recall after 6 months (95.65%) and a total of 124 restorations were evaluated. For G1, two restorations have failed in marginal adaptation but G1 and G4 presented a percentage of retention of 100%. For groups G2 and G3, 3 and 2 restorations were lost respectively (89.6% and 93.3%). One tooth presented sensibility in groups G2 and G4. The Yates's chi-squared test detected no significant differences in different groups between baseline and 6-month recall for retention, marginal discoloration, marginal adaptation, postoperative sensitivity or secondary caries ( $p > 0.005$ ).

#### ➔ Conclusion:

The incorporation of chlorhexidine on primer of Clearfil SE and AdheSE bonding systems did not influence the performance of non-cariou cervical lesion restorations in a short-period clinical evaluation.



## CLEARFIL™ SE BOND

### IADR 672 Effect of Pulp Chamber Cleaning on Adhesive Bond Strength

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**Objectives:** The aim of this study was to investigate the influence of cleaning methods to remove root canal sealer from pulp chamber dentin on the bond strength of a self-etching adhesive. **Methods:** Twenty bovine incisor crowns were cut to expose the pulp chamber. Endométhasone N (Septodont, Saint-Maur-Dês-Fossés, France) was placed in contact with the pulp chamber dentin, soon after its manipulation, for 10 minutes. Specimens were divided into four groups, according to cleaning method: G1, no root canal sealer (control); G2, 0.9% sodium chlorite (NaCl); G3, ethanol; G4, NaCl followed by drilling. After cleaning, the teeth were restored with a self-etching adhesive system Clearfil SE Bond (Kuraray, Kurashiki, Japan) and composite resin (Z250, 3M ESPE, St Paul, MN). Specimens were sectioned to produce rectangular beams and the dentin/resin interface was submitted to microtensile bond testing. Failure modes were observed and the bond strength (MPa) means were analyzed by ANOVA and the Tukey test for post hoc comparisons ( $\alpha = 0.05$ ).

**Results:** G1, G3, and G4 showed similar bond strength ( $p > .05$ ). A significant decrease in the bond strength was observed ( $p < .05$ ) in G2. In Groups 1, 3 and 4, the predominant failure mode was the mixed type. Adhesive failure was predominant in the NaCl group.

#### ➔ Conclusion:

Cleaning with ethanol or NaCl followed by drilling were able to reestablish bond strength to pulp chamber dentin. The use of NaCl was not effective in restoring the bond strength of a self-etching adhesive system.

## CLEARFIL™ SE BOND

### IADR 2312 Effect of Pulp Chamber Cleaning on Adhesive Bond Strength

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**Objectives:** The aim of this study was to examine the acid-base resistant zone (ABRZ) using different bonding systems on dentin. **Methods:** Two etch-&-rinse adhesives (One-Step Plus, Bisco and Clearfil Photo Bond, Kuraray) and two self-etching systems (Clearfil SE Bond, Kuraray and Filtek Silorane Adhesive, 3M ESPE) were applied to dentin disks according to the manufacturers' instructions or after the treatment with 10% NaOCl (ED-Gel, Kuraray), excepted for Filtek Silorane Adhesive. Restored teeth were submitted to an acid-base challenge and observed using SEM. **Results:** For both self-etching adhesive systems,

the ABRZ was observed adjacent to the hybrid layer. **For Clearfil SE Bond, the ABRZ was thicker and more evident.** The etch-&-rinse systems showed no ABRZ and erosive lesions. This demineralized dentin is composed of the completely demineralized dentin (top) and the partially demineralized dentin (bottom) containing hydroxyapatite. MDP monomer chemically reacted with HAP, which is stable against the acid-base attack. When the dentin surface is self-etched with SE Primer, rinsed with acetone/water and treated with 10% NaOCl the demineralized dentin collagen is removed. However, a thin layer beneath the interface of partially demineralized dentin remains because the stable MDP-HAP salt (ABRZ).

#### ➔ Conclusion:

The formation of ABRZ was material-dependent, and the morphology was different among the tested materials. The removal of the collagen changed the morphology of the bonding interface and the formation of ABRZ, especially for the etch-&-rinse adhesives.

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

### IADR 730 Effects of Evaporation on Water Kinetics of An Experimental Adhesive

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**Objectives:** To evaluate the influence of the solvent evaporation in the kinetics of water diffusion (water sorption- $W_{sp}$ , solubility- $W_{sl}$ , and net water uptake) of an experimental adhesive system compared to commercial products. **Methods:** Three commercial adhesives (Clearfil S<sup>3</sup> Bond (CS<sup>3</sup>)/Kuraray, Clearfil SE Bond – bond/control group (CSE)/Kuraray, Opti-bond Solo Plus (OS)/Kerr and one experimental adhesive L759 (EXL759)/3M/ESPE) were evaluated. Specimen disks (n=12) were obtained (5.0x1.0mm). Half of the specimens (n=6) was either evaporated for 30s (E) or not evaporated (N). The adhesives were photoactivated for 40s (450mW/cm<sup>2</sup>).

After desiccation, the specimens were weighed and then stored in distilled water for evaluation of the kinetics of water diffusion over a 7-day period (ISSO4049). The disks were incubated in either water or mineral oil. Net water uptake (%) was also calculated as the sum of  $W_{sp}$  and  $W_{sl}$ . Data were submitted to 2-way ANOVA /Tukey's test ( $\alpha=5\%$ ). **Results:** Statistical analysis revealed that only the factor 'adhesive' was significant ( $p<0.05$ ). Solvent evaporation had no influence in the  $W_{sp}$  and  $W_{sl}$  of the adhesives. **CSE (control) presented significantly lower net uptake (5.4%).**

Adhesive	Wsp Oil ( $\mu\text{g}/\text{mm}^3$ )	Wsl Oil ( $\mu\text{g}/\text{mm}^3$ )	Wsp Water ( $\mu\text{g}/\text{mm}^3$ )	Wsl Water ( $\mu\text{g}/\text{mm}^3$ )	Net Water Uptake ( $\mu\text{g}/\text{mm}^3$ )
CSE	7.6±2.1aB	1.1±3.4aA	57.1±5.8cA	-2.9±4.9cA	5.4±0.9a
CSE <sup>3</sup> N	9.6±4.3aB	17.8±7.7aB	117.3±8.8bA	66.4±7.7bA	18.4±1.6b
CSE <sup>3</sup> E	8.6±4.3aB	20.3±7.9 aB	135.3±56.9abA	78.7±35.2abA	21.4±9.2b
OSN	5.5±7.9aB	20.1±16.5 aB	133.0±18.9abA	82.4±6.9abA	21.5±2.5b
OSE	5.9±8.6aB	17.5±6.6aB	154.6±5.4aA	91.9±5.8aA	24.6±0.9b
EXL759N	5.3±6.8aB	18.4±11.9aB	113.3±10.3bA	71.9±13.9abA	18.5±2.2b
EXL759E	8.9±1.8aB	19.6±7.4aB	113.2±4.6bA	66.5±6.5bA	17.9±0.9b

Different letters (upper for rows, lower for columns): significant ( $p<0.05$ ).

### ➔ Conclusion:

Evaporation procedure has no influence in the kinetics of water diffusion of the experimental adhesive. Water sorption and solubility parameters of the experimental adhesive were similar to that of the majority of the commercial products. This abstract is based on research that was funded entirely or partially by an outside source: Decanato de Pesquisa e Pós Graduação, UnB; supported by 3M ESPE grant 5-58868

## CLEARFIL™ SE BOND · CLEARFIL AP-X

### PER/ADR 107 Microleakage of Self-adhesive Resins With or Without Bonding Agents

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**Objectives:** To compare the sealing ability of self adhesive resins in class V cavities and investigate whether provides advantageous using with bonding agent. **Methods:** Standardized class V cavities (3\*1.5\*1.5 mm) were prepared on buccal surfaces of 70 extracted human molars, with gingival margins 1.5 mm up the root.

Specimens were randomly divided into 7 groups of 10 each and restored with following materials; Group I: Fusio Liquid Dentin · Group II: Vertise Flow Group III: Fuji II LC · **Group IV: SE Bond/Clearfil APX** · Group V: Optibond/Fusio Liquid Dentin · Group VI: Optibond/Vertise Flow and Group VII: Optibond/Clearfil APX. Specimens were polished and thermocycled (x1000), and immersed in 0.5% basic fuchsine dye for 24h. Then they were multi sectioned buccolingually and dye penetration was scored at occlusal and gingival margins separately using 0–4 scale on stereomicroscope. Data were analyzed by Kruskal-Wallis and Mann-Whitney U tests ( $\alpha = 0.05$ ).

**Results:** Group IV showed better results than the other groups at gingival and occlusal margins ( $p < 0.05$ ). Group I, Group II and Group III showed no differences among each other at gingival margins ( $p > 0.05$ ). At occlusal margins Group I ( $p < 0.05$ ) and Group II ( $p < 0.05$ ) showed no difference between each other but better results than Group III. Using Optibond with self adhesive resins showed no difference on marginal microleakage.

#### ➔ Conclusion:

It is remarkable to notice Fusio Liquid Dentin and Vertise Flow showed lower leakage results at occlusal margins than Fuji II LC but **sealing ability didn't meet Clearfil APX used with SE Bond** and Optibond. It can be stated that self adhesive resins are not sufficient as composite materials used with bonding systems yet.

## CLEARFIL™ PROTECT BOND<sup>2</sup>

### AADR 785 Bond Strength to Enamel Using 5th and 6th Generation Adhesives

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**Objectives:** To determine the shear bond strengths of different dental adhesives on enamel. **Methods:** Human molars and pre-molars were ground to 60 grit creating a flat surface on the enamel and polished to 320 grit. The five adhesives utilized were Moxie TE/5th generation and Moxie SE/6th generation (Discus Dental); OptiBond Solo Plus/5th generation and Optibond XTR /6th generation (Kerr-Sybron); and Clearfil SE Protect Bond/6th generation (Kuraray). Composite resin (SDI Glacier) was bonded and cured to the enamel according to manufacturer's instructions. Specimens (n=10; cylinder mold, d=2.3) were stored in 100% humidity at 37°C for 24 hours. An Instron was utilized to load specimens and calculate the shear bond strength in megapascals (MPa). Analysis was performed using ANOVA and post hoc Tukey test at 0.05 level of significance.

**Results:** The mean bond strengths, MPa, and standard deviations of the five adhesives were: Moxie TE 12.0 (5.0), Moxie SE 9.3 (4.1), OptiBond Solo Plus 11.2 (1.8), Optibond XTR 11.7 (3.6), and Clearfil SE Protect Bond 15.0 (4.9). No adhesives were significantly different from each other except between Clearfil SE Protect Bond and Moxie SE ( $p=0.02$ ).

#### ➔ Conclusion:

Enamel bond strengths were similar among Moxie TE, Moxie SE, OptiBond Solo Plus and Optibond XTR,  $p>0.05$ , while Clearfil SE Protect Bond was significantly greater than Moxie SE. Acknowledgements: Materials were provided by manufacturers.

## CLEARFIL™ PROTECT BOND

### IADR 2967 Relationship between f-release and morphology of acid-base resistant zones

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**Objectives:** The aim of this study was to investigate the relationship between f-release from adhesives and morphology of acid-base resistant zones (ABRZ) at the interface. **Methods:** Seven experimental adhesives with different concentrations of NaF; 0%, 10%, 20%, 50%, 75%, 90% and 100% (F0~F100) were prepared. Concentration of NaF was originally based on that of Clearfil Protect Bond (PB, Kuraray Medical, Japan). These adhesives were inserted in a silicon mold and light cured to make adhesive disks (n=4). Each specimen was immersed in 1 mL of deionized water in a polyethylene vial for 24 hours. Fluoride ions released from the adhesive were measured using a combination fluoride ion-selective electrode connected to an ion analyzer. Obtained data were statistically analyzed with regression analysis. Meanwhile, dentin disk sandwiches were prepared from human molars with one of the above adhesives and a resin composite (Majesty

LV, Kuraray Medical, Japan), sectioned and embedded in epoxy resin. The specimens were subjected to a demineralizing solution adjusted to pH 4.5 for 90 min and 5% NaOCl for 20 min. The specimens were then sectioned, polished, and argon-ion etched. The ABRZ formation was observed by SEM. The area of the ABRZ was calculated using ROI mode of the device. The data were statistically analyzed using one-way ANOVA and multiple comparisons were made using Dunnett's C test at p<0.05 level. **Results:** The amounts of fluoride ion release from the adhesives increased with the increase of fluoride concentration (R2=0.9923). The area of the ABRZ increased with higher concentration of NaF in the adhesive except for F0, F10, and F20 (p<0.05). **The largest ABRZ was obtained in F100 (original PB)**, in which slope formation was clearly observed at the bottom of the ABRZ.

#### ➡ Conclusion:

Concentration of NaF in the adhesive influenced the area of ABRZ in the two-step fluoride-releasing adhesive system.

## CLEARFIL™ PROTECT BOND · CLEARFIL™ SE BOND

### AA DR 1198 Bond durability of four contemporary self-etching systems

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**Objectives:** To evaluate the bonding performance of four commercial available self-etching systems stored in water for 24 hours, 3 months, 6 months and 12 months. **Methods:** Four self-etching adhesives were employed in this study: Easy Bond (EB, 3M ESPE, USA), G-Bond plus (GBp, GC Corporation, Japan), SE Bond (SE, Kuraray Medical INC. Japan) and Protect Bond (PB, Kuraray Medical INC. Japan). 32 human third molars without caries were separated into 4 groups with 8 teeth in each adhesive system. After bonding followed by the manufacture's instruction, the teeth were build-up with resin composite (Clearfil AP-X) and being stored in 37 °C distilled water for 24 hours. The bonded specimens sectioned into 1.0 mm<sup>2</sup> sticks and storage into 37 °C distilled water again for 24h, 3m, 6m, and 12m. Micro-tensile bond strength (μTBS) test was performed, after different water-storage duration at a crosshead speed of 1mm/min. The obtained data were expressed as MPa and statistically analyzed by Games-Howell test and regression analysis (p<0.05).

**Results:** Regarding all-in-one adhesives, EB showed significant decrease in μTBS at all the storage durations. GBp indicated significant decrease after 12m storage in comparison with 24h. **On the other hand, two 2-step self-etching systems expressed no decrease in μTBS during 12m water-storage.**

#### ➔ Conclusion:

Water could affect bonding performance of all-in-one system significantly, especially on HEMA-contained all-in-one system. 2-step self-etching systems might have consistent bonding performance comparing with that of all-in-one systems.

## CLEARFIL™ PROTECT BOND<sup>2</sup> · CLEARFIL™ SE BOND

### IADR 2838 Antibacterial evaluation of Clearfil SE Protect against oral bacteria

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**Objectives:** To evaluate the antimicrobial efficacy of Clearfil SE Protect against five individual oral microorganisms as well as in a mixed bacterial culture and compare the results of the study with Clearfil SE Bond (CB). **Methods:** Bacterial culture of single species of Streptococcus sobrinus, Streptococcus gordonii, Actinomyces viscosus and Lactobacillus lactis, as well as mixed bacterial culture created were obtained from ATCC. Dentin bonding discs (6 mm diameter) were prepared, cured and placed on the bacterial culture of single species or multispecies oral bacteria for 15, 30 and 60 minutes. The antimicrobial property of the two dentin bonding agents was determined by MTT and Live/dead bacterial viability assays, and also by measuring their metabolic activity by XTT assay. All assays were done in triplicates and each experiment repeated at least three times. Significance of results was determined by ANOVA and Scheffe's f-test.

**Results:** CB had no significant ( $p < 0.05$ ) effect on the viability or metabolic activity of the test microorganisms when compared to the saline control. The CP was found to be significantly effective against the organisms tested both on single strain and on mixed strain bacterial culture. **Clearfil SE Protect exhibited antimicrobial effect on single strain of bacteria.** Greater than 50% killing was seen within 15 minutes, and the killing progressed with increasing time of incubation. However, longer (60 min) period of incubation was required by CP to achieve similar antimicrobial effective against mixed bacterial culture tested

#### ➔ Conclusion:

The results demonstrated the antimicrobial efficacy of CP both on single and mixed bacterial culture. The use of CP may be beneficial in reducing incidence of bacterial infections in the cavity preparation.

This abstract is based on research that was funded entirely or partially by an outside source: Fapesp # 2010/50414-8



## NOTES

# COMPOSITE RESINS

CLEARFIL™ AP-X

CLEARFIL MAJESTY™ Posterior

## CLEARFIL™ AP-X

### IADR 27 Six-year Clinical Evaluation of Two Types of Resin Composite Restorations

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**Objectives:** This randomized controlled clinical trial evaluated 6-year clinical performance of two types of resin composite restored with a one-step self-etch system.

**Methods:** Ninety-eight non-carious cervical lesions in 22 patients (11 men and 11 women) with a mean age of 61.9 years (range 29-78) were involved for the study. No consideration was given to the recruitment. Enamel bevel was placed and dentin walls were lightly ground, and restored with S3 Bond (Kuraray Medical) in conjunction with a hybrid (AP-X: AP, Kuraray) or a flowable resin composite (Flow FX: FX, Kuraray). Each patient received both restorative groups randomly. All restorations (48 restorations for AP and 50 restorations for FX) were placed by one dentist between May and November in 2005. The restorations were blindly evaluated every year using modified USPHS criteria by two examiners. The data were statistically analyzed using the Fisher's exact test, Kaplan-Meier method and Cox proportional hazards model.

**Results:** Twenty patients and 90 restorations were evaluated at the 6-year recall. **Retention rates for AP and FX were 100% and 91%.** Secondary caries was detected around three restorations restored with FX. There was a significant difference in 6-year survival rates between AP and FX. Marginal staining increased with time, and 6-year staining rates for AP and FX were 35% and 56%, respectively. Tooth type and location had a significant effect on marginal staining. There were no significant differences in the clinical performances between AP and FX for other variables, such as marginal adaptation, fracture, sensitivity, color change and wear.

#### ➔ Conclusion:

The findings of this study suggested that the flowable resin demonstrated poorer clinical performance in non-carious cervical lesions compared to the hybrid resin composite after 6 years clinical service. This study was supported by KAKENHI (20592230 and 23592802). This abstract is based on research that was funded entirely or partially by an outside source: KAKENHI (20592230 and 23592802)

## CLEARFIL™ AP-X

### AADR 474 Discoloration of Resin Composites After Immersion in Different Oriental Foodstuffs

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**Objectives:** The discoloration of restoratives is esthetic problem in clinical situation. The purpose of this study was to examine the discoloration of resin composites after immersion in different oriental foodstuffs. **Methods:** Three types of resin composite restorative materials; Filtek Supreme Plus Restorative (SUP: nano-hybrid type, 3M ESPE), Clearfil AP-X (APX: semi-hybrid type, Kuraray medical) and Solare (SOL: MFR type, GC) were used. Composite was filled in a silicone mold (diameter of 20.0mm, thickness of 3.0mm) and irradiated with a halogen curing light source (Optilux501; Demetron). Cured specimens were polished with a series of SiC paper up to 2000 grit. The baseline measurement of the CIE-L\*a\*b\* color was obtained using a colorimeter (CR-400, Konica minolta). 48 specimens were divided into four groups and were immersed in foodstuffs at 37 °C, distilled water (W: Taiyo Med), soy sauce (S: Kikkoman), perilla drink (P: Kisyu Kumano) and black vinegar (V: Kewpie). After 2 weeks of immersion, the specimens were rinsed with distilled water and then measured using colorimeter. The color differences (delta E\*ab) were calculated and analyzed using two-way ANOVA and q-test (n=4).

**Results:** Color changes (delta E\*ab) were SUP-W:  $0.34 \pm 0.32$ , SUP-S:  $0.57 \pm 0.33$ , SUP-P:  $3.12 \pm 1.53$ , SUP-V:  $0.92 \pm 0.18$ , APX-W:  $0.41 \pm 0.18$ , APX-S:  $0.98 \pm 0.29$ , APX-P:  $0.51 \pm 0.36$ , APX-V:  $0.54 \pm 0.30$ , SOL-W:  $0.72 \pm 0.64$ , SOL-S:  $0.81 \pm 0.58$ , SOL-P:  $0.51 \pm 0.30$ , SOL-V:  $0.64 \pm 0.19$  (mean  $\pm$  S.D.). Each of the factors (foodstuff, composite) was a statistically significant factor ( $p < 0.01$ ) and also there was an interaction with another factor ( $p < 0.01$ ). Color change after immersion in P was higher than that in W ( $p < 0.01$ ) and V ( $p < 0.05$ ). And also SUP showed the highest color change than that of APX and SOL ( $p < 0.05$ ).

#### ➔ Conclusion:

Color of immersed resin composite was influenced by the types of foodstuff and resin composite in this study. It seemed that the filler contents of composite and the polyphenol of foodstuff influenced discoloration.

This abstract is based on research that was funded entirely or partially by an outside source: Grant-in-Aid for Scientific Research (C), (No. 22592131), MEXT of Japan

## CLEARFIL MAJESTY™ Posterior · CLEARFIL™ AP-X

### IADR 2314 SS-OCT Real-time monitoring of resin composite placement by layering technique

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**Objectives:** The aim of this study is to evaluate in real time the layering technique with different composites using SS-OCT. **Methods:** Dentin discs (Ø: 6 mm; n=6 x 3) with cavities (Ø: 3 mm; h: 1,5 mm) were prepared. All prepared discs were randomly divided into three groups, each differing by the composite composition (Clearfil AP-X/APX; Clearfil Majesty Posterior/MP; Beautifill Flow F00/BF). All groups were subdivided by half according to the filling technique (layering and bulk technique). Self-etch adhesive system (Bond-Force, Tokuyama) was applied and photopolymerized for 20s and fill it with composite using three increments and polymerizing each for 40s. Each specimen was image using an SS-OCT unit and recorded during 120 seconds. Still images were taken at 10s, 20s, 30s, 40s, 60s, 80s, 100s and 120s. Real-time gap formation of the interlayer interface between layering technique groups was observed. Real-time Gap formation at the cavity floor between all groups was observed. The limits between composites and dentin were identifying. All the obtained images were processed with imaging analysis software (ImageJ) to quantify interfacial gap resulting from polymerization shrinkage, dimensional contraction and structural voids. Confocal laser scanning microscope (CLSM) analysis at 20x and 100x magnification was made to confirm SS-OCT findings. Data were statistically analyzed by 2-way ANOVA to determine correlations between time and percentage interfacial gap formation.

**Results:** Image analysis obtained from SS-OCT revealed significant differences of interfacial gaps at the interlayer interfaces between groups and composite cavity floor (P<0.05). The optical contrast between the material and air helped in detection of interfacial gaps. CLSM analysis confirmed that SS-OCT was capable of detecting interfacial gaps and structural voids on submicron-scale.

#### ➔ Conclusion:

SS-OCT can be used to provide non-destructive real-time assessment between restoratives materials for gap detection at the layer interfaces and composite cavity floor of resin composites restorations.

# RESIN CEMENTS

PANAVIA™ SA CEMENT

PANAVIA™ F2.0

CLEARFIL™ ESTHETIC CEMENT

## PANAVIA™ SA CEMENT<sup>3</sup>

### AAADR 633 Tensile Bond Strength of Different Luting Agents at the Cement-Restoration-Interface

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**Objectives:** The aim of this study was to evaluate the tensile bond strength at the cement-restoration interface of different luting agents bonded to different restoration materials by using a newly developed testing procedure. **Methods:** Disc-shaped specimens made from Y-TZP zirconia, glass ceramic and non-precious metal were used as restoration materials. A cylinder with smaller diameter was cemented onto each disc-shaped specimen. The five luting agents (Clearfil SA Cement CSA and Panavia F2.0 PF, Kuraray, Japan; RelyX Unicem RX, 3M ESPE, Germany; Maxcem Elite ME, KerrHawe, Switzerland, iCem iC, Heraeus Kulzer, Germany) were used according to the manufacturers' instructions. The cemented specimens (n=6 for each restoration-cement-combination) were stored for 23 days in distilled water (37°C). A universal testing machine (Z010, software: TestXpertII V3.0, Zwick/Roell, Germany) was used for testing and results were statistically analyzed (ANOVA,  $\alpha=0.05$ , univariate, bifactorial, SPSS 19.0).

**Results:** Debonding of samples during storage was noticed (CSA:0, PF:1, RX:2, ME:7, iC:3). In these cases, tensile bond strength was considered to be 0 MPa. Regarding the restoration material, no significant influence on tensile bond strength could be detected. **For ME and iC, tensile bond strength was significantly lower than for RX and PF. For RX and PF, tensile bond strength was significantly lower than for CSA.** There was no significant difference between ME and iC nor between RX and PF. **CSA showed the highest values for tensile bond strength.**

#### ➔➔ Conclusion:

The newly developed procedure for tensile bond strength testing offers the major benefit of a real tensile test without a component of shear bond strength. The testing procedure is specially designed for luting agents, as the functionality of anorganic cements is based on friction, which is not provided by this specimen-geometry. For such cements, a newly developed procedure for shear bond strength testing could be applied.

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## PANAVIA™ SA CEMENT<sup>3</sup>

### AADR 1011 Effect of Acid Etching on Lithium Disilicate Glass Ceramic

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**Objectives:** The purpose of this study was to evaluate the effect of two different types of acid etching on the bond strength of lithium disilicate glass ceramic (IPS e.max Press, Ivoclar) cemented with self-adhesive resin cement. **Methods:** IPS e.max Press specimens polished by 600-grit SiC paper were etched with 5% hydrofluoric acid (HF) or 36% phosphoric acid (PO), or air-abraded with 50 µm alumina. Each pretreated surface was cemented to a stainless steel rod of 3.6 mm diameter and 2.0 mm height with self-adhesive resin cement (Clearfil SA Cement, Kuraray) using a Teflon mold followed by the application of the ceramic primer (control+pri) or not (control-pri). Shear bond strength between ceramic and cement were measured after 24 h storage in 37°C distilled water. The microstructure morphology of the surface after pretreatment was evaluated using SEM analysis. Statistical analyses were performed by one-way ANOVA and Holm-Sidak test.

**Results:** The bond strength of HF followed by AA (HF+AA, 28.1±6.0 MPa), PO followed by AA (PO+AA, 17.5±4.1 MPa), and HF (21.0±3.0 MPa) were significantly greater than that of control+pri (9.7±3.7MPa) or control- pri (4.1±2.4 MPa) ( $p<0.05$ ). In addition, with HF or PO (14.0±2.5 MPa) the shear bond strength significantly increased over AA (13.2±3.7 MPa) ( $p<0.05$ ). HF+AA, PO+AA and HF exhibited cohesive failure, while the others mainly exhibited adhesive failure. SEM images of AA showed formation of conventional microretentive grooves, but acid etching with HF or PO produced a porous surface.

#### ➔ Conclusion:

Hydrofluoric acid or phosphoric acid etching yields higher bond strengths between lithium disilicate ceramic and a self-adhesive resin cement without creating microcracks by air abrasion.



## PANAVIA™ SA CEMENT<sup>3</sup>

### IADR 2965 Evaluation of Resin-Cements Marginal Adaptation to Class-V Cavity Using SS-OCT

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**Objectives:** The aim of this study is to investigate the use of swept-source optical coherence tomography (SS-OCT) to evaluate marginal adaptation of resin cements with different curing modes for indirect restorations in class V cavities. **Materials and Methods:** Round-shaped class V cavities (3 mm diameter x 1.5 mm depth) were prepared at the dentin buccal cervical surface of bovine teeth. The cavities were restored with pre-fabricated resin composite inlays (Clearfil Majesty Posterior) and cemented with self-adhesive resin cements (G-CEM, RelyX Unicem Clicker and Clearfil SA Cement) or a self-etching resin cement (Panavia F2.0) and polymerized by self-cure or light-cure mode. After 24 hs storage in water, 2D scans of the restorations were obtained using SS-OCT. Ten cross-sectional 2D images of each material were analyzed to evaluate marginal adaptation. A digital image analysis software was used to detect high brightness areas at the interfacial zone (representing gap) and their percentage distribution was calculated by the area of cavity interface. The results were statistically compared with Mann-Whitney and Kruskal-Wallis test with statistical significance defined as  $p < 0.05$ .

**Results:** Generally, less bright areas at the interfacial zone were observed when the light-cure mode was used, indicating a better marginal adaptation. There was a significant statistical difference between self-curing and dual-curing mode within for Panavia F2.0 or RelyX Unicem Clicker. The presence of gap was confirmed by confocal laser microscope with gaps ranging from 1  $\mu\text{m}$  to 70  $\mu\text{m}$ . There was no difference among light-cured cements, however in the self-cure mode, Clearfil SA Cement showed lower gap than RelyX Unicem Clicker ( $p < 0.05$ ). SS-OCT images can be observed below.

#### ➡ Conclusion:

SS-OCT can be considered as a non-invasive technique for fast detection of gaps of resin cements interfacial zone.

## PANAVIA™ SA CEMENT<sup>3</sup>

### IADR 1992 Bond Strength of Resin Cements to Zirconia Ceramic Using Primers

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**Objectives:** To evaluate the influence of primers on the microshear bond strength of resin cements to zirconia ceramic. **Methods:** Fifty zirconia plates (12 X 5 X 1.5 mm high) of a commercial zirconium-oxide ceramic (ZirCad, Ivoclar Vivadent) were sintered, sandblasted with aluminum oxide particles and placed into ultrasonic cleaning machine before bonding. Plates were randomly divided into 5 groups (n=10): G1: control group (RelyX ARC, 3M ESPE / no primer), G2: Multilink, Ivoclar Vivadent (no primer), G3: Zirconia Metal Primer, Ivoclar Vivadent + Multilink, G4: Clearfil SA Cement, Kuraray (no primer) and G5: Alloy Primer, Kuraray + Clearfil SA Cement. The primers and resin cements were used according to manufacturers' recommendations. Resin cement cylinders were constructed on zirconia surfaces by using matrix (Tygon) with 0.7 x 1 mm (diameter x height). The samples were stored in distilled water for 24 hours at 37 °C and tested in a testing machine (0.5 mm/min, EZ-test, Shimadzu). Bond strength data were analyzed by two-way ANOVA and Dunnett's test (5%).

**Results:**The bond strength means (SD) were (in MPa): G1: 28.1(6.6), G2: 37.6(4.5), G3: 55.7(4.0), G4: 46.2(3.3) and G5: 47.0(4.1).

#### ➔ Conclusion:

The Zirconia Metal Primer increased the bond strength of Multilink resin cements to zirconia. RelyX ARC showed the lowest bond strength to zirconia.

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## PANAVIA™ SA CEMENT<sup>3</sup>

### PER/IADR 333 Adhesion of Resin-cements to Dentin After Cleaning and Repeated Cementation

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**Objectives:** The aim of this study was to evaluate the effect of different dentin cleansing regimens prior to recementation and their influence on the micro-shear bond strength ( $\mu$ SBS) of resin cement to dentin. **Methods:** The labial surfaces of mandibular incisors (N=200) were ground to expose the coronal dentin. The teeth were randomly divided into two subgroups (n=100) to receive the resin cements: Clearfil SA (S) and Panavia F 2.0 (P) resin cements were condensed into polyethylene mold and polymerized. Specimens were stored in humid condition for 24h and tested to failure using  $\mu$ SBS (MPa) testing (0.05 mm/min). Four different dentin cleansing regimens were used: a) composite finishing bur (cb), b) tungsten carbide bur (ob), c) ultrasonic scaler tip (sc) and d) pumice-water slurry (pw) to remove cement remnants after debonding (n=20). Control group (cn) received no cleansing regimen. Recementation procedures were achieved with the same resin cements on the cleaned dentin surfaces and  $\mu$ SBS test was repeated. Failed surfaces were observed under Scanning Electron Microscope (SEM). Data were analyzed using Mann-Whitney U, Wilcoxon Signed Ranks and Bonferroni tests ( $\alpha=0.05$ ).

**Results:**  $\mu$ SBS (MPa) of S ( $6.42\pm 2.96$ ) and P ( $7.88\pm 3.49$ ) cements in Group a showed statistically significant differences ( $p<0.05$ ). The  $\mu$ SBS values of Group b were as follows: Scb: $3.42\pm 1.47$ ; Ssc: $4.25\pm 2.74$ ; Sob: $4.96\pm 3.28$ ; Spw: $8.42\pm 5.06$ ; Scn: $5.83\pm 2.87$ ; Pcb: $4.82\pm 2.54$ ; Psc: $4.54\pm 2.58$ ; Pob: $4.29\pm 2.17$ ; Ppw: $5.8\pm 2.5$ ; Pcn: $9.84\pm 4.88$ .

#### ➔ Conclusion:

On uncontaminated dentin Panavia F 2.0 performed better than Clearfil SA in terms of bond strength. While pumice-water cleansing was more effective for Clearfil SA, for Panavia cleansing method did not play a significant role but also cleansing was less effective compared to control group for this cement.

## PANAVIA™ F2.0

### PER/ADR 395 Influence of Various Canal-Sealers on Push-Out-Bond Strength of a Glass-fiber-post

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**Objectives:** The aim of this in vitro study was to evaluate three different endodontic sealers; iRoot SP (Veriodent-Canada), Sealapex (Kerr-Italy) and MetaSEAL (Sun Medical-Japan) on push-out-bond strength of a glass fiber post (Polydentia GF Posts-Switzerland) cemented with Panavia F 2.0 (Kuraray Medical-Japan) resin cement. **Methods:** Forty straight, single-rooted premolars and incisors were used in this experiment. The teeth were decoronated at the cemento-enamel-junction under water cooling. All root-canals were then instrumented with ProTaper-files up to size F5 with irrigating 2 ml of 0.5% NaOCl between each files and 2 ml of 17 % EDTA as a final irrigant. The root specimens were randomly divided into four groups (n=10) and filled as follows: group 1: control group (gutta-percha points only, no sealer); group 2: calcium silicate-based sealer (iRoot SP); group 3: resin-based sealer (MetaSEAL) and group 4: calcium hydroxide-based sealer (Sealapex) with gutta-percha. Glass fiber posts were cemented into the prepared post spaces with adhesive resin cement-Panavia F 2.0. The root specimens were sectioned perpendicular to the long axis with a water-cooled diamond disc (Buehler-USA). Four slices, having 0.6-mm thickness, were obtained from the coronal part of each root. The test specimens were then subjected to the push-out test method using a Universal Test Machine and failure modes were observed. Data were statistically analyzed (One-way ANOVA and post-hoc Tukey tests).

**Results:** No significant differences were observed between the test groups ( $P>0.05$ ). iRoot SP, MetaSEAL and Sealapex sealers demonstrated similar push-out-bond strength values. Although MetaSEAL group showed slightly better bond strength values than other sealers, it was not statistically significant.

#### ➔ Conclusion:

Within the limitations of this study it can be concluded that using iRoot SP, MetaSEAL and Sealapex sealers in the obturation of teeth that needs posts does not affect push-out bond strength of glass fiber posts cemented with Panavia F cement.

## PANAVIA™ F2.0

### PER/IADR 581 Effect of resin cement on bond strength of adhesively-veneered 3Y-TZP

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*Adhesive joining veneers to cores provides an alternative veneering method for 3Y-TZP.*

**Objectives:** To investigate the effect of resin cement selection on microtensile bond strength ( $\mu$ TBS) of adhesively-veneered 3Y-TZP. **Methods:** Zirconia discs were fabricated from commercial 3Y-TZP powders (Tosoh), 50 $\mu$ m Al<sub>2</sub>O<sub>3</sub>(Renfert) air-abraded (0.25MPa, 15s), cleaned and pre-treated (Metal/Zirconia primer, Ivoclar). Veneer discs were sectioned from feldspathic block (Vitablocs Mark II, Vita Zahnfabrik), etched (IPS ceramic etching gel, Ivoclar) and silanated (Monobond-S, Ivoclar). Pre-treated surfaces of zirconia and veneer discs were bonded with one of three resin cements: Multilink N (MN, Ivoclar); RelyX Unicem (RU, 3M/ESPE); Panavia F (PA, Kuraray), light-cured and cut into micro-bars (5.10mm×1mm×1mm) respectively. Microbars (n=15 per group) were randomly chosen, stored in

distilled water (37°C, 24h) and tested by universal testing machine (EZ-L, Shimadzu; cross-head speed=0.5mm/min). Load values at failure were determined and analyzed by One-way ANOVA and Tukey's test ( $\alpha=0.05$ ). Fracture zirconia surfaces were examined using stereomicroscope (KH-1000, Hirox) and scanning electron microscope (SSX-550, Shimadzu) to determine the failure modes. **Results:** Mean and standard deviation of  $\mu$ TBS and percentage of failure distribution of three groups were listed in table.  $\mu$ TBS was significantly influenced by resin cements ( $p<0.001$ ) and the ranking order was PA, UN and MN. All failures occurred at the zirconia/resin cement interface partly (mixed failure) or completely (adhesive failure) without zirconia fracture.

Group	$\mu$ TBS (MPa)	Adhesive failure (%)	Mixed failure (%)
PA	37.94±4.54 <sup>a</sup>	40	60
UN	25.70±4.92 <sup>b</sup>	93	7
MN	20.85±4.81 <sup>c</sup>	60	40

#### ➔ Conclusion:

Adhesively-veneering method can prevent zirconia core from being damaged under tensile forces, regardless of the resin cement used. **Resin cement with 10-MDP monomer high modulus and good wettability like Panavia F, could be recommended to bond feldspathic veneers onto 3Y-TZP cores.**

## CLEARFIL™ ESTHETIC CEMENT

### AADR 640 Efficacy of silane after thermal-fatigue on bond-strengths degradation to zirconia

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**Objectives:** To evaluate the influence of artificial aging by thermocycling on deterioration of microtensile bond-strength ( $\mu$ TBS) to zirconia. **Methods:** Pre-sintered zirconia blocks (e.max ZirCAD, Ivoclar) were cut (14.5x15.5x7 mm) and fully sintered according to manufacturer's instruction. After air-abrasion with Al<sub>2</sub>O<sub>3</sub> particles, three different silanes were tested: 1. Clearfil Ceramic Primer (CLE; Kuraray); 2. Mono-bond Plus (MB; Ivoclar); 3. Z Prime Plus (ZP; Bisco), and control group (CO) without silane application. A dual-cure resin cement (Clearfil Esthetic, Kuraray) was standardized for all experimental groups. Each group was assigned into three subgroups subjected to different thermal fatigue as: 5,000 thermal cycles (5K), 20,000 thermal cycles (20K), or no thermal

cycles (NA) of artificial aging. Thermocycling was performed at 5°C and 55°C (SD-Mechatronik). All specimens were sectioned with a precision saw and 0.64mm<sup>2</sup> sticks obtained. The sticks were tested for  $\mu$ TBS, using a universal testing machine (Instron) at a crosshead speed of 0.5 mm/min. The data were submitted to Kruskal-Wallis and Mann-Whitney Tests ( $\alpha=0.05$ ). **Results:** (Pre-testing failure/number of specimens) Mean $\pm$ SD in MPa. Means with same lower-case letters, in the same row, are not statistically different. Means with same upper-case letters, in same column, are not statistically different.

	Artificial aging		
	No aging	5,000 cycles	20,000 cycles
<b>CLE</b>	(0/68) 52.47 $\pm$ 11.1 <sup>aA</sup>	(0/66) 24.54 $\pm$ 5.3 <sup>bA</sup>	(0/71) 24.19 $\pm$ 4.9 <sup>bA</sup>
<b>MB</b>	(0/68) 43.94 $\pm$ 13.9 <sup>aB</sup>	(0/70) 26.43 $\pm$ 6.4 <sup>bA</sup>	(0/71) 22.82 $\pm$ 6.3 <sup>cA</sup>
<b>ZP</b>	(0/72) 20.13 $\pm$ 7.1 <sup>aC</sup>	(12/69) 4.88 $\pm$ 3.6 <sup>bB</sup>	(52/71) 1.00 $\pm$ 2.0 <sup>cB</sup>
<b>CO</b>	(70/70) 0.00 $\pm$ 0.0 <sup>aD</sup>	(69/69) 0.00 $\pm$ 0.0 <sup>aC</sup>	(67/67) 0.00 $\pm$ 0.0 <sup>aB</sup>

#### ➔ Conclusion:

Thermocycling significantly reduces  $\mu$ TBS to zirconia. **CLE** and **MB** were able to withstand intense thermal fatigue, without pre-testing failure. Silane is essential for stable long-term bond-strength to zirconia.

## CLEARFIL™ ESTHETIC CEMENT

### AADR 219 Resin Cements Bonded on Different Treated Non-precious and Semi-precious Metal-alloys

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*With self-etch resin cements becoming more popular, some dentists may be tempted to use them to bond Maryland bridge.*

**Objectives:** To compare the shear bond strength of a self-etch versus a total-etch resin cements when bonded to enamel and metal-alloy treated surfaces.

**Methods:** Flat enamel (E) surfaces (n=15/group), non-precious metal-alloy (Np) disks (n=15/group), semi-precious metal-alloy (Sp) disks (n=15/group) were mounted in cold-cure acrylic rings. Cylinders (d=2.38mm x h=3.0mm) of Clearfil Esthetic Cement & DC Bond Kit (C) and RelyX Unicem (R) resin cements were bonded to enamel and to metal-alloy prepared surfaces (sandblast (SB), SB + sulfuric

acid (SA) or SB + tin deposition (T)), polymerized, stored (H<sub>2</sub>O, 37°C, 24hours) and thermocycled (500 cycles). Samples were tested individually in an Instron Universal Testing machine (1mm/min). Statistical analysis was done using a T-test comparison followed by a post hoc multiple comparisons (p£0.05). **Results:** For each group, values with same superscript are not significantly different. Results on enamel showed that the total-etch resin cement (C) had a statistically significant higher bond strength (33.97±17.18 MPa) than the self-etch resin cement (R) (10.48±11.23 MPa) (p£0.05). In the Sp group, a thin layer of tin added on the sandblasted metal surface reduced the bond strength significantly with the C resin cement (p£0.05).

Bond strength MPa	Enamel	Non-precious metal-alloy		Semi-precious metal-alloy	
	(Mean ± sd)	(Mean ± sd)		(Mean ± sd)	
Resin Cements		SB	SB+SA	SB	SB+T
<b>Clearfil Esthetic Cement</b>	33.97 ± 17.18 a	25.21 ± 14.66 a, b	34.11 ± 15.57 a	34.60 ± 14.86 a	18.43 ± 10.83 a
RelyX Unicem	10.48 ± 11.23 b	15.96 ± 3.37 b	18.90 ± 9.55 b	11.41 ± 10.53 a	10.08 ± 7.68 a

#### ➔ Conclusion:

The total-etch resin cement (C) showed a statistically significant higher bond strength than the self-etch resin cement (R) on enamel and on three (Np-SB+SA; Sp-SB; Sp-SB+T) of the four metal-alloy surface groups.

# CERAMICS



## Cerabien™ ZR

### AADR 202 4-8 years Retrospective Dental Laboratory Survey of Zirconia-Based Restorations

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**Objectives:** To survey frequency of remakes of zirconia-based restorations in a dental laboratory at 4-8 years after delivery. **Methods:** Data was collected on crowns and fixed partial dentures (FPDs) in which zirconia copings/frameworks were milled (Lava™, 3M ESPE) and layered with veneering porcelain all in one dental laboratory. Cantilever FPDs, splinted crowns, implant-supported restorations, and restorations delivered less than 4 years prior to survey were excluded. Data included tooth numbers, time from delivery, brand of veneering porcelain, and requests for remakes. Probability distributions of remakes were reported. Chi-square test was used for inter-group comparisons ( $P < 0.05$ ). **Results:** 24392 zirconia-based crowns (21220 (87%) posterior and 3172 (13%) anterior) were delivered. 605 were layered with Lava™ Ceram and 23787 were layered with Noritake CZR. **Requests for remake were made for 185 (0.75%) of 24392**

**zirconia-based crowns.** 72 out of 185 crowns were sent for remake prior to cementation (posterior 67 (0.2%) and anterior 5 (0.02%)). Other remakes of crowns were requested after cementation within a timeframe ranging from 1 to 33 months of service (posterior 97 (0.3%) and anterior 16 (0.06%)). Total number of remakes was 164 (0.67%) for posterior and 21 (0.08%) for anterior crowns ( $p = 0.57$ ). Remakes were 44 (0.18%) for premolars and 120 (0.49%) for molars ( $p = 0.05$ ). Comparisons were statistically not significant. 1482 zirconia-based FPDs with 580 posterior, 578 anterior, and 324 mixed with both posterior and anterior abutments ( $n = 324$ ) were also surveyed. There were only 4 (0.2%) remakes. 2 posterior restorations were re-sent for remake before cementation. 2 remakes of anterior restorations were requested within a time frame of 3 and 26 months after cementation.

#### ➔ Conclusion:

Within limitations of this retrospective survey it may be concluded that frequency of remakes of zirconia-based restorations after 4-8 years of service made by one dental laboratory was low for crowns and FPDs.

### IADR 189 Low Temperature Degradation of Three Zirconia's

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**Objectives:** Assess the low temperature degradation of 2 commercially available zirconia materials and a newly developed zirconia (NZ) by evaluating flexural strength and structure after hydrothermal accelerated aging. **Methods:** Thin bars of zirconia: Prettau (Zirkonzahn), Zirprime (Noritake), and NZ (Noritake) n=30 for each brand (22mmX3mmX0.2mm) were cut and ground from blocks sintered according to the manufacturer's specifications. Control specimens for each brand were evaluated in the as-received condition. Experimental specimens were artificially aged at standard autoclave sterilization conditions, 134°C at 0.2MPa (n=5 per group at 5, 50, 100, 150, and 200 hours). Tetragonal to monoclinic transformation was measured by using X-ray diffraction for all groups. Flexural strength was measured in 4-point bending (ASTM1161-B) for all groups. Fracture surfaces were examined by scanning electron microscopy (SEM). Linear regression (with heteroscedasticity-consistent standard errors) for a linear time trend was performed separately for each group.

**Results:** After 200 hours at 134°C and 2 bar, flexural strength decreased from 1328(89.9)MPa to fractured during aging for Prettau; 1041(130)MPa to 779(137)MPa for Zirprime; and 1436(136)MPa to 1243(101)MPa for NZ. Linear regression showed a significant decrease in strength with time. After 200 hours at 134°C and 2 bar some tetragonal crystals transformed to the monoclinic phase in all specimens. The relative XRD peak intensity of the monoclinic to tetragonal crystal phases increased from 0.03 to 3.75 for Prettau, from 0.02 to 2.97 for Zirprime, and from 0.14 to 0.46 for TR-6. The decrease in flexural strength is related to the increase in monoclinic phase.

#### ➔ Conclusion:

Hydrothermal aging of Y-TZP caused a statistically significant decrease in flexural strength of thin bars of Y-TZP which was the result of the transformation from tetragonal to monoclinic crystal structure. **NZ exhibited the least decrease in strength and smallest amount of monoclinic phase after aging.**

### AA DR 235 Biaxial Flexural Strength of a New Dental Zirconia Ceramic

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**Objectives:** To determine and compare effects of storage conditions on biaxial flexural strength of a new type dental zirconia. **Methods:** Disc-shaped specimens (14.0±2 mm diameter, 1.2±0.2mm thick) of three types of zirconia were prepared according to ISO Specification test ISO 6872:2008(E): Noritake: NZ-HIP (fully sintered), NZ-No HIP; LAVA (3M ESPE). One side of each specimen was polished to a final finish of 6.7 microns (JIS #2000). Specimens were water-stored at room temperature at least 2 months, or were heated in an oven at 100°C for 8 hours to remove surface and inner water content, and then immersed in light mineral oil. Specimens were tested in a biaxial flexure jig in a universal testing machine (Model 8562, Instron, Norwood, MA) at a crosshead speed of 0.5 m/min. Peak load at failure was noted and biaxial flexural strength (FS) was de-

termined using methods prescribed in the ISO specification (section 7.3.3.4). Twenty specimens per test condition were made and randomly tested. Statistical analysis consisted on a 2-way ANOVA among results, with Tukey's post-hoc test applied for pairwise means differences at a pre-set alpha of 0.05. **Results:** Table presents mean biaxial flexure strength (MPa)(SD). ANOVA indicated significant effect of major factors (material p<0.001; storage p=0.005) as well as their interaction (p=0.014). Pooled storage effect among material indicated FS NZ-HIP>NZ-NO HIP>LAVA. Within each storage treatment, NZ-HIP>NZ-NO HIP>LAVA. FS of specimens stored in oil were significantly greater than those stored in water for all materials, except NZ-HIP, where there was no significant difference.

Material	Storage	
	Oil	Water
NZ-HIP	2233 (190) <sup>aA</sup>	2273 (160) <sup>aA</sup>
NZ-NO HIP	1805 (195) <sup>aB</sup>	1628 (211) <sup>bB</sup>
LAVA	1224 (144) <sup>aC</sup>	1091 (99) <sup>bC</sup>

Within column (upper case) or row (lower case), values having similar letters are not significantly different.

#### ➔ Conclusion:

Storage condition had no significant effect on HIP-treated specimens, while exposure to water significantly lowered FS of other zirconia groups. In all instances, FS NZ-HIP > NZ-NO HIP > LAVA.

# FUNDAMENTAL TECHNOLOGIES

## MDP BASED ADHESIVES

### PER/ADR 630 Zinc Compromises Chemical Interaction of Acidic Functional Monomers with Dentine

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**Objectives:** Recently, Zn-doped dental adhesives have been advocated to interfere with matrix metalloproteinase-mediated collagen degradation. Nevertheless, there is no information about the effects induced by the inclusion of Zn nitrate on the chemical-physical properties of MDP-based (10-methacryloyloxi-decyl-phosphate) self-etch adhesives. Thus, this investigation aimed at evaluating whether the presence of Zn interfere with the chemical interaction of MDP-based adhesives and dentine. **Methods:** MDP was solvated in a 1:1-water:ethanol solution. MDP was mixed with 0, 1, 2, 3 or 4 ppm of calcium in presence or absence of zinc. Remaining free-calcium (calcium not chemically bonded to MDP) was measured by atomic absorption spectroscopy (AAS). Further AAS was realized with zinc+MDP following a similar protocol in absence of calcium to evaluate the potential formation of MDP-zinc salt. Three experimental solutions were prepared and analysed for each mixture (n=3). MDP-containing self-etch adhesives Clearfil SE Bond (CSE, primer solution) and Clearfil S3 (S3) (Kuraray Medical, Japan) were doped with 5wt% of zinc. Zinc-doped and zinc-free adhesives were applied following manufacturer's recommendations to flat dentine surfaces of extracted human third molars and restorations were built up. After 24h immersion in distilled water at 37°C, the bonded teeth were cut into ~1mm<sup>2</sup> sticks and submitted to  $\mu$ TBS testing. Data were analysed with ANOVA, paired t-test and Tukey's test ( $p < 0.05$ ).

**Results:**  $\mu$ TBS showed mean bond strength (MPa) of  $39.8 \pm 7.3$  for CSE,  $19.3 \pm 4.9$  for zinc-doped CSE and  $38.3 \pm 8.6$  for S3. Zinc-doped S3 was not tested because all sticks failed prematurely. AAS showed reduced free-calcium by adding MDP ( $p < 0.001$ ) and an increase in free calcium when adding zinc ( $p = 0.009$ ). Formation of monomer-zinc salt occurred as a reduction of free-zinc by adding MDP ( $p < 0.001$ ).

#### ➔ Conclusion:

These outcomes showed that zinc may jeopardise the bond strength of MDP-containing adhesives and MDP-calcium salts formation. Addition of zinc should be avoided in this type of self-etch adhesive.

## NEW FUNCTIONAL MONOMER

### 484 Chemical Interaction Of Novel Functional Monomer With Hydroxyapatite And Enamel/dentin

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**The functional monomer 10-methacryloxydecyl dihydrogen phosphate (10-MDP) ionically bonds to hydroxyapatite (HAp)** (Yoshida et al., JDR 2004; 83:454-8). This bond is very stable, as was confirmed by the low dissolution rate of 10-MDP's calcium salt in water.

**Objectives:** To chemically unravel the adhesion mechanism of a novel functional monomer MF8P (6-Methacryloxy-2,2,3,3,4,4,5,5-octafluorohexyl dihydrogen phosphate) (Kuraray, Japan) by studying its molecular interaction with hydroxyapatite (HAp), dentin and enamel using X-ray diffraction (XRD) and solid-state NMR (31P MAS NMR, 31P CP-MAS NMR and 1H MAS NMR). **Methods:** Synthetic HAp particles (Pentax, Japan) were added to ethanol solutions of 0.005mol MF8P for 5min, 1hr, 3hrs, 6hrs and 24hrs (referred to as 'MF8P-HAp\_5min/1h/3h/6h/24h'), after which HAp was washed with ethanol and air-dried. As a reference, MF8P\_Ca-salt was used. The same MF8P ethanol solution was applied on dentin and enamel for 20s, and subsequently air-dried

without washing (referred to as 'MF8P\_D/E'). **Results:** XRD showed the formation of CaHPO4 · 2H2O (DCPD) on HAp, but only after 3hrs. Only MF8P-HAp\_24h samples showed three characteristic peaks representing MF8P\_Ca-salt formation. NMR confirmed adsorption of MF8P onto HAp, even after 5min. XRD of MF8P\_D and MF8P\_E showed three characteristic peaks representing MF8P\_Ca-salt formation already after 5min.

#### ➔ Conclusion:

The novel functional monomer readily adsorbed to HAp, enamel and dentin; MF8P\_Ca-salt was formed on dentin and enamel at short term, but only at long term upon reaction with HAp.

## NOTES

## REFERENCES

- 1* The brand name of CLEARFIL™ PROTECT BOND in the American market is CLEARFIL™ SE PROTECT
- 2* The code name of CLEARFIL™ S3 BOND PLUS at the time of the study is MTB-200
- 3* PANA VIA™ SA CEMENT is the new brand name for CLEARFIL™ SA CEMENT from 1 september 2012
- 4* NZ is the code name for a New Noritake Zirconia.



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## NOTES

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