



<b>Title</b>	<b>Effect of delayed activation of light-cured composites on all-in-one adhesives</b>
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**113** Nitric Acid-Modified N-Phenyliminodiacetic Acid - A Total Self-Etching Primer. J.M. ANTONUCCI\*, G.E. SCHUMACHER<sup>2</sup> F.C. EICHMILLER<sup>2</sup>. (\*NIST, <sup>2</sup>ADAHF-PRC, Gaithersburg, MD USA).

N-Phenyliminodiacetic acid (PIDAA) in aqueous acetone is an effective self-etching primer for dentin, but in this form, is not able to adequately etch the surfaces of prepared enamel. **Objective:** To develop a total self-etching primer based on aqueous acetone mixtures of nitric acid and PIDAA. **Materials & Methods:** A two-step bonding protocol was used: 1) aqueous acetone (1:1 by mass) solutions of PIDAA (6.4 %) and nitric acid (NA) at two concentrations (2.5 % or 1.25 % or 0.625 %) were applied to either bovine enamel or human dentin surfaces for either 30 or 60 s, rinsed with H<sub>2</sub>O and air dried. 2) Five coats of 20 % photoactivated PMGDM in acetone were then applied, air dried and irradiated with visible light for 20 s prior to placement of a photocurable composite (irradiation time=60 s). **Results:** Shear bond strengths (SBS) in MPa after 24 h storage in water at 23 °C were:

Bovine Enamel			Human Dentin		
Self-etching Primer	Time (s)	SBS (SD)	Self-etching Primer	Time (s)	SBS (SD)
PIDAA + 1.25 % NA	60	27.9 (9.6)	PIDAA (Control)	60	24.7 (8.4)
PIDAA + 1.25 % NA	30	27.3 (8.5)	PIDAA (Control)	30	24.6 (6.5)
PIDAA + 2.5 % NA	60	32.8 (6.1)	PIDAA + 1.25 % NA	60	28.1 (7.0)
PIDAA + 2.5 % NA	30	28.0 (6.5)	PIDAA + 1.25 % NA	30	27.3 (11.1)

A two-way ANOVA ( $\alpha=0.05$ ) indicated that there was no statistically significant interaction between application time and NA concentration ( $p=0.4$  for enamel and  $p=0.74$  for dentin). SEM analysis of enamel treated with PIDAA primers showed that effective surface etching occurred. **Conclusion:** Simplified dental bonding systems, equally effective on both dentin and enamel, are feasible with acid-modified PIDAA. Support: NIDCR-NIST Interagency Agree. Y1-DE-7006-0 and the ADAHF.

**114** Effects of pH of Single-Bottle Adhesive on Shear Bond Strength. B.I. SUH, M.Y. SCHILTZ\*. (Bisco, Inc., Schaumburg, IL).

**The purpose** of this study is to evaluate the effect of the adhesive pH on shear bond strength (SBS) of a light-cured (L/C) (BISFIL™) and a self-cured (S/C) composite (BISFIL 2B). The acidic monomer (SBA-HEMA) was used to modify the pH of the single-bottle adhesive, ONE-STEP<sup>®</sup>. **Method:** The pH was measured using an Orion pH meter and NIST standards in EtOH-H<sub>2</sub>O (70:30) solution. A 2g of sample was dissolved in 3g of solution. Reading was taken 5min. after mixing. The specimens for SBS were prepared in usual manner using gel cap#5 (dia. 4.6mm) for composite post and kept in 37 °C de-ionized water for 2 hours prior to breaking by Instron at the speed of 5mm/min. **Results:** A decrease of SBS occurs at a concentration of 1.2% (pH=2.94)

% SBA-HEMA	pH	SBS (MPa) n=5		% SBA-HEMA	pH	SBS (MPa) n=5	
		L/C	S/C			L/C	S/C
0	4.55	20.1(2.8)*	25.8(1.7)*	1.1	3.43	23.1(4.0)*	22.4(3.2)*
1	3.70	23.4(3.9)*	20.5(3.2)*	1.2	2.94	24.4(2.8)*	13.9(2.0)*
2	2.28	20.0(3.0)*	7.8(1.4)*	1.7	2.48	20.9(4.1)*	13.1(3.8)*
3	2.01	12.2(4.0)*	6.0(2.7)*	1.9	2.45	21.4(5.7)*	11.7(3.7)*

*Groups with same letter have no statistically significant difference.*  
SBA-HEMA for S/C composite. For L/C composite, the decrease occurs between 2-3% (pH=2.28-2.01). **Conclusion:** The SBS is affected by the pH of single-bottle adhesives. The SBS of L/C composite is less affected by the pH of adhesive than the S/C composite.

**115** Tensile Bond Strength of Clearfil Liner Bond 2V Using Different Application Techniques. J. D. LAFUENTE\*, (School of Dentistry, University of Costa Rica, Costa Rica)

The application technique of a dentin bonding system can affect the bond strength to dentin. In this study, a self-etch dentin adhesive (Clearfil Liner Bond 2V) was applied to dentin using different techniques. Thirty recently extracted, caries free human molars were embedded in acrylic resin and polished with 600 grit SiC paper until superficial dentin was exposed. Specimens were divided into three groups (n=10). All specimens received CFLB2V before receiving Z-100 composite. In the first group, the primer and the adhesive were brushed, five specimens were etched with 35% phosphoric acid prior to DBS application and the other five were not. The second group was divided as the first, into two subgroups, both primer and adhesive were brushed over the surface, but the adhesive was air-thinned before being light-cured. The third group, divided into two subgroups as before had the primer and the adhesive rubbed over the surface. After dentin bonding was applied, composite was cured over the dentin in two increments each one cured for 40 seconds to form an inverted cone. Specimens were stored in 100% relative humidity, at 37 °C for 24 hours before being tested in tensile at an Universal Testing Machine (Instron 1000) at a crosshead speed of 0.1 cm/min. Results listed in table. Bond strength in MPa

Group	With acid etching		Without acid etching	
	Mean	SD	Mean	SD
Group 1	17.2	4.1	6.1	2.1
Group 2	10.0	3.5	14.3	4.2
Group 3	19.2	4.2	14.2	3.8

was calculated and data were compared using a two-way ANOVA at a 0.05 significance level. Tukey-Kramer intervals for comparisons of means, calculated also at a 0.05 significance level, were 4.2 for comparisons among groups and 2.9 for comparisons between groups etched and not etched. **It dentin is etched is better to rub the DBS components, if not air-thin the adhesive to obtain better bond strength. The way the DBS components are applied affect the bond strength.**

**116** Effect of delayed activation of light-cured composites on all-in-one adhesives BI SUH\*, FR TAY<sup>2</sup>, NM KING<sup>2</sup>, DH PASHLEY<sup>3</sup> (Bisco, Inc., IL, USA; <sup>2</sup>The University of Hong Kong, HK SAR, China; <sup>3</sup>Medical College of Georgia, GA, USA)

This study examined the effect of delayed activation of light-cured composites on the microtensile bond strengths of two all-in-one adhesives to sound dentin. Flat dentin surfaces prepared from caries-free third molars were bonded with either Prompt L-Pop (PLP; ESPE) or One-Up Bond F (OUB; Tokuyama Corp.). Each adhesive was divided into 5 subgroups each containing three teeth. A hybrid composite was left to contact the cured adhesive surfaces for 0, 2.5, 5, 10 or 20 min before light-activation. Composite-dentin beams, with average cross-sectional areas of 0.85 mm<sup>2</sup> were prepared for bond testing. Fractured beams were prepared for SEM examination. Additional unfractured specimens were prepared in the 0 and 20 min-delay subgroups for TEM examination. **Results:** Kruskal-Wallis ANOVA and Dunn's tests indicated significant differences among the subgroups of each adhesive. Regression analyses showed that for each adhesive, there was an exponential decline in mean bond strength with increasing time-delay in light-activation. The correlation was highly significant ( $r = -0.99$  for PLP;  $r = -0.96$  for OUB). Voids were consistently observed in the 10 min- and 20 min-delay subgroups on SEM examination. They were located exclusively along the adhesive-composite interfaces. They corresponded to soap-sud-like blisters that were observed with TEM in the 20 min-delay subgroups. It is concluded that adverse interactions between acidic resin monomers and some components of light-cured composites can occur on prolonged contact with the cured adhesive layer. (Supported by grant DE06427, NIDCR)

**117** Solvent-induced Shrinkage of Moist Demineralized Dentin. DH PASHLEY\*, KA AGEE, FJ HARMON, RM CARVALHO, FR TAY, M NAKAJIMA, KW LEE (Medical College of Georgia, Augusta, GA, USA).

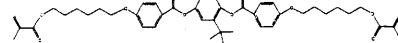
The purpose of this study was to test the null hypothesis that there are no differences among anhydrous polar solvents in their ability to shrink moist demineralized dentin. Thin (0.2 mm) disks of dentin were prepared from mid-coronal dentin of extracted human third molars. After complete demineralization in 0.5 M EDTA (pH 7), the specimens were placed in the well of a linear variable differential transformer (LVDT) contact probe that measured changes in the height of the matrix during application of the neat polar test solvents. Changes in height produced by solvents were compared by ANOVA and post-hoc multiple comparisons at  $\alpha = 0.05$ . The results demonstrated that the matrix shrank up to 25-30% depending upon the polar solvent that was applied. Pure acetone, propanol and HEMA produced maximal shrinkage. More polar solvents (methanol, formamide, ethylene glycol) induced smaller shrinkages that recovered by partial re-expansion. When the H-bonding ability of the solvents were ranked by their Hansen's solubility parameter for H-bonding ( $\delta_b$ ), a highly significant ( $p < 0.01$ ) positive correlation was obtained between matrix height and solvent  $\delta_b$ . **The use of polar solvents with high  $\delta_b$  prevents collapse of the matrix, while polar solvents that do not H-bond well (acetone, propanol, HEMA) cause partial collapse of the matrix. Methanol, formamide and ethylene glycol produced the least amount of shrinkage. Methanol may be a superior solvent to those currently employed. The null hypothesis was rejected. Supported, in part, by DE06427, from NIDCR.**

**118** Bonding to Moist Dentin. A.J. WAHL<sup>1</sup>, E.C. COMBE<sup>1</sup>, A. ABOUSHALA<sup>2\*</sup> (University of Minnesota, Minneapolis, Minnesota<sup>1</sup>, and Tufts University School of Dental Medicine, Boston, Massachusetts<sup>2</sup>)

Dentin bonding involves application of an acid etchant, which is used to remove the smear layer, open and widen dentinal tubules, and demineralize intertubular dentin. The etchant is washed off, leaving the surface "moist" prior to application of the one-step adhesive and subsequent restorative material. However, it is often difficult for the operator to determine "how moist is moist?" This work was undertaken in attempts to develop a dentin bonding technique that is fast, reproducible and controllable. Specifically the aims of this study were to verify that desiccation of dentin is deleterious to bond strength, and to explore experimental variables in the rewetting of desiccated dentin. The teeth used in this study were unerupted human third molars stored in isotonic saline for no more than 3 weeks after surgical extraction. Single Bond Adhesive (3M Dental Products, St Paul, MN) was used to bond Restorative Z100 (3M Dental Products, St Paul, MN). There were four experimental groups, and 10 measurements per group: (1) Dentin wet after washing (control), (2) Desiccated dentin, (3) Dentin rewetted with water, (4) Dentin rewetted with a solution of glycerol, a humectant, in ethanol and water. Shear bond strength data (MPa) were: Group (1), 21.3±4.1; Group (2), 6.2±1.6; Group (3), 21.2±4.4; Group (4), 19.1±4.4. Statistical analysis (ANOVA) showed that Group (2) had significantly lower bond strength than the other three groups ( $p < 0.001$ ). **It was concluded that desiccation of dentin reduces bond strength, but dried dentin can be rewetted with water or a humectant solution to give acceptable bond strength.** Supported by NIH/NIDCR 2 T35 DE07098-21.

**119** Cure shrinkage of experimental LC monomer based composite resins. B.K. NORLING\*, N. SATSANGI and B.R. Furman. (University of Texas Health Science Center, San Antonio, TX 78229-3900)

Cure (polymerization) shrinkage remains a major barrier to universal use of resin restoratives in large posterior cavity preparations. We have developed a new monomer system structurally related to that which we have reported before, shown here



The purpose of this study is to determine the cure shrinkages of a new experimental liquid crystal (LC) monomer system unfilled and 80% filled with 1.5 μm fused silica (Si1) both freshly (F) silanated (<3 days old) and after aging (A) (> 5 years). Specimens of 0.08 to 0.12 g of resin were placed in a NIST Paffenbarger Research Institute mercury dilatometer and shrinkage followed for 90 min after a 120 sec visible light exposure. The unfilled LC monomer (LCU) was first blended with camphorquinone/DMAEMA initiator and ca. 30 wt. percent unsaturated Aerosil OX50 for rheology, although shrinkage was based on neat monomer. The resultant volumetric cure shrinkages are (mean ± s.d.): -LCU (4.2 ± 0.3), Si1-F (1.3 ± 0.1), Si1-A (0.84 ± 0.05). Student Newman Keuls post-hoc analysis at the 0.05 level placed each group in a statistical subset. **The liquid crystal monomer yielded a cure shrinkage by itself which is about twice that of another LC monomer (2.0 ± 0.4) which we have previously reported. Even so, the Si1-A composite shrinkage is lower than that of any commercial resin composite which we have so far measured. The reason for the significant difference between the cure shrinkages of the aged and un-aged fillers deserves further investigation.** Supported by NIH-NIDR Grant P01DE1668.

**120** Shrinkage of Oxirane-Polyol and Conventional Composites versus Time Post-Irradiation. M.G. REIMAN, S.M. ROZZI\*, S.C. WILLIAMS (3M Company, St. Paul, Minnesota, USA)

Cationic curing oxirane-polyol resins have been shown to produce lower polymerization shrinkage than free radical curing methacrylate resins when measured at relatively short times after irradiation (i.e., 1 hour) (Eick 1999; Tilbrook 2000). The objective of the present study was to assess the polymerization shrinkage of oxirane-polyol and methacrylate dental composites at both short (one hour) and long times post-irradiation (24 hours and one month). The rationale for the study was that cationic curing polymerization is known to be a long living reaction especially in comparison to free radical polymerization. The oxirane-polyol composite contained resins diglycidyl ether of Bisphenol F, 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexene carboxylate, and polytetrahydrofuran. The methacrylate composites tested were Filtek™ Z250, Surefil™, Prodigy™ C, and Tetric Ceram™. Dilatometry was used to characterize the shrinkage up to one hour after irradiation with a halogen curing light. From one hour after irradiation, shrinkage was determined by density measurements on the cured composites. Measurements were made on samples stored both wet and dry at 37°C. At one hour, the shrinkage of the composites in descending order was (N=3): Prodigy C 3.43% (SD 0.19), Tetric Ceram 2.84% (SD 0.05), Surefil 2.70% (SD 0.14), Z250 2.05% (SD 0.07), and oxirane-polyol 1.08% (SD 0.07). After one month in water, Z250 was 2.70% (SD 0.20) and the oxirane-polyol was 1.20% (SD 0.09). **The results of this study show that despite the living polymerization in cationic oxirane-polyol composites, they still exhibit more than a 50% reduction in shrinkage compared to methacrylate composites.** smrozzi@mmm.com