



Title	Chemical-cured composite weakens bonding of adhesives by surface interaction
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1697 Effect of Phosphoric Acid Concentration on Enamel Bonding. M.J. SHINCHI*, K. SOMA, N. NAKABAYASHI (Tokyo Med Dent Univ, Tokyo, Japan).

It was hypothesized that the decreased concentration of phosphoric acid for etching could minimize adverse effect of enamel treatment. It would require choosing the adhesive having high potential to promote monomer diffusion into the etched enamel. The purpose of this study was to demonstrate the relationship between depth of penetration and tensile bond strength of a photo-cured resin to phosphoric acid etched enamel and the efficacy of enamel etchants that were less aggressive than a concentration of 10% H₃PO₄. The tensile bond strength and length of tags produced by a photo-cured (20s) resin consisting of pre-polymerized TMPT/silica in 3% HNPM-TEGDMA on acid-etched enamel was determined. The enamel etchants tested were various concentrations (3% to 65%) of phosphoric acid. The resin was applied to enamel samples that had been abraded to #600-grit SiC paper and acid etched (30s) to create test specimens that were loaded to fracture on a testing device. The HCl-treated then fractured specimens were examined under SEM and light microscopy. The tensile bond strength (10MPa) of resin to enamel pre-treated with various acid concentrations did not vary significantly (p>0.01). But resin tag length decreased significantly from 22µm for 35% H₃PO₄ to 12µm for 20% H₃PO₄ to 9µm for 5%, 10% and 5% H₃PO₄ to 5µm for 3% H₃PO₄ (p<0.01). The length of the tags contributes little to TBSs, and TBSs to H₃PO₄ etched enamel are mainly attributable to the resin's ability to penetrate between the enamel crystallites and rods. Further, enamel pre-treatment by phosphoric acid etchants of concentrations lower than 10% may be satisfactorily employed. The use of less aggressive acid concentrations might minimize any potential adverse effects to enamel substrates. This study was supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science, Culture and Sports, Japan (No.10877335).

1698 A Comparison of Fifth Generation Bonding Agent Adhesive Properties. T.J. HILTON*, W. JIA, and J.L. FERRACANE (OHSU School of Dentistry, Portland OR, USA)

The purpose of this study was to determine the shear bond strength and microleakage of various fifth generation bonding agents. The buccal and lingual enamel of 40 recently extracted human molars was removed, the resulting dentin surface prepared with 600 grit sandpaper, and teeth randomly divided into 4 groups (n=10). Group A: Prime and Bond NT (Dentsply); Group B: Single Bond (3M); Grp C: Optibond Solo Plus (Kerr); Grp D: Excite (Vivadent). Bonding agents were applied according to manufacturer instructions. Composite (Prodigy, Kerr) was applied in two-2mm increments using a single plane shear testing device (Watanabe jig), cured each for 40 sec, stored in 37°C water for 24 hours, and tested in shear on a universal testing machine (crosshead=5.0mm/min). Class 5 cavities (5 X X 2mm deep, n=5) were prepared at the CEJ with a 1mm enamel bevel. Bonding agents were applied as above, preparations filled in 2 increments, immediately finished, thermocycled 400 X (5/55°C), soaked in silver nitrate, sectioned mid-restoration, and highest score used. Leakage at enamel and dentin margins were scored separately. Results were as follows:

Group	SBS-MPa (SD)	Leakage-EN (KW rank)	Leakage-DE	ANOVA/Scheffe's applied to
A	13.4 (3.1)	5.0	9.2	SBS, Kruskal Wallis/Wilcoxon
B	14.8 (2.5)	11.4	9.5	Sign Rank to leakage; Asterisk
C	*20.1 (4.0)	16.4	8.0	shows statistical differences.
D	12.0 (3.7)	9.2	15.3	Group C had significantly higher SBS than all other groups. Dentin margin leakage showed an inverse trend, while enamel margin leakage showed a direct trend with SBS. This study was supported by Kerr, Inc.

1699 N-acryloyl Amino Acids with Mono- or Di-carboxyl Groups as Conditioner/primer. K. ITOU*, Y. TORII, K. ISHIKAWA, K. SUZUKI and K. INOUE (Depts. of Operative Dentistry and Biomaterials, Okavama University Dental School, Okavama, Japan)

This study investigated the effect of self-etching primers containing N-acryloyl amino acids with one or two carboxyl groups on dentin adhesion. N-acryloyl aspartic acid (N-AAsp) was synthesized according to our previous study. N-acryloyl glycine (N-AGly) was newly synthesized by a reaction of acryloyl chloride and glycine. The aqueous solutions containing 1.2 mol of each of N-AAsp or N-AGly were prepared as self-etching primers. The pH of both primers were approximately 1.7. Flat bovine dentin surfaces were treated with the primer for 60 s, air-dried without rinsing and applied with a bonding resin (Clearfil Photo Bond, Kuraray, Japan) prior to filling of a resin composite (Clearfil AP-X, Kuraray). Tensile bond strength (TBS) was measured after 24-hour storage in water (n=10, cross-head speed=2mm/min). Resin-dentin interfaces were also observed with SEM. TBS in N-AAsp group (14.7±4.5MPa) was significantly higher than those in N-AGly group (9.0±2.6MPa), non-etching group (6.9±2.8MPa) or phosphoric acid etching/water rinse group (7.6±2.8MPa) (one-way ANOVA & Fisher's PLSD method, p<0.05). The hybrid layers with thickness of approximately 1.5µm were formed in both specimens treated with N-AAsp and N-AGly primers. Our results indicate that the self-etching primer containing N-acryloyl amino acid, which has two carboxyl groups in the molecule, is able to promote the adhesion between resin and dentin more effectively than that containing mono-carboxyl acid monomer, although both primers facilitate hybrid layer formation at resin-dentin interfaces.

1700 Chemical-cured Composite Weakens Bonding of Adhesives by Surface Interaction. A. SANARES*, A. ITTHAGARUN, N.M. KING, F.R. TAY¹ and D.H. PASHLEY² (¹University of Hong Kong, HKSAR; ²Medical College of Georgia, USA)

This study examined the effect of types of adhesives and composite curing modes on bonding to sound dentin. Dentin surfaces were prepared from 24 extracted human molars. After conditioning, they were bonded with one of four adhesives: Prime & Bond NT (PB), Optibond Solo (OP), Single Bond (SB) or One-Step (OS). Composite buildups were made with either a light-cured (LC) composite (Z-100) or a chemical-cured (CC) composite (BisFil 2B). After storing in water for 1 d, the teeth were vertically sectioned into 0.9 x 0.9 mm composite-dentin beams for evaluation of microtensile bond strength (µTBS). After classification of the failure modes with optical microscopy, beams from each group were prepared for SEM examination. The pH of the adhesives were also measured. Results:

µTBS(MPa)	PB	OP	SB	OS
Light-cured	58.80±13.33 ^a	58.11±8.50 ^a	60.75±12.03 ^a	60.66±11.56 ^a
Chem-cured	5.43±9.96 ^b	9.64±5.07 ^b	12.39±9.81 ^b	33.08±12.35 ^b
Adhesive pH	2.68	2.81	3.60	4.60

A two-way ANOVA showed that the effect of curing mode was significant (p<0.001). SNK test showed that µTBS of the four adhesives, were not significantly different with the LC composite (p>0.05) but they were significantly different when used with the CC composite (p<0.01). Interfacial failure occurred along the composite-adhesive interface, with porosities evident on the surface of the adhesives, as well as voids present within the composite. It is concluded that decreased bond strengths could be caused by air incorporated as well as surface interaction of acidic adhesive monomers with catalyst components in the CC composite. (Supported by RGC Grants 10202534 HKU, and DE06427, NIDCR)

1701 Effect of deproteinization on Shear Bond Strength and Microleakage. FS. AGUILERA, R. OSORIO*, M. TOLEDANO. (University of Granada, Spain).

The purpose of this study was to determine the shear bond strength (SBS) and Microleakage (ML) of Syntac Sprint (Vivadent), using PO₄H₃ as conditioner of dentin versus PO₄H₃ followed by 5% NaOCl. For SBS, 20 third molars were used. Superficial and deep dentin was exposed by occlusal parallel sections. SBS was determined in a universal testing machine by the Watanabe technique (J Dent Res 1994). Obtained data were statistically analyzed by Student t test. For ML, 12 third molars for standardized Class V cavities were used. Cavities were treated with PO₄H₃ or PO₄H₃+NaOCl. Syntac Sprint bonding system and Tetric resin composite were used for restorations. The specimens were polished and stored in water for 24 hours at 37 °C and thermocycled (500x). After immersion in 0.5 basic fuchsin for 24 hours, dye penetration was measured according to an ordinal scale. Data were analyzed by Mann-Whitney U test.

SBS	Superficial Dentin				Deep Dentin			
	PO ₄ H ₃	PO ₄ H ₃	+NaOCl		PO ₄ H ₃	PO ₄ H ₃	+NaOCl	
MPa (SD)	5.08 (3.17)	5.45 (3.15)			6.43 (4.6)	5.62 (1.24)		

No differences were found between groups (p>0.05).

Dentin treatment	Occlusal margin ML				Gingival margin ML			
	0	1	2	3	0	1	2	3
PO ₄ H ₃	8	3	1		12			12
PO ₄ H ₃ +NaOCl	3	1	2	12			12	12

The results suggest that deproteinization did not affect mean SBS or ML values when Syntac Sprint was used as dental adhesive. Dentin depth did not affect SBS values. Occlusal microleakage values were always lower than gingival ones. (Supported by Spanish Government Grant #MA198-0937-C02).

1702 Effect of smear layers on bonding of a self-etching primer to dentin. FR Tay¹, H Sano², RM Carvalho³, DH Pashley¹ (¹University of Hong Kong, HKSAR; ²Hokaido University, Japan; ³University of São Paulo, Brazil; ⁴Medical College of Georgia, USA)

This work evaluated the effect of the absence and presence of smear layers on bonds made to dentin using a self-etching primer system, Clearfil Mega Bond (Kuraray). Dentin surfaces with different smear layer thickness were created from mid-coronal sound dentin in extracted, human third molars. For the control group, the middle dentin surface was cryofractured to create a bonding surface that was devoid of a smear layer. The experimental teeth were ground with wet 60, 180 or 600 grit SiC abrasive paper. They were bonded using Clearfil Mega Bond, followed by resin composite buildups. After 1 d, bonded specimens were sectioned into an array of 0.9 x 0.9 mm beams, with the top half in composite and the bottom half composed of dentin. Microtensile bond strengths were determined and the results analyzed by ANOVA and Student-Newman-Keuls test. Fractured beam surfaces were examined with SEM on selected specimens after failure. Fractographic study of cross sections of failed interfaces from the dentine side of representative beams was performed using TEM. Clearfil Mega Bond produced high bond strengths (ca. 50 MPa) to both smear layer-free and smear layer-covered dentin. There was no consistent difference among the fractured and smear layer-free covered dentin. SEM examination was inadequate to define the exact nature of interfacial failures. TEM observations demonstrated a thin hybrid layer on the fractured dentin and thicker (ca. 1-4 µm) hybrid layers on smear-layer covered dentin. This included a thick hybridized smear layer and a thin, underlying authentic hybrid layer in the intact dentin. Separation of the two hybrid layers was not evident in interfacial failures and dentinal tubules were still sealed with hybridized smear plugs. Partial cohesive failure within the hybridized smear layer was occasionally observed in to-60 grit group but was caused by secondary bifurcation cracks that branched out from the primary crack plane. It is concluded that both hybrid layers may function as a unit during loading without separation. Occurrence of secondary bifurcation cracks exemplifies the complex reaction to tensile stresses in multi-layered biological joint systems that comprise materials of variable compliance. (Supported, in part, by DE06427, NIDCR)

1703 Evaluation of Dentin Bonding by CLSM and SBS Measurement. S.J. Park*, W.K. Yang, B.H. Cho, H.H. Son. (Department of Conservative Dentistry, Seoul National University, Seoul, Korea).

To evaluate the relationships between the hybrid layer thickness and the bond strength in different dentin bonding systems (DBS), the Confocal Laser Scanning Microscopic (CLSM) images of the resin-dentin interfaces and the shear bond strengths (SBS) of two DBS (All bond 2 (AB2) and One Step (OS)) were investigated. Thirty-six extracted human third molars were randomly divided into two groups of eighteen teeth each. In six teeth of each group, notch-shaped class V cavities (depth of 1.5mm) were prepared on the buccal and lingual surfaces at the cemento-enamel junction (twelve cavities per group). Bonding resins of each DBS were mixed with rhodamine B and primer of AB2 was mixed with fluorescein. Prepared teeth of each group were treated with one of AB2 and OS, according to the manufacturer's instructions. The treated teeth were then packed with composite resin (Elitefil) and light-cured. Each specimen was sectioned longitudinally with diamond saw and observed with CLSM. For the remaining twelve teeth of each group, the SBS's of these systems to the occlusal dentin of the freshly extracted third molars were measured by the regulation of the ISO TR 11405. Results: The hybrid layer thicknesses of AB2 and OS were 2.59 ± 0.47 µm and 2.28 ± 0.53 µm, respectively. The SBS's of AB2 and OS were 14.87 ± 1.74 MPa and 12.41 ± 1.63 MPa, respectively. There was no statistically significant difference between the hybrid layer thicknesses of the two groups (p>0.05). But the SBS of AB2 was statistically significantly higher than that of OS (p<0.05). These results revealed that the hybrid layer thickness could not be a parameter of the shear bond strength of dentin bonding system.

1704 Shear Bond Strength to Enamel & Dentin using Three One-Bottle Filled Adhesives. C.A. MUNOZ, J.R. DUNN, N. JESSOP, J.SY-MUNOZ* Loma Linda University, Loma Linda, CA, USA

This study evaluated the shear bond strength of three recently introduced dentin adhesive systems, Excite (Ivoclar), Single-Bond (3M) and Prime & Bond NT (L.D. Caulk) at three time periods 30 min, 24 hrs and 1 week with 2000 thermocycles. Extracted human molars were ground with 600-grit SiC paper to expose the enamel or dentin surfaces and assigned to 12 groups (n=10). The adhesive systems were applied following recommended manufacturer's directions, placed in a bonding jig, cured and their respective restorative material placed, cured, and stored in 37 °C water. A load was applied to the specimen at a crosshead speed of 1.0 mm/min and the shear bond strength calculated and expressed in MPa (SD). Results were analyzed with ANOVA and Newman Keuls used to identify any differences (p>0.05). Means with same letters are statistically the same.

	30 minutes		24 hours		One week/Thermocycled	
	Enamel	Dentin	Enamel	Dentin	Enamel	Dentin
Excite	26.32 (4.9)a	41.32 (6.8)a	33.31 (7.4)a	49.82 (4.6)a	30.35 (5.2)a	52.07 (5.3)a
Single-Bond	32.33 (4.2)ab	38.47 (4.3)ab	34.90 (4.7)a	51.70 (5.2)a	32.19 (2.8)a	48.03 (3.9)a
P&B NT	31.66 (4.6)b	34.33 (5.8)b	40.07 (11.8)a	34.81 (9.5)b	41.58 (10.4)b	42.23 (7.5)b

Excite and Single-Bond bonded to enamel and dentin had similar bond strengths at all three time periods and were not statistically different. P&B NT had higher enamel bond strengths but were only statistically different at one week and when bonded to dentin it had lower bond strengths but was not statistically different at 30 min. and one week. All three dentin adhesives exhibited excellent dentin and enamel shear bond strengths.