Bond Strength of a Sealant to Primary and Permanent Enamel: Phosphoric Acid Versus Self-etching Adhesive

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Abstract

Purpose: The objective of this study was to compare the effect of phosphoric acid and a self-etching adhesive on the short and long-term bond strength of a light-curing sealant to unground primary and permanent enamel.

Methods: A light-curing resin sealant (Delton Light Curing Pit & Fissure Sealant–CLEAR) was bonded to the flattest, peripheral surface of 40 primary and 40 permanent molars following conditioning of the cleaned enamel with 38% phosphoric acid or with the self-etching adhesive Prompt L-Pop (N=10/group). After either 1 week or 1 year in water, shear bond strengths were measured. Failure mode was determined in a stereo microscope.

Results: There was no significant difference in bond strengths between the phosphoric acid-etch and the self-etching adhesive groups, nor between the 1-week and 1-year results ($P>.05$). However, the bond strengths to primary enamel were lower than those to permanent enamel ($P=.0021$). The number of pure adhesive failures in each of the 8 groups varied between 0 and 3 (0-30%), and the remaining teeth displayed mixed adhesive-cohesive failures.

Conclusions: The self-etching adhesive studied seems an attractive alternative to the acid-etch technique for sealant application in young children where simplifications in the clinical procedure are warranted. (*Pediatr Dent.* 2004;26:240-244)

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In a review based on almost 1,500 references, Simonsen concludes that application of pit and fissure sealants for caries-susceptible teeth is a safe and effective caries preventive treatment.[1] Contrary to glass ionomer sealants, resin sealants have shown excellent retention,[2-4] and despite some caries preventive effect caused by the fluoride release from the glass ionomers even after apparent loss of the material,[5,6] recent studies find resin sealants to be superior also with respect to caries prevention.[3,4]

Effective blocking of the fissure system is obtained through formation of a strong bond of the sealant to enamel. Since the introduction of the sealant technique, as a direct clinical benefit from Buonocore's work,[7] the standard clinical procedure has involved etching with phosphoric acid of cleaned enamel, rinsing with water spray, establishing of a dry field followed by application and curing of the sealant. Saliva contamination of enamel has been shown to reduce the bond strength significantly,[8] but it may be exceptionally difficult to avoid considering that most fissure sealants are placed in young patients who often display less than optimal patient behavior. Indeed, patient behavior and compliance have been shown to play a significant role for the success of sealants.[9] The self-etching adhesives, which are gaining popularity, eliminate the rinsing procedure, whereby the time of treatment and the need for patient compliance is reduced. As these factors may ultimately increase sealant success, self-etching adhesives appear to be an attractive alternative to acid etching.

Today, most fissure sealants are placed following an individual risk analysis based mainly on prior caries experience of the patient, fluoride history of the patient, fissure anatomy, and plaque load.[10,11] This means that not only permanent molars, on which most sealant studies have focused, but also primary molars may be at risk for occlusal caries and may benefit from sealant application.[12-15] Due to the presence of a prismless superficial layer, primary

enamel was believed not to etch well and, therefore, to be difficult to bond. In an attempt to overcome this "resistance to etching," double the etching time of permanent enamel was advocated for primary enamel. However, clinical studies of fissure sealants in primary teeth have shown retention rates as high as those for permanent enamel with the same etching times.[12-15] In regard to self-etching adhesives, few studies have reported on the bonding capacity to primary enamel, and none of these studies used unground primary enamel.[16,17]

This in vitro study evaluated the short and long-term shear bond strength of a light-curing sealant to unground permanent and primary enamel, which had been conditioned with either phosphoric acid or a self-etching adhesive. The null hypotheses to be tested were:

1. use of self-etching adhesive would result in bond strengths equivalent to those obtained with conventional phosphoric acid etching;
2. bond strengths to primary enamel would be equivalent to the bond strengths to permanent enamel;
3. bond strengths measured after 1-year storage would be equivalent to the bond strengths measured after 1 week.

Methods

Forty caries-free primary molars and 40 caries-free permanent molars were used. After extraction, the teeth were cleaned of gross debris and stored in a 0.5% chloramine T solution. The teeth were embedded in stone (Vel-Mix Stone, Kerr, Salerno, Italy) with the flattest of the peripheral surfaces (ie, the labial, lingual, mesial, or distal surface) parallel to the horizontal plane. Keeping primary and permanent teeth apart, they were randomly divided into 8 groups of 10. All enamel surfaces were cleaned with a slurry of pumice and white rubber cup, rinsed, and air dried. In 2 groups of primary teeth and 2 groups of permanent teeth, the enamel was etched for 30 seconds with 38% phosphoric acid gel (Etch-Rite, Pulpdent, Watertown, Mass), rinsed for 20 seconds, and air dried. In the 2 other groups of primary teeth and permanent teeth, the enamel was treated with Prompt L-Pop (3M ESPE, Seefeld, Germany) as follows.

First, Prompt L-Pop was applied, rubbed lightly for 15 seconds, and air dried into a homogeneous, shiny layer. The test area was now defined by application of a piece of self-adhesive tape into which was punched a hole (diameter=2.5 mm). Each stone block was then mounted in a holder, which allowed a mold (diameter=2.1 mm, height=1.5 mm) to be pressed into contact with the enamel surface. The mold was filled with Delton Light Curing Pit & Fissure Sealant—CLEAR (Dentsply, York, Penn), which was light cured for 40 seconds with an XL 3000 light-curing unit (3M, St. Paul, Minn) whose power density was determined by an Optilux radiometer (Kerr, Danbury, Conn) to be 500 mW/cm.[2]

Ten minutes after the end of light curing, the stone block was removed from the holder and placed in deionized water at 37°C for 1 week or 1 year before measurement of shear bond strength (MPa) in an Instron 5566 (Instron, High Wycombe, England) at a crosshead speed of 1 mm/minute and with a static load cell of 10 kN. The fractured assemblies were examined in a stereo microscope at ×18 to ensure that the Delton button had been positioned on enamel only and characterize the failure mode.

The failure mode was categorized as adhesive if the enamel surface appeared free of any adhesive/sealant. If the total enamel bonding area was covered by adhesive/sealant, the failure mode was categorized as cohesive. Finally, if only part of the enamel bonding area was covered with adhesive/sealant, the failure mode was categorized as mixed. Statistical evaluation of the shear bond strength data was performed using 3 factorial ANOVA (SAS 8e software, SAS Institute, Cary, NC) to identify differences between conditioning agent, tooth type, and storage time. Differences in failure mode were analyzed by the Fisher exact probability test. Statistical significance was considered as $P<.05$.

Results

The mean and standard deviation values of shear bond strength are shown in Table 1, and the results of the 3 factorial ANOVA are shown in Table 2. The factor conditioning agent and factor storage time had insignificant effects, whereas the factor tooth type had a significant effect ($P=.0021$). There were no significant interactions between the 3 factors. Three specimens had to be discarded, as the Delton button had been positioned partly on dentin.

Two types of failure mode were observed:

1. adhesive failure at the interface between enamel and sealant;
2. mixed failure where part of the failure had occurred at the interface and another part had occurred cohesively within the sealant.

The number of specimens exhibiting 100% adhesive failure in each of the 8 groups varied between 0 and 3 (10-30%). None of the 3 factors—conditioning agent, tooth type, and storage time had a significant influence on failure mode.

Discussion

The self-etching adhesive, Prompt L-Pop, was found to mediate shear bond strengths to unground, human enamel of the same magnitude as did phoshoric acid etching: Thus, the first null hypothesis was accepted. This result is in agreement with that of Ibarra and coworkers measuring bond strengths of Herculite XRV resin composite to unground, bovine enamel.[18] The present result also corroborates that of Gillet and coworkers, who showed that Prompt L-Pop was as effective as phosphoric acid in obturating the fissures of extracted human bicuspids with Tetric Flow.[19] Furthermore, the result backs the work of Feigal and

Table 1. Shear Bond Strength of Sealant to Unground Enamel (MPa)

Conditioning agent	1-week storage time		1-year storage time	
	Primary enamel	Permanent enamel	Primary enamel	Permanent enamel
Phosphoric acid (38%)	11.2±4.6	13.7±3.6	11.5±3.5	16.5±6.2
Prompt L-Pop	13.1±3.5	15.8±5.1	11.5±4.3	13.9±2.2

Table 2. Results of the 3-factorial ANOVA, Dependent Variable: Bond Strength

Effect	SS	Df	ms	F	*P*
Tooth type	186.87	1	186.87	10.21	.0021
Conditioning agent	2.33	1	2.33	0.13	.7222
Storage time	0.20	1	0.20	0.01	.9175
Conditioning agent × Tooth type	7.09	1	7.09	0.39	.5357
Storage time × Tooth type	5.11	1	5.11	0.28	.5990
Storage time × Conditioning agent	50.90	1	50.90	2.78	.0999
Storage time × Conditioning agent × Tooth type	9.05	1	9.05	0.49	.4842
Error	1244.27	68	18.30	-	-

Quelhas, who found equivalent 2-year retention rates of sealants in permanent molars applied following the use of Prompt L-Pop or of acid etching.[20]

However, the result is in disagreement with that of Perry and Rueggeberg, who reported increased microleakage related to sealants placed in extracted permanent molars conditioned with Prompt L-Pop as compared with conventional acid etching.[21] The present finding is also in conflict with the finding of Bishara and coworkers, who bonded metal brackets to intact buccal enamel of extracted human molars. The bond strength obtained by use of Prompt L-Pop was significantly lower than the bond strength obtained by phosphoric acid etching.[22] Finally, the present result contradicts that of Pashley and Tay, who found that bond strengths of Z100 resin composite to unground, human enamel promoted by 3 self-etching adhesives were significantly lower than the bond strength promoted by a total-etch adhesive system.[23] However, they also showed that the lower bond strength obtained with Prompt L-Pop could not be explained by an insufficient etching effect, with the hybrid layer being as continuous and thick as that produced by phosphoric acid etching. Thus, the results obtained so far regarding the efficacy of Prompt L-Pop on enamel are as contradictory as results of in vitro and in vivo studies of the efficacy of Prompt L-Pop on dentin.[16,24-32]

A possible explanation for the differences in reported efficacy is that Prompt L-Pop is not equally compatible with all resin materials. Peutzfeldt and Asmussen have found that the shear bond strength of 6 different resin composites to dentin mediated by Prompt L-Pop varied between 1 and 13 MPa.[33] This implies that Prompt L-Pop may not be as effective as phosphoric acid etching in mediating a bond to enamel with all sealants. An additional factor that complicates comparisons between results of different studies is the fact that the bond strength is influenced by the mechanical properties of the resin material being bonded.[34-36] Had a more highly filled, and thus stronger, resin material been used as sealant in the present study, it might have resulted in a shift from predominantly mixed failures to predominantly or exclusively adhesive failures and in higher bond strengths. It is even possible that these changes would occur to different degrees for the 2 conditioning agents and result in differences in bond strength between phosphoric acid etching and the self-etching adhesive, Prompt L-Pop.

Irrespective of conditioning agent, the bond strengths to primary enamel were lower than the bond strengths to permanent enamel, which means that the second null hypothesis had to be rejected. Other in vitro studies have found contradictory results when comparing bonding efficacy to primary and permanent enamel,[17,37-39] whereas in vivo studies have shown equivalent retention rates of fissure sealants in primary and permanent molars.[12-15] Whether differences in bonding efficacy to permanent vs primary enamel are found may have to do with the surface of the teeth used for testing: certain areas of primary enamel, mostly in the cervical regions, more often than permanent enamel contain a prismless layer which may interfere with tag formation and alter resin penetration and adhesion.[40,41] Many laboratory studies make use of peripheral tooth surfaces instead of occlusal surfaces, and this may explain why some studies, including the present, find an

impaired bonding efficacy to primary enamel as compared to permanent enamel.

It is of crucial importance for the longevity of bonded restorations that the bond is stable over time in an aqueous environment. Bond durability is evaluated in vitro by thermocycling or by long-term immersion in water. The present study compared the durability of the bonds using long-term water storage. For both conditioning agents studied, the bond strengths measured after 1 year were equivalent to the bond strengths measured after 1 week, leading to acceptance of the third null hypothesis. Previous studies of the durability of bonds mediated by self-etching primers are limited. Testing other self-etching adhesives than Prompt L-Pop, Miyazaki and coworkers found a significant decrease in bond strength to ground, bovine enamel following thermocycling.[42]

On the other hand, in a study on monkeys, Sano and coworkers reported no change in bond strength to dentin of some of the same self-etching adhesives following up to 1-year water storage.[43] Likewise, in a study including Prompt L-Pop as well as total-etch 2- and 3-step adhesives, the bond strengths to human enamel and dentin were also found to be stable after 1-year water storage.[31] Being one of the most acidic self-etching adhesives, Prompt L-Pop has been found to produce an etching effect on unground enamel that approaches that of total-etch adhesives and results in a thick, continuous hybrid layer.[23] This aggressiveness may explain the stability of the bond mediated by Prompt L-Pop in comparison to the self-etching adhesives studied by Miyazaki and coworkers. Another explanation may reflect that thermocycling and long-term water immersion test 2 different facets of bond durability: thermocycling stresses the bond by utilizing the fact that substrate and adhesive materials differ with respect to coefficient of thermal expansion, whereas long-term water storage determines the resistance of the adhesive bond to hydrolytic degradation. It may be that self-etching adhesives are relatively resistant to hydrolytic degradation.

As previously mentioned, only very few studies have tested bonding to unground enamel. This is probably due to the difficulty in obtaining a sufficiently plane area large enough for bonding. By reducing the bond area from what is normally used for ground tooth surfaces, the present study hoped to be able to meet these criteria, and as a rule the method succeeded. However, the post-fracture microscopic examination revealed that in approximately 10% of the teeth, the sealant had spread out under the mold due to curvature of the bond area. In these cases, it may be argued that the bonding area may have been enlarged and, therefore, the bond strength value computed higher than the true value, which is a limitation of the method. More clinical studies are warranted in which the acid etching is substituted by a self-etching adhesive prior to application of fissure sealants.

Conclusions

The self-etching adhesive, Prompt L-Pop, is as effective as phosphoric acid in mediating a bond between a light-curing sealant and unground human enamel.

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