The Effect of Dentin Desensitizer on Shear Bond Strength of Conventional and Self-adhesive Resin Luting Cements After Aging

B Stawarczyk • R Hartmann • L Hartmann M Roos • M Özcan • I Sailer CHF Hämmerle

Clinical Relevance

Following tooth preparation, application of Gluma Desensitizer does not impair the bond strength of conventional resin luting cements with etch-and-rinse adhesive systems and self-adhesive cements. Based on bond strength data and failure types observed, use of Gluma Desensitizer cannot be advised before cementation with Panavia 21.

Rahel Hartmann, Dr Med Dent, private practice, Ilanz, Switzerland

Leonie Hartmann, Dr Med Dent, private practice, Uster, Switzerland

Malgorzata Roos, PhD, Division of Biostatistics, Institute of Social and Preventive Medicine, University of Zurich, Switzerland

Mutlu Özcan, Prof Dr Med Dent, PhD, Clinic of Fixed and Removable Prosthodontics and Dental Material Science, Center of Dental Medicine, University of Zurich, Switzerland

Irena Sailer, PD, Dr Med Dent, Clinic of Fixed and Removable Prosthodontics and Dental Material Science, Center of Dental Medicine, University of Zurich, Switzerland

Christoph H. F. Hämmerle, Prof Dr Med Dent, Clinic of Fixed and Removable Prosthodontics and Dental Material Science, Center of Dental Medicine, University of Zurich, Switzerland

*Corresponding author: Plattenstrasse 11, Zurich 8032 Switzerland; e-mail: bogna.stawarczyk@zzm.uzh.ch

DOI: 10.2341/10-292-L

SUMMARY

This study tested the impact of Gluma Desensitizer on the shear bond strength (SBS) of two conventional (RelyX ARC, Panavia 21) and two self-adhesive (RelyX Unicem, G-Cem) resin luting cements after water storage and thermocycling. Human third molars (N=880) were embedded in acrylic resin. The buccal dentin was exposed. Teeth were randomly divided into four main groups, and the following cements were adhered: 1) RelyX ARC, 2) Panavia 21, 3) RelyX Unicem, and 4) G-Cem. In half of the teeth in each group, dentin was treated with Gluma Desensitizer. In the conventional cement groups, the corresponding etchant and adhesive systems were applied. SBS of the cements was tested after 1 hour (initial); at 1, 4, 9, 16, and 25 days of water storage; and at 1, 4, 9, 16, and 25 days of thermocycling. SBS data were analyzed by one-way analysis of variance (ANOVA); this was followed by the post hoc Scheffé test and a t-test. Overall, the highest

^{*}Bogna Stawarczyk, Dipl Ing MSc, Clinic of Fixed and Removable Prosthodontics and Dental Material Science, Center of Dental Medicine, University of Zurich, Switzerland

mean SBS (MPa) was obtained by RelyX ARC (ranging from 14.6 \pm 3.9 to 17.6 \pm 5.2) and the lowest by Panavia 21 in combination with Gluma Desensitizer (ranging from 0.0 to 2.9 \pm 1.0). All tested groups with and without desensitizer showed no significant decrease after aging conditions compared with baseline values (p>0.05). Only the Panavia 21/Gluma Desensitizer combination showed a significant decrease after 4 days of thermocyling compared with initial values and 1 day thermocycling. Self-adhesive cements with Gluma Desensitizer showed increased SBS after aging conditions (ranging from 7.4 \pm 1.4 to 15.2 \pm 3) compared with groups without desensitizer (ranging from 2.6 \pm 1.2 to 8.8 \pm 2.9). No cohesive failures in dentin were observed in any of the test groups. Although self-adhesive cements with and without desensitizer presented mainly adhesive failures after water storage (95.8%) and thermocyling (100%), conventional cement (RelyX ARC) showed mainly mixed failures (90.8% and 89.2%, after water storage and thermocyling, respectively). Application of the Gluma Desensitizer to dentin before cementation had a positive effect on the SBS of self-adhesive cements.

INTRODUCTION

A series of studies reported that adhesive cementation techniques increase fracture resistance and thereby enhance the clinical performance of ceramic dental restorations. ¹⁻⁶ Resin luting cements not only improve the retention of dental restorations but also exhibit low solubility ⁷ and microleakage in the oral environment compared with traditional cements such as glass ionomer, zinc phosphate, and polycarboxylate cements. ⁸⁻¹² Furthermore, with translucent ceramic restorations, shade could be optimized with resin-based luting cements. ¹³

Conventional resin luting cements require pretreatment of the prepared dentin, which is technique-sensitive and time-consuming. For this reason, recently, self-adhesive resin luting cements have been promoted, because they do not require pretreatment of dentin. ¹² Their adhesive mechanism is derived from acidic monomers in their composition that infiltrate into the demineralized collagen network. ¹⁴ In some studies, the shear bond strength of self-adhesive resin luting cements on dentin was found to be comparable with that of conventional resin luting cements, ¹⁵⁻¹⁸ but in others, lower bond strengths were reported with such cements. ^{19,20}

During tooth preparation, after enamel is removed, depending on the amount of tooth reduction, millions of dental tubules are exposed. Dependent in tubules increase the risk of potential pulpal injury. Discussive or the heat generated during drilling, or it could be due to the chemical composition of the adhesives. Sensitivity after tooth preparation could be eliminated by pretreatment of dentin with desensitizing solutions. As soon as the dentin tubules are sealed, the hydrodynamic movement of dentin liquid is reduced, and eventually sensitivity is decreased.

One of the most broadly studied desensitizers, Gluma Desensitizer, is a glutaraldehyde-based substance. ^{25,29-32} It reduces dentin permeability and at the same time disinfects the dentin. ^{33,34} Especially when glutaraldehyde was combined with hydroxyethylmethacrylate (HEMA), improved bond strengths were obtained. ³⁵ Despite dentin precipitation after tooth preparation, the diffusion of monomers to dentin is likely to be accelerated by the presence of HEMA. ³⁶

Dentin sealing through hybrid layer formation could also be achieved with dentin adhesives. Particularly, Gluma Desensitizer and adhesives that include HEMA in their composition show increased wettability and good infiltration into the dentin tubules. This, in turn, produces a resin-reinforced layer on the dentin. ³⁷⁻³⁹

It has been shown previously that increased bond strength could be attained with self-adhesive resin cements in combination with Gluma Desensitizer compared with conventional resin cement (Panavia 21) after application of desensitizer. 32 Unfortunately, in that study, short-term aging was practiced. In fact, Panavia 21 showed excellent bond strength to dentin without a desensitizing agent.^{5,17,19} Several other *in* vitro studies have showed an inhibiting effect of Gluma Desensitizer on the bond strength of conventional resin cements as a consequence of coagulation of dentin fluid proteins, plugging the tubules. 30,31 Again in all these studies, the specimens were not aged. Therefore, information is lacking regarding whether application of this desensitizer remains stable after long-term aging conditions.

Because HEMA is a monomethacrylate with a -OH group that makes it hydrophilic and unstable, ⁴⁰ it can be anticipated that bond strengths would decrease after increased water storage or long-term aging conditions. In *in vitro* studies, the aging effect could be tested by storing the specimens in water at

37°C, or by subjecting them to accelerated aging in a thermocycling (5°C-55°C) machine, in which case the latter is usually considered as representing the worst case scenario.

Therefore, the objective of this study was to investigate the impact of Gluma Desensitizer on the bond strength of conventional and self-adhesive resin luting cements after water storage and long-term thermocycling. The hypotheses tested were 1) that bond strength results after Gluma Desensitizer application would decrease by aging, and 2) that thermocycling would decrease bond strength further than it would be reduced by water storage.

METHODS AND MATERIALS

Two conventional resin luting cements, one of which has an etch-and-rinse adhesive system (RelyX ARC, 3M ESPE, Seefeld, Germany) and the other a self-etch adhesive (Panavia 21, Kuraray, Osaka, Japan), and two self-adhesive resin cements (RelyX Unicem, 3M ESPE and G-CEM, GC Europe, Leuven, Belgium) were used in this study. The brands, batch numbers, abbreviations, manufacturers, and chem-

phosphate; 4-META, 4-methacryloyloxyethyl-trimellitat-anhydrid; and UDMA, urethane-dimethacrylate.

ical compositions of the tested materials are listed in Table 1.

Specimen Preparation

For this study, 880 caries-free human third molars were collected. The teeth were cleaned of periodontal tissue residue using a periodontal scaler and were stored in 0.5% Chloramine T at room temperature for a maximum of seven days. Afterward, they were stored in distilled water for a maximum of six months at 5°C until the experiments were begun.⁴¹

To achieve flat surfaces, buccal surfaces of the teeth were leveled out parallel to the tooth axis using a polishing machine (LaboPol-21, Struers, Bellerup, Denmark) with silicon carbide polishing paper (P400, ScanDia, Hagen, Germany). The teeth were then embedded in acrylic resin (ScandiQuick, ScanDia) and were ground finished with carbide polishing paper (P500, ScanDia) until a sufficient bond area of 4×4 mm dentin surface was exposed.

The specimens (N=880, n=220 per cement group) were then randomly divided into four main groups for each cement (RXA, PAN, RXU, GCM).

Brand (Batch No.)	Abbreviations	Manufacturer	Chemical Composition	
RelyX ARC (3415A1)	RXA	3M ESPE, Seefeld, Germany	BisGMA, TEGDMA, dimethacrylate, zirconia and silic fillers, initiators	
Adper Scotch Bond (N186179/N195379/N191364)		,	Etchant: phosphoric acid Primer: HEMA, ethanol Adhesive: HEMA, Bis-GMA	
Panavia 21 (00406C UNI TC/00647C CAT)	PAN -	Kuraray Dental Co Ltd, Osaka, Japan	Hydrophobic aromatic dimethacrylate, hydrophilic alipathic dimethacrylate, MDP, fillers, BPO, hydrophil	
Clearfil Porcelain Bond Activator (00208B)			dimethacrylate, fillers, DEPT, sodium aromatic sulfonate MDP, HEMA, water, MASA, accelator, water	
Clearfil SE Bond Primer (00769A)			MASA, Na-benzene sulfonate, accelator, water	
RelyX Unicem (352388)	RXU	3M ESPE, Seefeld, Germany	Powder: glass fillers, silica, calcium hydroxide, self- cure initiators, pigments, light-cure initiators Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-cure initiators	
G-CEM Capsule (803061)	GCM	GC, Leuven, Belgium	4-META, UDMA, alumina-silicate glass, pigments, dimethacrylates, water, phosphoric ester monomer, initiators, camphorquinone	
Gluma Desensitizer (20088)	G	Heraeus Kulzer, Hanau, Germany	HEMA, glutaraldehyde, distilled water	

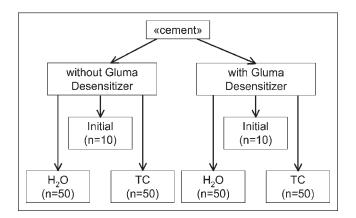


Figure 1. Test groups according to pretreatment and aging.

Bonding Procedures

The specimens were secured in a special holding device to keep the bonding surfaces parallel to the bench. Each main cement group was further randomly divided into two groups (n=110). In half of the groups, dentin surfaces were pretreated with Gluma Desensitizer (Heraeus Kulzer, Hanau, Germany) (RXA-G, PAN-G, RXU-G, GCM-G), according to the manufacturer's instructions (Figure 1). Although the self-adhesive cements did not require dentin conditioning, the corresponding etchant and adhesive systems of the conventional resin cements were applied to the dentin before cementation. All cements were applied according to the manufacturer's instructions.

For dual-polymerized RXC, an etch-and-rinse-based adhesive system was used whereby the dentin was etched and rinsed with $37\%~H_3PO_4$ for 15 seconds. Then primer and adhesive were applied accordingly. For chemically polymerized PAN, its corresponding self-etch adhesive was applied.

The cements were activated and placed in an acrylic mold (inner diameter: 2.9 mm; height: 4 mm) (D+R Tec, Birmensdorf, Switzerland) and were pressed onto the dentin surface by a holding device. To achieve homogeneous dispersion of the cement in the acrylic mold, a hexagonal steel screw with an outer diameter of 2.8 mm was inserted parallel to the long axis of the acrylic cylinder in its center. A load of 100 g was applied to the screw, and excess cement was removed using microbrushes. This procedure allowed a cement thickness of 0.5 mm in all specimens. After polymerization, the molds were gently removed.

The bonded specimens again were randomly distributed among 11 groups (n=10) according to the aging conditions. One group was tested 1 hour

after polymerization to attain the initial bond strength (n=10 per cement type and pretreatment).

Aging Conditions

Specimens were subjected to 1, 4, 9, 16, and 25 days of water storage or 1, 4, 9, 16, and 25 days of thermocycling. Water storage aging was carried out in distilled water at 37°C in an incubator (UMS, Memmert, Schwabach, Germany). Specimens for thermocycling aging were stored initially for 24 hours in distilled water at 37°C in the incubator, and then were aged in a thermocycling machine (HAAKE W15, Willitec, Feldkirchen-Westerham, Germany) at between 5°C and 55°C with a dwelling time of 20 seconds for each bath.

Shear Bond Strength

Shear bond strength was measured in a Universal Testing Machine (Zwick/Roell Z010, Ulm, Germany) at a crosshead speed of 1 mm/min. Specimens were positioned in the jig of the testing machine with the tooth surface parallel to the loading direction. Bond strength (MPa) was calculated using the following formula: Force to failure (N)/Bonding area (mm²).

Failure Analysis

Three failure types were expected to be observed: 1) adhesive (no cement remnants left on the dentin surface), 2) mixed failure (cement remnants partially left on dentin with dentin surface exposed), and 3) cohesive failure in dentin. Failure types were noted by one operator under an optical microscope (Wild M3B, Heerbrugg, Switzerland) at a magnification of 25×.

Statistical Analysis

Data were analyzed using statistical software (Statistical Package for the Social Sciences [SPSS], version 15, SPSS Inc, Chicago, IL, USA). Approximate normality of data distribution was tested using Komogorov-Smirnov and Shapiro-Wilk tests. Oneway analysis of variance (ANOVA) followed by the Scheffé post hoc test was used to detect significant differences between groups. The effects of aging conditions, cement type, and application of desensitizer were analyzed using a two-sample Student's *t*-test. *P* values smaller than 5% were considered statistically significant for all tests.

RESULTS

Specimens that did not survive the aging processes were considered as 0 MPa. Kolmogorov-Smirnov and

	Initial	1 day	4 days	9 days	16 days	25 days
Water (37°C)						
RXA	15.1 (5.6) ^{A,d}	16.0 (2.9) ^{A,d}	15.8 (3.3) ^{A,ef}	14.7 (4.9) ^{A,f}	14.6 (3.9) ^{A,e}	16.3 (5.1) ^{A,d}
RXA-G	10.6 (1.7) ^{AB,c}	14.7 (2.3) ^{AB,d}	16.3 (4.7) ^{B,f}	14.1 (4.7) ^{AB,ef}	10.5 (2.9) ^{A,cde}	11.3 (4.7) ^{AB,b}
PAN	6.2 (1.6) ^{A,ab}	5.0 (1.6) ^{A,abc}	5.7 (2.1) ^{A,abc}	6.6 (1.4) ^{A,abc}	6.4 (1.8) ^{A,abc}	3.9 (1.6) ^{A,a}
PAN-G	2.6 (0.2) ^{A,a}	2.9 (0.9) ^{A,a}	2.6 (0.8) ^{A,a}	2.4 (0.7) ^{A,a}	2.6 (0.6) ^{A,a}	2.7 (0.5) ^{A,a}
RXU	4.5 (0.7) ^{A,a}	8.4 (2.1) ^{B,bc}	8.0 (1.6) ^{B,bcd}	8.4 (1.5) ^{B,bcd}	8.8 (2.9) ^{B,bcd}	6.8 (3.1) ^{AB,a}
RXU-G	7.4 (1.4) ^{A,bc}	7.6 (1.5) ^{A,bc}	11.2 (1.9) ^{AB,de}	11.7 (2.5) ^{B,def}	13.1 (4.3) ^{B,de}	10.2 (2.4) ^{AB,b}
GCM	2.6 (1.2) ^{A,a}	4.7 (1.6) ^{AB,ab}	3.6 (1.9) ^{AB,ab}	4.2 (1.5) ^{AB,ab}	5.1 (2.9) ^{AB,ab}	6.1 (3.0) ^{B,ab}
GCM-G	8.6 (2.3) ^{A,bc}	8.9 (3.9) ^{A,c}	9.5 (4.5) ^{A,cd}	9.5 (3.2) ^{A,cde}	12.7 (2.2) ^{A,de}	12.8 (4.6) ^{A,cd}
Thermocycling	g (5°C-55°C)					
RXA	15.1 (5.6) ^{A,d}	14.7 (6.7) ^{A,cd}	14.9 (3.6) ^{A,cd}	17.6 (5.2) ^{A,e}	15.6 (3.4) ^{A,e}	16.7 (2.9) ^{A,de}
RXA-G	10.6 (1.7) ^{A,c}	19.3 (1.9) ^{C,d}	16.5 (2.7) ^{CD,d}	15.1 (2.1) ^{CD,de}	14.7 (2.1) ^{AB,de}	14.6 (5.2) ^{AB, ©}
PAN	6.2 (1.6) ^{A,ab}	5.8 (2.2) ^{A,ab}	4.8 (2.3) ^{A,ab}	5.2 (2.7) ^{A,ab}	4.1 (2.2) ^{A,ab}	2.7 (3.6) ^{A,ab}
PAN-G	2.6 (0.2) ^{B,a}	2.0 (0.4) ^{B,a}	0.8 (1.3) ^{A,a}	0 ^{A,a}	0 ^{A,a}	0 ^{A,a}
RXU	4.5 (0.7) ^{A,a}	4.7 (2.6) ^{A,ab}	5.9 (2.1) A,b	7.7 (2.3) ^{A,bc}	6.9 (3.3) ^{A,bc}	5.8 (1.4) ^{A,bo}
RXU-G	7.4 (1.4) ^{A,bc}	8.9 (3.8) ^{AB,b}	11.6 (1.6) ^{BC,c}	15.2 (3.0) ^{C,de}	10.4 (2.9) ^{AB,cd}	10.6 (2.7) ^{AB,c}
GCM	2.6 (1.2) ^{A,a}	5.5 (1.9) ^{A,ab}	5.0 (2.0) ^{A,ab}	4.1 (3.3) ^{A,ab}	3.5 (2.5) ^{A,ab}	4.3 (2.7) ^{A,ab}
GCM-G	8.6 (2.3) ^{A,bc}	9.3 (3.3) ^{A,bc}	13.1 (3.3) ^{A,cd}	10.4 (4.0) ^{A,cd}	10.7 (3.0) ^{A,cd}	11.2 (2.5) ^{A,d}

^a Small letters indicate statistical differences between the tested cements with and without Gluma Desensitizer within each aging phase and each aging type; capital letters represent significant differences between aging times within each resin cement group and each aging type.

Shapiro-Wilk tests indicated that the data were normally distributed because the tests were significant for 6 of 88 (6.8%) testing groups (α =0.05). Consequently, parametric statistical analyses were applied.

Bond Strength Stability

Water Storage (37°C)—In all water storage conditions (one hour to 25 days), mean bond strengths of conventional resin cement RXA and PAN showed no

significant difference (p>0.05). For the self-adhesive cements, RXU (p<0.001) showed a significant increase after one day but remained stable after 25 days of water storage. The other self-adhesive cement, GCM (p=0.011), showed a significant increase after 25 days compared with initial values (Table 2, Figure 2).

Among the desensitizer applied groups, PAN-G and GCM-G (p>0.05) showed no significant difference at all time points of water storage, but RXA-G

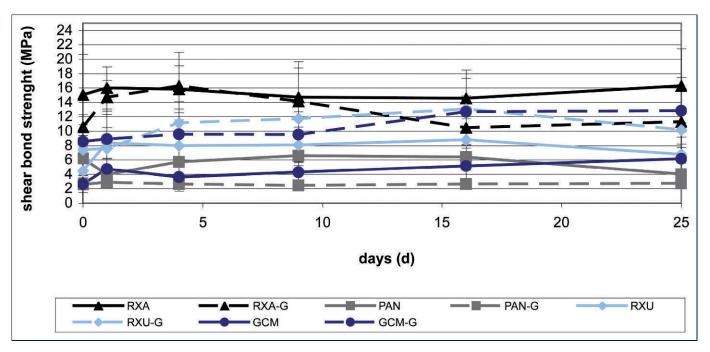


Figure 2. Mean bond strengths (SD) of experimental groups after water storage (1, 4, 9, 16, and 25 days).

(p=0.002) presented the only significant difference at four days and 16 days of water storage. Results remained stable for this group at 25 days. At baseline and one day of aging, RXU-G (p<0.001) showed the lowest bond strength results, but results were enhanced by further aging (Figure 2).

Thermocycling (5°C-55°C)—Thermocycling did not show a significant effect on the bond strength of all cements without desensitizer (RXA: p=0.721; PAN: p=0.115; RXU: p=0.089; GCM: p=0.197) (Table 2, Figure 3).

In comparison with conventional cements, RXA-G (p<0.001) showed enhanced results after one, four, and nine days of thermocycling. On the other hand, thermocycling decreased the mean bond strength for PAN-G (p<0.001) after four days of thermocycling, but it did not change the results for GCM-G (p=0.083) at all time points. Among the self-adhesive cements, the RXU-G group (p<0.001) showed an increase after four and nine days, but a decrease after 16 and 25 days of thermocycling (Figure 3).

Desensitizer Effect

Water Storage (37°C)—Considering the conventional cements, for RXA, the use of desensitizer (RXA-G) decreased the results significantly at baseline (p=0.036), 16 days (p=0.016), and 25 days (p=0.034) of water storage. Also, desensitizer use (PAN-G) decreased the bond strength results for PAN at all

aging conditions (p<0.001, p=0.032). The self-adhesive cements GCM and RXU showed significantly increased results with the desensitizer (GCM-G, RXU-G) (p<0.001, p=0.018) at all time points (Figure 2).

Thermocycling $(5^{\circ}\text{C}-55^{\circ}\text{C})$ —Use of desensitizer decreased the bond strength for PAN at all time points (p<0.001, p=0.041). Similar observations were made for RXA-G except at baseline (Figure 3).

When self-adhesive cements were used together with the desensitizer (RXU-G and GCM-G), results improved significantly (p<0.001, p=0.023) (Figure 3).

Water Storage vs Thermocycling

Water storage and thermocycling conditions showed different aging effects, depending on the cement type and the cement-desensitizer combination (Table 3). Thermocycling in general was more aggressive in all aging conditions on PAN-G.

Failure Type

No cohesive failures in dentin were observed in any of the test groups. Although self-adhesive cements with and without desensitizer presented mainly adhesive failures after water storage (97.6%) and thermocycling (100%), conventional cement RXA

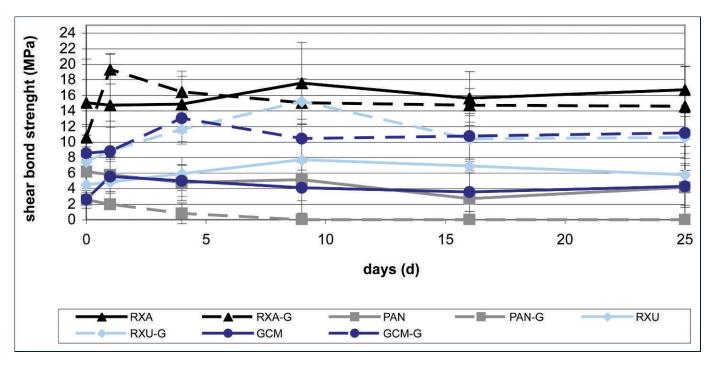


Figure 3. Mean bond strengths (SD) of experimental groups after thermocycling (1, 4, 9, 16, and 25 days).

showed mainly mixed failures (94% and 90%, after water storage and thermocycling, respectively).

DISCUSSION

The conventional resin luting cement RXA with an etch-and-rinse adhesive system showed the highest shear bond strength values of all tested cements with and without pretreatment with Gluma Desensitizer. The self-adhesive resin cement GCM showed the lowest initial bond strength. However, already after 24 hours of aging in water or thermocycling, comparable values were obtained with the conventional resin luting cement PAN. This phenomenon can be explained by the fact that GCM continues to polymerize after luting. Although in this study chemically polymerized PAN was used, in another study similar observations were made in which GCM and dual polymerized Panavia F2.0 presented similar results after 24 hours of water storage. 42 Therefore, post polymerization could be expected with this self-adhesive resin cement.

When both tested self-adhesive resin cements were combined with Gluma Desensitizer, a higher and more stable bond strength was attained compared with the conventional resin cement PAN. On the other hand, Gluma Desensitizer use decreased the bond strength of PAN (PAN-G) compared with PAN without desensitizer. In addition, thermocycling decreased the bond strength for PAN-G. After

nine days of thermocycling, specimens in the PAN-G groups were spontaniously debonded. Hence, the primary hypothesis could be partially accepted, because Gluma Desensitizer decreased bond strength after aging only for PAN.

Table 3:	Statistical Comparison Between Water Stored and Thermocycling (Two-Sample t-Test) ^a						
	1 day	4 days	9 days	16 days	25 days		
RXA	0.584	0.545	0.229	0.544	0.824		
RXA-G	<0.001 ↑	0.920	0.585	0.002 ↑	0.149		
PAN	0.392	0.352	0.160	0.023 ↓	0.335		
PAN-G	0.010 ↓	0.003 ↓	<0.001 ↓	<0.001 ↓	<0.001 ↓		
RXU	0.003 ↓	0.023 ↓	0.468	0.189	0.368		
RXU-G	0.347	0.558	0.011 ↑	0.124	0.732		
GCM	0.307	0.124	0.879	0.202	0.166		
GCM-G	0.809	0.061	0.574	0.119	0.336		
a ↓ Thermocycling was more aggressive, ↑ water storage was more							

^a ↓ Thermocycling was more aggressive, ↑ water storage was more aggressive.

Numerous *in vitro* studies have described the impact of Gluma Desensitizer on the resin-dentin interface of conventional resin luting cements. ^{30-32,43-45} Three studies reported no impact of the desensitization of dentin on bond strength with conventinal resin luting cements. ⁴³⁻⁴⁵ Other studies described a negative effect of the desensitizer on the bond strength of the conventional resin luting cement PAN. ³⁰⁻³² In another study, it was stated that the resin luting cement was not able to polymerize with the dentin desensitizer. ³² In this study, the long-term bond strength of both self-adhesive resin luting cements tended to be positively influenced by the application of desensitizers.

Yim and others reported that the bond strength of self-adhesive resin luting cements in combination with desensitizers exceeded the bond strength of self-adhesive resin cement alone. 30 This might occur because of the fact that Gluma Desensitizer contains glutaraldehyde and HEMA, resulting in hydrophilic properties that improve bonding to hydrophilic dentin. Self-adhesive resin luting cements contain phosphate groups or phosphoric ester monomers: RXU—methacrylated phosphoric esters; GCM-4-META. The previously mentioned observations may be explained by the possible condensation reaction between HEMA and phosphate in dentin through the elimination of water. 35 Alternatively, a reaction between glutaraldehyde and phosphate may have led to very strong and stable bonding of the Gluma Desensitizer in combination with self-adhesive resin luting cements.

Water storage and thermocycling aging affected the results, depending on the cement type. Thermocycling affected only the groups RXA-G (one day of aging), RXU (one day and four days of aging), PAN (16 days of aging), and PAN-G (in all terms of aging), yielding to significantly lower bond strength than with water-stored groups. Water storage significantly decreased the mean bond strength of RXA-G and RXU-G (nine days of aging) compared with thermocycling. No differences were found between the effects of aging conditions for all other groups. Because most of the results were not significantly different between the two aging types, no concrete conclusions could be drawn. Therefore, the second hypothesis was rejected. Regarding the effect of thermocycling, after 1500 cycles (5°C/55°C, transfer time of 10 seconds, dwelling time of 20 seconds), a significant influence was observed on the bond strength of resin cements (RXA, PAN, Multilink, RXU). 17 The variation between the results of this study and those of the previous study¹⁷ could be

explained by the differences in curing modes of the two cements, dentin quality, and the operator factor.

The failure types observed in this study should be considered in interpreting the results. In a previous study, although RXU with and without Gluma Desensitizer showed exclusively cohesive failure within the cement, PAN showed 70% and 80% mixed failures with and without Gluma Desensitizer, respectively.³⁰ On the contrary, another study observed predominantly adhesive failures for RXU (80%) and GCM (90%). 46 In that study, GCM showed higher µTBS values (16.9 ± 10.3 MPa) than RXU $(12.5 \pm 2.4 \text{ MPa})$, but with high standard deviation. In this study, self-adhesive resin cements with and without pretreatment with the Gluma Desensitizer showed only adhesive failures after thermocycling. GCM and GCM-G showed failure types exclusively after both water storage and thermocycling. In that respect, thermocycling seems to have a more detrimental aging effect on some cements; this needs to be further investigated.

Only RXA conventional cement with and without Gluma Desensitizer presented mainly mixed failure types. Even though higher shear bond strength results were observed compared with conventional cements, adhesive failures were observed more often with self-adhesive resin cements. This can be explained by the adhesion mechanism of self-adhesive cements. Cohesive failures are less likely with such resin cements because of partial retention of the smear layer and the low infiltration ability of these cements. ⁴⁷

One possible reason for the observed variation in bond strength values is the quality and micromorphology of the human teeth. 20,48 Moreover, the bonding performance of resin cements is dependent on the quality of the hybrid layer, which is established during pretreatment of dentin. 49 When this layer is porous, $\rm H_2O$ molecules may penetrate and yield to hydrolysis. Because in this study extracted human teeth were used, some loss of dentin fluid and proteins could be expected. Furthermore, it must be realized that apart from all efforts to standardize the test procedures, the possibility of error remains during application of the bonding agents or the resin cements. 50

Not only the bond strength of the cements but also the retention form of the preparation, marginal integrity, and clinical microleakage are key parameters in judging the effectiveness of any resin cement system. Future studies should assess the effects of other desentizing agents on the bond stength of tested cements.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following could be concluded:

- Conventional resin cement with an etch-and-rinse adhesive, RelyX ARC, showed the highest bond strength to dentin with and without pretreatment with Gluma Desensitizer.
- Self-adhesive cements with Gluma Desensitizer showed increased bond strength after aging conditions.
- The use of Gluma Desensitizer decreased the bond strength of the conventional resin luting cement, Panavia 21.
- No cohesive failures in dentin were observed in any of the test groups.

Acknowledgements

The authors thank 3M ESPE and GC Europe for their support with study materials. We are grateful to Martina A. Schmid for her assistance in editing this manuscript.

(Accepted 28 February 2011)

REFERENCES

- Bernal G, Jones RM, Brown DT, Munoz CA, & Goodacre CJ (1993) The effect of finish line form and luting agent on the breaking strength of Dicor crowns *International* Journal of Prosthodontics 6(3) 286-290.
- Christensen GJ. (1999) Porcelain-fused-to-metal vs. nonmetal crowns Journal of American Dental Association 130(3) 409-411.
- Malament KA, & Socransky SS (2001) Survival of Dicor glass-ceramic dental restorations over 16 years. Part III: Effect of luting agent and tooth or tooth-substitute core structure *Journal of Prosthetic Dentistry* 86(5) 511-519.
- Burke FJ, Fleming GJ, Nathanson D, & Marquis PM (2002) Are adhesive technologies needed to support ceramics? An assessment of the current evidence *Journal* of Adhesive Dentistry 4(1) 7-22.
- Stewart GP, Jain P, & Hodges J (2002) Shear bond strength of resin cements to both ceramic and dentin. Journal of Prosthetic Dentistry 88(3) 277-284.
- Behr M, Rosentritt M, Mangelkramer M, & Handle G (2003) The influence of different ceramics on the fracture resistance and marginal adaptation of all-ceramic and fiber-reinforced crowns *International Journal of Prostho*dontics 16(5) 538-542.
- Gardolle DA, Mortier E, Jacquot B, & Panighi MM (2008)
 Water sorption and water solubility of current luting cements: An in vitro study Quintessence International 39(3) e107-e114.
- 8. Shortall AC, Fayyad MA, & Williams JD (1989) Marginal seal of injection-molded ceramic crowns cemented with three adhesive systems *Journal of Prosthetic Dentistry* **61(1)** 24-27.

White SN, Sorensen JA, Kang SK, & Caputo AA (1992)
 Microleakage of new crowns and fixed partial denture
 luting agents Journal of Prosthetic Dentistry 67(1)
 156-161.

- White SN, Yu Z, Tom JF, & Sangsurasak S (1994) In vivo microleakage of luting cements for cast crowns *Journal of Prosthetic Dentistry* 71(4) 333-338.
- Li ZC, & White SN (1999) Mechanical properties of dental luting cements Journal of Prosthetic Dentistry 81(5) 597-609.
- Behr M, Rosentritt M, Regnet T, Lang R, & Handel G (2004) Marginal adaptation in dentin of a self-adhesive universal resin cement compared with well-tried systems Dental Materials 20(2) 191-197.
- Karaagaclioglu L, & Yilmaz B (2008) Influence of cement shade and water storage on the final color of leucitereinforced ceramics Operative Dentistry 33(4) 386-391.
- Bayle MA, Gregoire G, & Sharrock P (2007) The role of acrylophosphonic acid monomers in the formation of hybrid layers based on self-etch adhesive *Journal of Dentistry* 35(4) 302-308.
- De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, & Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin Dental Materials 20(10) 963-971.
- 16. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH, & Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel Clinical Oral Investigations 9(3) 161-167.
- Holderegger C, Sailer I, Schuhmacher C, Schlapfer R, Hämmerle CHF, & Fischer J (2008) Shear bond strength of resin cements to human dentin *Dental Materials* 24(7) 944-950.
- 18. Erdemir U, Sar-Sancakli H, Yildiz E, Ozel S, & Batur B (2011) An in vitro comparison of different adhesive strategies on the micro push-out bond strength of a glass fiber post *Medicina Oral Patologia Oral Cirurgia Bucal* [Epub ahead]
- 19. Lüthy H, Loeffel O, & Hämmerle CHF (2006) Effect of thermocycling on bond strength of luting cements to zirconia ceramic. *Dental Materials* **22(2)** 195-200.
- Yang B, Ludwig K, Adelung R, & Kern M (2006) Microtensile bond strength of three luting resins to human regional dentin *Dental Materials* 22(1) 45-56.
- Richardson D, Tao L, & Pashley DH (1991) Dentin permeability: effect of crown preparation *International* Journal of Prosthodontics 4(3) 219-225.
- Pashley DH, & Pashley EL (1991) Dentin permeability and restorative dentistry: A status report for the American Journal of Dentistry American Journal of Dentistry 4(1) 5-9.
- Zach L, & Cohen G (1962) Thermogenesis in operative techniques: Comparison of four methods *Journal of Prosthetic Dentistry* 12(5) 977-984.
- Camps J, Pizaht S, Dejou J, & Franqiun JC (1998) Effects of desensitizing agents on human dentin permeability American Journal of Dentistry 11(6) 286-290.

- 25. Felton DA, Bergenholtz G, & Kanoy B (1991) Evaluation of the desensitizing effect of Gluma Dentin Bond on teeth prepared for complete-coverage restorations *International Journal of Prosthodontics* **4(3)** 292-298.
- Watanabe T, Sano M, Itoh K, & Wakumoto S (1991) The effect of primers on the sensitivity of dentin *Dental Materials* 7(3) 148-150.
- 27. Dondi dall'Orologio G, & Malferrari S (1993) Desensitizing effects of Gluma Primer and Gluma 2000 on hypersensitive dentin *American Journal of Dentistry* **6(6)** 283-286.
- Schüpbach P, Lutz F, & Finger WJ (1997) Closing of dentinal tubules by Gluma desensitizer European Journal of Oral Science 105(5 Part 1) 414-421.
- Bowes JH, & Cater CW (1966) The reaction of glutaraldehyde with protein and other biological materials Journal of Royal Microscopical Society 85(2) 193-200.
- Yim NH, Rueggeberg FA, Caughman WF, Gardner FM, & Pashley DH (2000) Effect of dentin desensitizers and cementing agents on retention of full crowns using standardized crown preparation. *Journal of Prosthetic Dentistry* 83(4) 459-465.
- 31. Huh JB, Kim JH, Chung MK, Lee HY, Choi YG, & Shim JS (2008) The effect of several dentin desensitizers on shear bond strength of adhesive resin luting cement using self-etching primer *Journal of Dentistry* **36(12)** 1025-1032.
- 32. Sailer I, Tettamanti S, Stawarczyk B, Fischer J, & Hämmerle CH (2010) In vitro study of the influence of dentin desensitizing and sealing on the shear bond strength of two universal resin cements *Journal of Adhesive Dentistry* **12(5)** 381-392.
- 33. Meiers JC, Griffo TE, & Miller GA (1992) Antimicrobial activity of dentin bonding systems and glass ionomers *Journal of Dental Research* **71(Supplement 1)** Abstract #1639.
- 34. Schmidlin PR, Zehnder M, Gohring TN, & Waltimo TM (2004) Glutaraldehyde in bonding systems disinfects dentin in vitro *Journal of Adhesive Dentistry* **6(1)** 61-64.
- 35. Munksgaard EC, & Asmussen E (1984) Bond strength between dentin and restorative resins mediated by mixtures of HEMA and glutaraldehyde *Journal of Dental Research* **63(8)** 1087-1089.
- Nakabayashi N, Watanabe A, & Gendusa NJ (1992)
 Dentin adhesion of modified 4-META/MMA-TBB resin: function of HEMA Dental Materials 8(4) 259-264.
- 37. Erickson RL (1989) Mechanism and clinical implications of bond formation for two dentin bonding agents *American Journal of Dentistry* (**Supplement 2**) 117-123.

- 38. Edler TL, Krikorinon E, & Thompson VP (1991) FITR surface analysis of dentin and dentin bonding agents Journal of Dental Research 70(Supplement 1) Abstract #1534.
- Dagostin A, & Ferrari M (2002) Effect of resin sealing of dentin on the bond strength of ceramic restorations Dental Materials 18(4) 304-310.
- 40. Salz U, Zimmermann J, Zeuner F, & Moszner N (2005) Hydrolytic stability of self-etching adhesive systems Journal of Adhesive Dentistry 7(2) 107-116.
- 41. ISO/TS 11405: 2003. Dental materials—Testing of adhesion to tooth structure. Brussels, Belgium: European Committee for Standardization http://www.iso.org/iso/store.htm
- 42. Aguiar TR, Francescantonio M, Ambrosano GM, & Giannini M (2010) Effect of curing mode on bond strength of self-adhesive resin luting cements to dentin *Journal of Biomedical Materials Research Part B Applied Biomaterials* 93(1) 122-127.
- 43. Cobb DS, Reinhardt JW, & Vargas MA (1997) Effect of HEMA-containing dentin desensitizers on shear bond strength of a resin cement *American Journal of Dentistry* **10(2)** 62-65.
- 44. Soeno K, Taira Y, Matsumura H, & Atsuta M (2001) Effect of desensitizers on bond strength of adhesive luting agents to dentin *Journal of Oral Rehabilitation* **28(12)** 1122-1128.
- 45. Aranha AC, Siqueira Junior Ade S, Cavalcante LM, Pimenta LA, & Marchi GM (2006) Microtensile bond strengths of composite to dentin treated with desensitizer products *Journal of Adhesive Dentistry* **8(2)** 85-90.
- 46. Viotti RG, Kasaz A, Pena CE, Alexandre RS, Arrais CA, & Reis AF (2009) Microtensile bond strength of new self-adhesive luting agents and conventional multistep systems The Journal of Prosthetic Dentistry 102(5) 306-312.
- Monticelli F, Osorio R, Mazzitelli C, Ferrari M, & Toledano M (2008) Limited decalcification/diffusion of self-adhesive cements into dentin *Journal of Dental* Research 87(10) 974-979.
- 48. Oilo G (1993) Bond strength testing—What does it mean? *International Dental Journal* **43(5)** 492-498.
- Triolo PT, Kelsey WP, & Barkmeier WW (1995) Bond strength with various dental substrates Journal of Prosthetic Dentistry 74(5) 463-468.
- Pashley DH, Ciucchi B, Sano H, Carvalho RM, & Russell CM (1995) Bond strength versus dentin structure: A modelling approach Archives of Oral Biology 40(12) 1109-1118.