Bond strength of self-etching primers to enamel and dentin of primary teeth

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Abstract

***Purpose**: The objective of this in vitro study was to evaluate the tensile bond strength of three self-etching primers to human primary enamel and dentin.*

***Methods**: Forty (40) freshly extracted primary molars were sectioned bucco-lingually and embedded in self-curing acrylic resin with the facial or lingual surfaces exposed. The specimens were wet ground to 600 grit SiC paper to expose a flat enamel or dentin surface. The materials tested were: Prompt L-Pop (LP2, ESPE), Clearfil SE Bond (CSE, Kuraray America), Etch and Prime 3.0 (EP, Degussa) and a control, Prime and Bond NT (NT, Dentsply/Caulk). The adhesive systems were applied according to the manufacturers' instructions. An inverted, truncated cone of composite (Pertac II, ESPE) with a 2-mm bonding diameter was cured using a polytetrafluoroethylene jig. The specimens were debonded in tension using a universal testing machine (Instron) at a crosshead speed of 0.5 mm/min.*

***Results:** Analysis of variance (ANOVA) showed significant differences existed between the four systems and two surfaces. To enamel of primary teeth, Prime and Bond NT had significantly higher bond strength (25.9 MPa) than when bonding with the three acidic primers Prompt L-Pop (18.5 MPa), Etch and Prime 3.0 (19.3 MPa) and Clearfil SE Bond (18.7 MPa). Complete bond failures occurred with Prompt L-Pop and Etch and Prime 3.0 to dentin of primary teeth. With a mean of 39 Mpa, the bond strength to dentin of primary teeth with Clearfil SE Bond was significantly higher than with Prime and Bond NT (12.5 MPa).*

***Conclusions**: The results of this in vitro study show that the four adhesive systems tested bonded effectively to enamel of primary teeth, but only CSE achieved adequate bond strengths to dentin of primary teeth. (Pediatr Dent 23:481-486, 2001)*

Resin composite is increasingly the material of choice for the restoration of primary teeth and new materials with simplified procedures are increasingly being offered. The majority of the newer bonding agents are hydrophilic and capable of forming a hybrid layer between resin and dentin. The bonding mechanism is based on the combined effect of hybridization and formation of resin tags.[1-3] Hydrophilic adhesives have been marketed in the form of multi-bottle systems, with separate conditioner, primer and bonding resin agents, and with primer and bonding agents together. A recent development involves the use of acidic or self-etching primers which combine acid conditioning with the priming procedure.[4] Self-etching primers were developed to simplify the bonding procedure and are based on the use of non-rinsed acidic polymerizable monomers which serve as conditioner, primer and resin.[5]

Apart from simplification, the rationale behind these systems is to superficially demineralize dentin and simultaneously penetrate it with monomers, which can be polymerized *in situ*. While bonding to permanent teeth has been studied extensively, few studies have addressed resin bonding to primary teeth.[6-12] When comparing the same adhesive systems, Fagan et al reported no statistically significant difference between dentin bond strengths of primary and permanent dentin.[6] While Hosoya et al achieved higher bond strength in dentin of primary teeth, others reported significantly lower bond strength in primary teeth compared to permanent teeth.[7-12]

Chemical, physiological and micromorphological differences between primary and permanent teeth are thought to be responsible for lower bond strength.[13-15] Based on a SEM comparison of the resin-dentin interface in permanent and primary teeth, Nör et al observed that dentin of primary teeth was more reactive to acidic conditioners than permanent dentin.[16] Therefore, they suggested that a different protocol with shorter acid conditioning times be used when bonding to primary teeth.[17] Currently, the same protocol is being recommended by the manufacturers for bonding to primary and permanent teeth. The purpose of this in vitro study was to evaluate the tensile bond strength of three new self-etching primers to enamel and dentin of primary teeth.

Methods

Forty non-carious primary molars were collected and stored in a physiologic saline solution containing 0.20% sodium azide for no longer than 6 months. The teeth were sectioned bucco-lingually, placed in a 15-mm diameter PVC ring and embedded in self-curing acrylic resin with the buccal or lingual surfaces positioned for surface treatment and composite bonding. The surface of the teeth was ground flat with a series of silicon carbide paper ending with the 600 grit used on a polisher (Ecomet Grinder- Polisher Buehler Ldt, Lake Bluff, IL) to obtain flat

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Table 1. Materials with Batch Number, Composition, and Manufacturers

Adhesive system	Characteristic	Main components	Batch number	Code
Prime and Bond NT Dentsply/Caulk	Primer and adhesive with phosphoric acid	Di- und tri-methacrylates, PENTA, nanofillers, photoinitiators, stabilizers, acetylamine-hydrofluoride, aceton	P&B NT: 990930 34% conditioner: 990907 Adhesive: 990920	PB
Clearfil SE Bond Kuraray Dental	self-etching	*Primer:* MDP,HEMA, Hydrophilic dimethacrylate,N,N-Diethanol-p-toluidine, water	CSE BOND:61122 Primer: 00109A Bonding: 00043A	CSE
		Bonding: MDP,Bis-GMA,HEMA hydrophobic dimethacrylate, dl-Campherquinone, N,N-Diethanol-p-toluidine, silanated silicate		
Prompt-L-Pop 2 ESPE	Self-etching all-in-one system	*Compartment1:* Methacrylated phosphoric esters, initiators, stabilizers.	FW0058402	LP2
		Compartment 2: water, fluoride complex, stabilizers		
		Compartment 3: microbrush		
Etch and Prime 3.0 Degussa	Self-etching	*Catalyst*: Tetra-methacryloxy-ethylpyrophosphate, HEMA, initiators, stabilizers	E&P 3.0: 1998001 Catalyst: 019921 Universal: 099812	EP
		Universal: HEMA, ethanol, distilled water, stabilizers		

MDP = 10-Methacryloyloxydecyl-dihydrogenphosphate, HEMA= Hydroxyethyl-Methacrylate, Bis-GMA = Bis-phenol-A-glycidyl Methacrylate , PENTA = Di-Pentaerytrithol-Penta-Acrylate-Monophosphate

Fig 1. Jig and composite resin cone on embedded tooth specimen

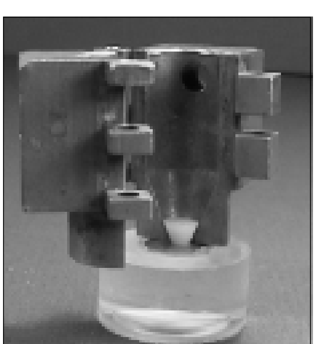

Fig 2. Loading jig for tensile bond strength test

enamel (n=40) or dentin surfaces (n=40). A polytetrafluoroethylane jig was placed over each of the specimens to limit and standardize the area available for bonding to 2 mm in diameter. For each type of surface, the specimens were randomly divided into four adhesive treatment groups of 10 specimens each. The following four commercial adhesive systems were used in this study and applied as recommended by the manufacturers: Prime and Bond NT (Dentsply/Caulk) is based on the acrylic PENTA and, unlike true self-etching primers, requires the etching of enamel as a separate step; Clearfil SE Bond (Kuraray Dental) and Etch and Prime 3.0 (Degussa) are two-step self-etching systems; and Prompt L-Pop 2 (ESPE) is a true all-in-one self-etching system. The composition of the materials used and the batch numbers are listed in Table 1.

To exclude possible influences of different restorative resins on the adhesives performance, all bonding agents were used in combination with the light-cured resin composite (Pertac II, ESPE). Specifically, the following bonding protocols were used for 10 enamel and 10 dentin specimens:

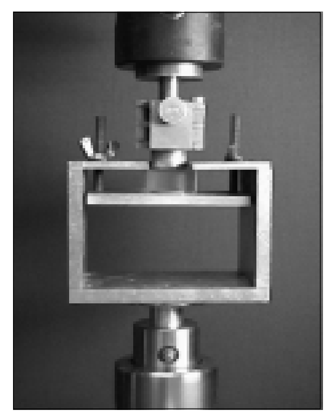

Fig 3. Specimen attached to Instron machine with a self-aligning system for tensile bond strength test

Group 1: Prime & Bond NT (Control): Phosphoric acid was applied for 15 seconds and rinsed with water for 10 seconds. Excess water was blotted dry and the primer and bonding materials were applied and dried with oil-free compressed air for 5 seconds. Polymerization for 10 seconds followed.

Group 2: Clearfil SE Bond: The acidic primer was applied for 20 seconds and dried with a thin air stream. The bonding agent was then applied and thinned with a gentle stream of air followed by a 10-second polymerization.

Group 3: Prompt L-Pop: The material is packaged in a foil pack with three distinct, sealed compartments. The material was activated as described by the manufacturer and applied onto the tooth surfaces with a saturated microbrush and rubbed in for 15 seconds. A thin air stream was then applied to create an even, shiny film and was followed by a 10-second polymerization.

Table 2. Analysis of Variance for Tensile Bond Strength					
Source	Df	Sum of squares	Mean square	F-value	P-value
Tooth	1	1197	1197	56.2	0.0001
Product	3	5186	1728	81.1	0.0001
Tooth * Product	3	5315	1771	83.2	0.0001
Residual	72	1533	21.3		

Dependent: Bond strength, MPa

Table 3. Tensile Bond Strength (MPa) to Primary Enamel and Dentin		
Product	Enamel	Dentin
Prime and Bond NT	25.9 (6.9)*	12.8 (1.5)
Clearfil SE Bond	18.8 (4.0)	39.0 (8.5)
Prompt L-Pop 2	18.6 (4.1)	0.0 (0.0)
Etch and Prime	19.4 (3.7)	0.0 (0.0)

*Mean bond strength with standard deviation in parentheses (n = 10) are listed in MPa. Turkey-Kramer intervals for comparisons of means at the 0.05 significance level among products and tooth surfaces were 3.8 and 2.0 MPa, respectively

Group 4: Etch and Prime 3.0: As recommended by the manufacturer, the double-coating technique was used. An equal amount of primer and adhesive were mixed and applied to the surfaces for 30 seconds. The surfaces were then dried for 5 seconds and polymerized for 10 seconds. The last three steps were repeated. After application of the bonding systems, the jig was used to create an inverted, truncated cone of resin composite directly onto all tooth surfaces (Figure1). This technique first described by Barakat and Powers was modified due to the smaller surfaces available for bonding in primary teeth.[18] The size of the cone was reduced from the original 3 mm diameter at the base to 2 mm. The composite was incrementally placed into the jig and each 2-mm increment was light cured for 40 seconds. The light source was placed directly in contact with the jig to ensure equal distance of the light source for all samples. Adequate and consistent curing light intensity was assured by monitoring the curing light unit output using a light meter.[19] The specimens were mounted on a loading jig and debonded under tension using a universal testing machine (Model 8501, Instron Corp., Canton, MA) at a crosshead speed of 0.5 mm/min (Figure 2). This self-aligning system allows for the forces to be applied perpendicular to the specimens. (Figure 3) Bond strength was recorded in Newtons and calculated in Mega-Pascal (MPa). Bond failure sites were observed visually under x 2 magnification to determine failure site.

Means and standard deviations (n=10) were calculated. Bond strength data were analyzed by two-way ANOVA (SuperANOVA, Abacus Concepts, Berkeley, CA, Table 2). Means were compared with a Tukey-Kramer interval (SuperANOVA) calculated at the 0.05 significance level. Differences between two means that were greater than the respective Tukey-Kramer interval were considered statistically significant. Bond failure sites were not analyzed statistically.

Results

Mean bond strengths and standard deviations are listed in Table 3. The ANOVA showed significant differences existed among the materials for enamel of primary teeth. Prime and Bond NT had significantly higher bond strength (25.9 MPa) than when bonding with the three acidic primers Prompt L-Pop (18.5 MPa), Etch and Prime 3.0 (19.3 MPa) and Clearfil SE Bond (18.7 MPa).

Complete bond failures occurred with Prompt L-Pop and Etch and Prime 3.0 to dentin of primary teeth. With a mean of 39 MPa the bond strength of Clearfil SE Bond to dentin of primary teeth was significantly higher than with Prime and Bond NT (12.5 MPa).

The observed fracture modes are listed in Table 4. The majority of specimens had adhesive fractures and mixed fractures.

Discussion

This study evaluated the tensile bond strength of four of the most contemporary bonding agents to enamel and dentin of primary teeth. All materials tested achieved adequate bond strength to primary enamel. It is generally accepted that in permanent teeth bond strengths of 17-24 MPa are required to effectively resist the polymerization contraction forces of composite resin.[20] Although the adhesive systems based on self-etching primers did achieve bond strength above the minimal required for permanent teeth, Prime and Bond NT, which requires a separate etching step, achieved significantly higher bond strength to enamel of primary teeth.

The prismless outer layer found in primary enamel is believed by some authors to reduce the effectiveness of acid etching and result in a shallower etching pattern. Recommendations for etching times for enamel of primary teeth with phosphoric acid have therefore been varied. Suggested etching times ranged from 15 seconds to 4 minutes, depending on the author.[21-26] However, a detailed etch pattern could be demonstrated with a 15-30 second application of 35% phosphoric acid, and based on these findings, a prolonged etching time for enamel of primary teeth was not recommended to avoid possible over-etching. [26-28]

Table 4. Locations of Bond Failures*		
Material	Enamel	Dentin
Prompt L-Pop	87Adhesive/13 Cohesive	100 Adhesive†
Clearfil SE Bond	63 Adhesive/37 Cohesive	71 Adhesive/29 Cohesive
Etch and Prime 3.0	40 Adhesive/60 Cohesive	100 Adhesive
Prime and Bond NT	71 Adhesive/29 Cohesive	100 Adhesive†

*Percent adhesive failures and cohesive failures within the composite are listed. Cohesive failures within the enamel or dentin were not observed.
† Failed before testing

Significantly higher bond strength was achieved for Prime and Bond NT to enamel of primary teeth compared to the three acidic primers evaluated in this study. Prime and Bond NT is based on the acrylic PENTA, which contains a hydrophilic and a hydrophobic group. The hydrophilic portion of the molecule is a phosphoric group, which resembles phosphoric acid. Similar to a self-etching primer, it is able to demineralize the dentin but the effect is not sufficient to etch enamel. The company therefore recommends the additional use of 34% phosphoric acid to etch the enamel as a separate step. The stronger bond strength of Prime and Bond NT is in agreement with Hosoya, who reported higher bond strength to primary enamel when using phosphoric acid compared to the self-etching primer Fluorobond.[29]

It should be noted that the grinding of the enamel specimens that was necessary to achieve a flat surface resulted in a negligent amount of prismless layer. This could, however, have an influence on the bond strength to uncut primary enamel in vivo.

Depending on the materials tested in this study, the bond strength to dentin of primary teeth ranged from complete bond failures (Etch and Prime 3.0 and Prompt L-Pop) to very high values (Clearfil SE Bond). With the exception of Clearfil SE Bond, bond strength for dentin of primary teeth for all materials tested were lower in comparison to the bond strength reported for the same materials to dentin of permanent teeth using the same methodology.[30] Due to the reduced mineral content of primary dentin compared to permanent dentin, a different effect of acid conditioning on primary dentin has been suggested as a possible explanation. Using 10% maleic and 10% phosphoric acid and two different bonding systems, Nör et al reported that the hybrid layer produced in primary teeth was 25-30% thicker then in permanent teeth and concluded that primary dentin was more reactive to acidic conditioners.[16] Olmez later confirmed these observations using 37% phosphoric acid.[31] The reasons for this phenomenon are not understood, but may be due to differences in chemical composition or micromorphologic characteristic between dentin of primary and permanent teeth.

Koutsi suggested that, since primary teeth have fewer dentinal tubules compared to permanent teeth, the acidic conditioners may not be diluted as readily.[15] However, a recent study demonstrated an increased number of dentinal tubules with a larger diameter in dentin of primary teeth compared to dentin of permanent teeth.[32] Since the penetration of acids occurs primarily along the tubules it could be possible that a larger number of tubules with a larger diameter could result in a deeper penetration of the acidic conditioner and therefore stronger demineralization.[33] The role of the microcanals or giant dentin tubules that have been observed in dentin of primary teeth in the bonding process is unclear, but they may also contribute to an additional reduction in bond strength.[32,34] It has been suggested that a shorter etching time for dentin of primary teeth might result in a thinner hybrid layer with more complete penetration of resin.[17]

Recently, effective conditioning of dentin of primary teeth with acidic conditioners has been reported.[29] In our study, the use of self-etching primers on dentin of primary teeth resulted in complete bond failures for Prompt L-Pop and Etch and Prime 3.0 and very high bond strength (39 MPa) for Clearfil SE Bond. The depth of dentin demineralization achieved with these self-etching primers might differ depending on the pH. Marshall indicated that the lower the pH of the conditioner the deeper the depth of demineralization of dentin.[34] According to a recently proposed categorization of adhesives by application modes, Etch and Prime 3.0 can be described as a two-step smear layer dissolving system with a strongly acidic primer (pH 0.6). Prompt L-Pop is a one-step smear layer modifying adhesive system with a pH of 1 and Clearfil SE Bond is a two-step smear layer modifying bonding system.[35] With the highest pH (2.0), Clearfil SE Bond achieved the highest bond strength to primary dentin. It is possible that the other two acidic primers evaluated in this study caused excessive demineralization of the dentin. The resulting increased thickness of the hybrid layer and the subsequent lack of complete penetration of adhesive resin into previously demineralized dentin may have contributed to the lower bond strength to dentin of primary teeth observed in this study.

Water is the primary medium for all three acidic primers evaluated in this study. According to Jacobsen et al, bonding systems based on water result in lower bond strength due to incomplete monomer polymerization.[36] While his conclusion could not be confirmed with Clearfil SE Bond, some interesting observations were made during this study when using Prompt L-Pop and Etch and Prime 3.0. After application of the bonding systems according to the manufacturers' instructions, a shiny dentin surface should be observed. To achieve a shiny surface, it was sometimes necessary to repeat the application up to five times.

When removing the jig that helped form the composite resin cone, a spontaneous bond failure occurred and a watery film could be observed on the dentin surface. A possible reason for this phenomenon could be the high water content of these bonding systems released during polymerization. While Prompt L-Pop and Etch and Prime 3.0 have a water content of over 70%, the acidic primer of Clearfil SE Bond has a water content of only 50%. Incomplete removal of water from the collagen network results in the competition between the monomer and the remaining water inside the demineralized dentin and might inhibit polymerization of the bonding agent.[37,38] Phase separation of the hydrophobic and hydrophilic monomer components causing blister-like spaces and globule formation of the bonding agent within the hybrid layer has been observed in overwet conditions.[39-41] In addition, excess water may also dilute the primer and render it less effective.[42]

Two of the materials evaluated in this study were filled adhesive systems. While Clearfil SE Bond is 10% filled, the filler content of Prime and Bond NT is about 3%. Studies have demonstrated that filled bonding systems may absorb in part the polymerization shrinkage stress of composite material by elastic elongation, preventing the interface from detaching.[39,43] Other studies did not find any difference when comparing filled and unfilled adhesive systems.[44,45] Compared to unfilled systems, both filled bonding materials tested in this study resulted in statistically higher bond strengths with both enamel and dentin of primary teeth than unfilled adhesive system. However, it remains unclear if the filler content is actually the reason for this difference. Typical fracture modes for enamel and dentin of primary teeth were adhesive fracture and mixed fractures. As has been previously reported, there was no correlation between the enamel-resin fracture mode and the bond strength in any of the adhesive systems.[29,46-48] The results of this study show that, whereas effective in vitro bonding to enamel of primary teeth could be observed for all four adhesive systems tested, only Clearfil SE Bond achieved adequate bond strengths to dentin of primary teeth. However, long-term clinical studies are required to evaluate the clinical efficacy and durability of these bonding systems.

Conclusions

1. Although all self-etching primers tested demonstrated adequate in vitro bond strength to enamel of primary teeth, Prime and Bond NT had significantly higher bond strength.
2. For dentin of primary teeth, Prompt L-Pop and Etch and Prime 3.0 resulted in complete bond failures, whereas the use of Clearfil SE Bond resulted in significantly higher bond strength then when bonding with Prime and Bond NT.
3. Based on the results of this study, further laboratory and in vivo studies are recommended prior to using these self-etching adhesive systems for restoration in primary teeth.

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Abstract of the Scientific Literature

Replantation of Avulsed Primary Incisors: a Risk-Benefit Assessment

Risk vs. benefit of replanting primary incisors was determined by a review of the available dental literature since there is "not a single protocol-based prospective outcome study of replantation of avulsed primary teeth." The conclusion verified the prevailing textbook opinion regarding avulsion management: avulsed primary incisors should not be replanted. Parents should be made aware that the potential risks (e.g. dental abscesses, root resorption, ankylosis, deflection of permanent incisors, hypoplastic and morphological changes to the permanent crowns) far outweigh the benefits, that at most, are based on anecdotal information. The downside would be more time in the dental chair, the use of additional local anesthesia, unnecessary radiation exposure, and, in all probability, the ultimate extraction of the avulsed tooth.

Comments: This is a valuable contribution to the pediatric dental literature in that a collection of references has been gathered that substantiate current opinion on avulsion management of primary teeth. The authors provide insight into the reality of decision-making processes in the office setting. And we are one step closer to evidence-based dentistry, albeit the prospective outcome study has yet to be done. **JDR**

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Replantation of avulsed primary incisors: a risk-benefit assessment. Zamon EL, Kenny DJ. *J Can Dent Assoc* 67:386, 2001. http://www.cda-adc.ca/jcda/vol-67/issue-7/386.html

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