Bond Strength of Self-etching Primer and Total-etch Adhesive Systems to Primary Dentin

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ABSTRACT

Purpose: The objective of this study was to assess in vitro the tensile bond strength of a self-etching and 2 total-etch, single-bottle adhesive systems to primary dentin.
Methods: Thirty-six sound primary canine buccal surfaces were randomly assigned to 3 groups (N=12), corresponding to the tested adhesive systems: (1) group I=Excite (EX); (2) group II=Single Bond (SB); and (3) group III=Prompt L-Pop (PLP). After 24-hour storage in distilled water, tensile bond strength was tested for failure at a crosshead speed of 0.5 mm/min.
Results: Means (MPa) and standard deviation (±) were: (1) EX=12.72 (±2.89); (2) SB=10.86 (±2.09); (3) PLP=8.66 (±2.23). Single Bond and Excite showed statistically similar results ($P > .05$) and provided the highest means. Prompt L-Pop was statistically different from the other groups ($P < .05$) and reached the lower bond strength to primary dentin.
Conclusions: Total-etch agents provided the best overall bonding performance, whereas the all-in-one, self-etching, self-priming adhesive system yielded remarkably lower bond strength to primary teeth dentin. (*J Dent Child*. 2004;71:131-134)

KEYWORDS: BOND STRENGTH, SELF-ETCHING PRIMER, PRIMARY DENTIN

In most currently available adhesive systems, bonding protocol relies on acid etching of teeth by a strong inorganic acid. This acid:

1. opens and widens dentinal tubule entrances;
2. increases intratubular dentin exposure/permeability;
3. demineralizes intertubular dentin.

The infiltration and further polymerization of a hydrophilic monomer, capable of interweaving with the exposed collagen network in dentin matrix, results in a resin-dentin interdiffusion zone or hybrid layer—which is generally accepted as the major factor to achieve optimal dentin bonding.[1,2]

The goals of self-etching primer adhesive systems are to:

1. simplify the bonding procedure;
2. reduce technique sensitivity of the adhesive protocol by eliminating the need for acid conditioning, rinsing, and drying of dental substrate.

This promising approach to adhesion uses primers with acidic monomers that:

1. simultaneously etch and prime dentin;
2. enable dissolution of mineral crystals around collagen fibrils and resin infiltrating beyond the smear-covered surface into the underlying dentin matrix.

Consequently, the hybrid layer is formed with the smear layer incorporated in it.[3-5] This mechanism claims to prevent:

1. formation of widely demineralized areas;
2. lack of resin monomer impregnation, since the demineralizing component of the primer is also the infiltrating resin.[6,7]

Earlier investigations have shown improved interaction between self-etching primers and tooth substrates, as compared to the conventionally acid-etched substrate.[8-11] Based on the clinical success of resin and adhesives in permanent and primary teeth, more conservative preparation can be performed when using resin-based composites to maintain tooth structure.

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Few articles investigate adhesive system bonding ability in primary teeth.[10-16] Due to an increasingly widespread use of adhesive restorative systems, it seems relevant to assess behavior of such materials on primary tooth structure.

The aim of this study was to assess the bond strength of 1 self-etching and 2 total-etch, single-bottle adhesive systems to primary teeth dentin in vitro.

METHODS

Thirty-six sound primary canines exfoliated/extracted within a 6-month period and stored in 0.4% sodium azide solution at 4°C were selected and carefully cleaned with water/pumice slurry using dental prophylaxis cups. When necessary, roots were sectioned 2 mm below the amelocemental junction, and crowns were embedded in polyester resin using polyvinyl chloride rings (2.1-cm diameter, 1.1-cm height).

After resin polymerization, the rings were discarded and the buccal surfaces of teeth were ground in a polishing machine (Politriz, Struers A/S, Copenhagen, DK-2610, Denmark) using water-cooled no. 320- to 400-grit silicon carbide (SiC) paper. This removed the overlying enamel and exposed flat, smooth dentin surfaces.

Additional grinding with no. 600-grit SiC paper was performed for 30 seconds to produce a standardized smear layer. To delimit the dentin bonding site, a piece of insulating tape with a 3-mm diameter central hole, created with a modified Ainsworth rubber-dam punch, was attached to the specimen surface. Limitation of the bonding site attempted to:

1. define a fixed test surface so that the bond strengths recorded would be related solely to the evaluated area;
2. warrant that the truncated resin composite cone would be further adhered precisely to the treated dentin surface, thus avoiding accidental adhesion to the surrounding enamel.

The specimens were randomly assigned to 3 groups of equal size (N=12), corresponding to the adhesive systems used:

1. group I=Excite (EX), an ethanol-based, total-etch single-bottle bonding agent;
2. group II=Single Bond (SB), an ethanol- and water-based total-etch adhesive system;
3. group III=Prompt L-Pop (PLP), a self-etching primer adhesive system.

Adhesive systems for all groups were placed according to the manufacturers' instructions. The agents were carefully applied onto the limited dentin surface with microbrush disposable brush tips. This avoided excess and pooling of adhesive along the edges of the insulating tape that could compromise the distribution of tensions during the test and hence the validity of results.

In group I, dentin surfaces were etched with a 35% phosphoric acid gel (Scotchbond Etchant, 3M/ESPE,) for 10 seconds, rinsed thoroughly, and excess water was blotted with absorbing paper to keep the surface moist. Excite was applied to dental substrates with a light scrubbing motion for 10 seconds. Next, the remaining solvent was evaporated with a brief, mild air blast, and the bonding agent was light cured for 20 seconds with a visible light curing unit using a 450 mW/cm^2 output (XL 3000, 3M/ESPE).

In group II, dentin sites were acid etched, rinsed and dried, as previously described. Then, 2 consecutive coats of Single Bond were applied to the etched surface, slightly thinned with a mild oil-free air stream, and light cured for 20 seconds.

In group III, Prompt L-Pop components were mixed and activated according to the manufacturer's specifications, and a uniform layer of the resulting mixture was applied onto dentin surfaces with a saturated microbrush, gently air thinned, left undisturbed for 15 seconds, and light cured.

Once the bonding protocols were completed, specimens were individually fixed in a metallic clamping device (developed by Houston Biomaterial Research Center,) that allows the test dentin surface to remain parallel to a flat base. A split-bisected polytetrafluoroethylane jig was positioned on the tooth/resin block. This provided an inverted conical cavity with the smaller diameter coincident with the delimited bonding site (Ø 3 mm). A hybrid, light-cured composite resin (Filtek Z250, 3M/ESPE) was inserted into the jig in increments and polymerized for 40 seconds each. As the cavity completely filled, the specimen was removed from the clamping device. The jig was opened and separated, leaving adhered to the delimited dentin site an inverted, truncated resin composite cone with a 6-mm diameter tapering to a 3-mm diameter and 4-mm height.

After 24-hour storage in distilled water at 37°C, the cone-shaped composite/polyester resin blocks were individually placed into an apparatus with an internal taper, corresponding to the resin cone's shape. This configuration was loaded in tension to failure using a universal testing machine (Mod. MEM 2000,) at a crosshead speed of 0.5 mm/minute and with 50 kgf load cell. This self-aligning system allows the forces to be applied perpendicular to the specimens' surface. Bond strengths were recorded in kgf/cm and converted into MPA, and means were calculated. Sample distribution and homogeneity were analyzed. Since a normal and homogeneous distribution was observed, data were submitted to one-way analysis of variance, using a factorial design with adhesive system as variable. Multiple comparisons were performed via Tukey test at 0.05 significance level.

Fractured specimens were observed with a ×40 stereomicroscope to assess the failure modes, which were classified as adhesive, cohesive, or mixed.

RESULTS

Mean bond strengths and standard deviation to primary dentin are listed in Table 1.

Table 1. Bond Strength Means (MPa) and Standard Deviation (±) to Primary Dentin for the Experimental Groups*

Groups		
Excite (I)	Single Bond (II)	Prompt L-Pop (III)
12.72 (±4.89) *ab*	10.86 (±4.09) *bc*	8.66 (±2.23) *c*

***Same letters indicate statistical similarity.**

The analysis of data revealed that, comparing the bonding agents assessed in this study, Single Bond and Excite total-etch adhesive systems showed statistically similar results ($P>.05$) and yielded the highest means to primary dentin. On the other hand, Prompt L-Pop self-etch primer reached a remarkably lower bond strength and was statistically different from the other groups ($P<.05$).

The analysis of bonding sites after tensile strength test revealed that adhesive failures minimally occurred (EX=20%; PLP=20%). SB total-etch system had an equal number of mixed failures (50%) and cohesive failures (50%), whereas EX showed a 70% rate of cohesive failures and a 10% rate of mixed failures. For the self-etching system, a mixed-failure mode was predominantly observed (80%).

DISCUSSION

The mechanism responsible for the adhesion of newer-generation dentin bonding systems is related to the formation of a resin-dentin in-diffusion zone or hybrid layer.[2,17] This is, at the molecular level, a durable and acid-resistant intermixture of adhesive resin and dentin components.[2] Based on current understanding, the characteristics of the dentin as a substrate for bonding of composite resin have an outstanding influence on the resin-dentin interface morphology[4,18] and possibly on the system's ultimate performance.[19]

Self-etching primers have been developed to:

1. simplify bonding procedures;
2. minimize the technique sensitivity for bonding to tooth substrates;
3. prevent discrepancies between the depth of dentin demineralization by the acid and the primer's ability to penetrate this demineralized layer.[20,21]

One of the basic concepts behind the self-etching primer approach is that tooth structure demineralization and bonding agent diffusion and embedding around dentinal collagen fibers happens at the same time and to the same depth. Recently, these adhesives systems have been classified into mild, moderate, and aggressive, depending on their ability to solubilize the smear layer and demineralize the underlying subsurface dentin.[5]

According to this classification, Prompt L-Pop, tested in the present study, should be considered an aggressive, self-etching primer, self-priming adhesive system. Prompt L-Pop has been known to dissolve the smear layer and smear plugs and form authentic hybrid layers, approaching the thickness of those observed in dentin etched with strong inorganic acids requiring additional rinsing.[5]

Therefore, the bonding mechanism of this self-etching primer to dentinal substrate is assumed to be closer to that of total-etch systems, with regard to hybridization and resin tag formation. This means that practically all the mineral content is removed from the collagen network, avoiding any interaction between hydroxyapatite and functional monomers.[22] Data from the manufacturer indicates that this system allows for adequate bond strengths to both enamel and dentin. In the current work, Prompt L-Pop bond strengths to dentin were remarkably lower than those recorded for the total-etch systems tested.

Agostini et al[23] evaluated the tensile bond strength of 3 of the most contemporary self-etching primers (Prompt L-Pop, Clearfil SE Bond, and Etch & Prime 3.0) to primary enamel and dentin. They reported that, although all the tested adhesive systems bonded effectively to primary enamel, only Clearfil SE Bond achieved adequate bond strengths to the dentin of primary teeth (39 MPa). Prompt L-Pop and Etch & Prime 3.0 resulted in complete bond failures and markedly lower bond strengths. The depth of dentin demineralization achieved with these self-etching primers might differ, depending on the pH. The lower the pH of the conditioner, the deeper the depth of demineralization.[24]

According to a recently proposed categorization of adhesives by application modes, Etch & Prime 3.0 can be described as a 2-step smear layer dissolving system with a strongly acidic primer (pH=0.6). Prompt L-Pop is a 1-step smear layer modifying adhesive system (pH=1), and Clearfil Se Bond is a 2-step smear layer modifying bonding system[25] with the highest pH (2), Clearfil Se Bond achieved the highest bond strength to primary dentin. Agostini et al[23] speculate that it is possible the other 2 acidic primers evaluated in their study caused excessive dentin demineralization. The hybrid layer's resulting increased thickness and the subsequent lack of complete penetration of the adhesive resin into previously demineralized dentin may have contributed to the lower bond strengths obtained.[25]

It has been reported that bonding systems based on water result in lower bond strength, due to incomplete monomer polymerization.[25] In most of single-bottle, total-etch adhesive systems, the hydrophilic monomers are dissolved in solvents like ethanol/water or acetone.[26] It has been reported that adhesive systems containing acetone should better interact with substrates, greater amount of water to allow an adequate diffusion of the resin monomers through the exposed collagen fiber network.[22] On the other hand, adhesives containing alcohol and water as solvents in their formulation, such as Single Bond and Excite, need a slightly moist substrate for optimal bonding.[22] Prompt L-Pop has a water content of greater than 70%. Although no published data support this assumption, it may be inferred that these shortcomings had a definite role on the bonding performance of Prompt L-Pop.

The goal of this study was to assess in vitro the tensile bond strength of 1 self-etching and 2 total-etch, single-bottle adhesive systems to primary dentin. It is important, however, to note that the lack of studies testing the same methodology and materials on primary teeth was definitely a hindrance to stating a reliable comparison between the outcomes of the conducted research and available data. While bonding to permanent teeth has been studied extensively, few studies have addressed resin bonding to primary teeth.

The tested self-etching, self-priming adhesive system, Prompt L-Pop, incorporates all elements of contemporary systems in a single solution, resulting in the first true "one-step" agent for enamel and dentin bonding. This formulation led to a considerable decrease in operating time, which might be of particularly interest for bonding teeth that cannot be adequately isolated—as is common in pediatric dental practice.[27]

In spite of its expected bonding ability and advertised benefits, however, Prompt L-Pop did not perform as well as the total-etch adhesive systems. This leads to the assumption that an undermined clinical outcome may also be expected.

Therefore, taking into account the outstanding approach of adhesive dentistry in pediatric patients, it is mandatory that further studies be developed with the aim of:

1. investigating
   a. bond strength;
   b. resin/dentin interface characteristics;
   c. the type of interaction occurring between the currently available self-etching primer adhesive systems and the primary substrates;
2. foreseeing, with some degree of reliability, the quality of the adhesion obtained.

CONCLUSIONS

Based on the current study's findings, and within the limitations of an in vitro investigation, it may be concluded that the tested total-etch agents provided the best overall bonding performance. The all-in-one, self-etching, self-priming adhesive system, conversely, yielded a remarkably lower bond strength to primary teeth dentin.

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