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Evaluation of Shear Bond Strength and SEM Observation of All-in-one Self-etching Primer Used for Bonding of Fissure Sealants

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Abstract

Objectives: To evaluate and compare enamel shear bond strength of an all-in-one self-etching primer (Prompt™ L-Pop™) to regular acid etch material when bonded to two-fissure sealant systems (Concise and Dyract Seal).

Method and Materials: Forty newly extracted non-carious first permanent molars were embedded in a Teflon mold. The teeth were divided into four groups and each consisted of ten specimens. The bonding surfaces were treated with either Prompt L-Pop as recommended by the manufacturer or etched with phosphoric acid. After 24 hours of water storage, the specimens were evaluated for shear bond strength using an Instron testing machine. Scanning electron microscope (SEM) examinations were carried out to evaluate the failure sites of the sealants.

Results: The mean shear bond strengths using Prompt L-Pop were Concise: 23.46 MPa and Dyract Seal: 20.34 MPa. These values were higher than and statistically different from those of Dyract Seal (9.99 MPa) and Concise (8.85 MPa) when phosphoric acid was used. The failure was predominantly of the adhesive type. The SEM examination showed the failure of debonding was predominantly related to the type of etching systems used rather than the type of fissure sealants.

Conclusion: The use of the all-in-one self-etching adhesive Prompt L-Pop improves the mean enamel shear bond strength of fissure sealants.

Keywords: Enamel shear bond strength, self-etching adhesive, fissure sealants

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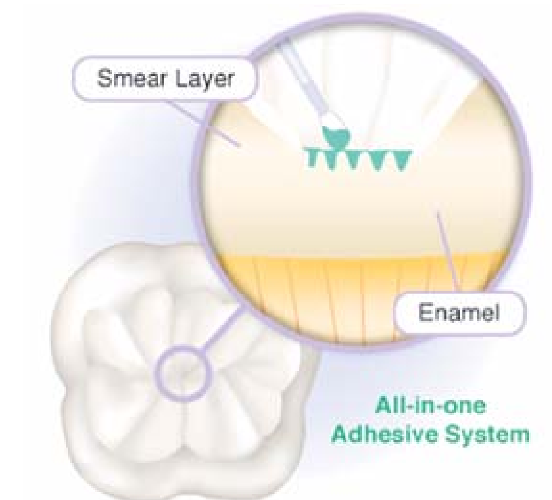
Introduction

The technique of pit and fissure sealants plays undoubtedly a fundamental role in preventing occlusal caries in both primary and permanent teeth.[1] Rinsing the tooth after acid etching can be unpleasant and become a source of disruptive behavior, particularly in young children.

The effectiveness of sealants depends on their ability to penetrate fissures before hardening, thus, producing a mechanical barrier to caries. To produce bonding and retention, the sealant material must flow over the etched enamel surface and penetrate micropores in the etched surface.[2] Because of this intimate relationship, it was felt by some researchers the penetrative ability of the sealant would affect its ability to bond to enamel.[3] Resin penetration, however, was found to be dependent on the underlying etch pattern, wetting ability of the enamel, the material's surface tension, viscosity, and rate of polymerization.[3,4]



The all-in-one adhesive system (Prompt™ L-Pop™) combines etching, priming, and adhesive potentials in only one solution. The theory behind this new adhesive was Prompt L-Pop, when applied on enamel, would dissolve the smear layer by rubbing it in for 15 seconds and at the same time it would etch the enamel surface to create the hydroxyapatite etching pattern. By air-drying, a very thin film of Prompt L-Pop is formed on top of the enamel. This enables the micromechanical retention of a restorative material in combination with a chemical bond between this thin Prompt L-Pop layer and the restorative material.[5] *In vitro* tests with Prompt L-Pop show the bond to dentin is variable, while bond to enamel showed acceptable micromorphology and microtensile bond strength.[6] A study by Faontes et al., testing the bond provided by Prompt L-Pop between enamel and the sealant, exhibited minimal microleakage on dry enamel and also minimal microleakage in the face of salivary contamination of the enamel.[7] An *in vivo* study evaluated the success rate of sealants with Prompt L-Pop through 24 months post-insertion which showed a 60% success rate with no difference between successful cases and the matched controls using a phosphoric acid etchant.[8]

The objective of this study was to study the all-in-one self-etching primer (Prompt L-Pop) in an *in vitro* comparative evaluation of two-fissure sealants bonded to teeth with either phosphoric acid or Prompt L-Pop by measuring the shear bond strength. Scanning electron microscope (SEM) will later be used to examine the interfacial morphology at the failure sites of the shear bond strength.

Method and Materials

For this study, 40 caries-free human first permanent molars extracted for orthodontic reasons were used. The mesial and distal surfaces were used in the study. Materials used were Concise (3M Dental Products Division Laboratory, St. Paul, MN, USA), Dyract Seal (Dentsply DeTrey GmbH, Konstanz, Germany), 37% phosphoric acid in a gel form, and a new-self-etching adhesive Prompt L-Pop (3M ESPE America, Norristown, PA, USA). The rational behind choosing the above mention sealants was its availability and the frequent use by members of the dental institution where the study was conducted. Also, both Concise and Prompt L-Pop are 3M products.

The teeth were randomly distributed into four groups each with ten teeth.

- **Group 1:** Concise sealer following 37% phosphoric acid etching gel (Densply) etched for 30 seconds, then rinsed for 20 seconds, and dried for 30 seconds.
- **Group 2:** Dyract Seal after etching with phosphoric acid.

- **Group 3:** Concise sealer using Prompt L-Pop as described by the manufacturer and applied onto tooth surface with a saturated microbrush and rubbed in for 15 seconds. Then a thin air stream was applied for 10 seconds followed by 10 seconds for polymerization.
- **Group 4:** Dyract Seal after etching with Prompt L-Pop as described for Group 3.

The mesial or distal surfaces at the gingival level of each tooth were polished with silicon carbide paper (0.5 mm of enamel was removed) before etching in order to obtain uniform enamel surfaces and remove any plaque or calculus deposition to which sealant could be applied. A plastic ring-shaped mold 3 mm deep and 3 mm in diameter was made of Teflon material and placed over each tooth perpendicular to the polished surfaces. The test materials were placed in the mold to form a button and cured according to the manufacturer's instructions. Once the materials were light-cured, the specimens were stored in 37ºC distilled water for 24 hours in order to avoid dehydration.

The specimens were then mounted in a metal ring with treated surfaces parallel to the shearing rod of the testing machine Instron (Instron Corp, High Wycombe, Buckinghamshire, England) at a crosshead speed of 0.5 mm/minute and the results were recorded in Megapascals (MPa). The mean and standard deviations (SD) were subjected to student-t-test for any significant findings. Also, two-way analysis of variance (ANOVA) was used for analysis of Prompt L-Pop or phosphoric acid and its relation to the fissure sealant systems in order to demonstrate a significant effect of the etching system type bonded to sealant materials.

Scanning Electron Microscopy (SEM)

After determining the peak force required to break the bond, the crowns were mounted on aluminum stubs using conductive carbon paints. Then, all specimens were coated with gold palladium (Balzers-S CD 050) using a sputter coater and observed under a SEM (JEOL, 5600 LV Japan) at 25kV of accelerating voltage while the working distance was kept at 20 mm. The specimens were then photographed at 1000x magnification.

Results

Shear Bond Strength

The mean (± SD) values of the shear bond strength of the two materials etched with either phosphoric acid or Prompt L-Pop in this study are presented in Table 1. Statistical analysis of the data was accomplished by using Student t-test and ANOVA, accepting an alpha level of 0.05 of significance.

Higher values of adhesion were observed using either Dyract (20.34 MPa) or Concise (23.46 MPa) etched by Prompt L-Pop. There were significant statistical differences among the materials by etching method: 9.09 MPa for Dyract fissure sealant etched with phosphoric acid and 20.34 MPa for Dyract etched with Prompt L-Pop at level $P < 0.05$. In addition Concise showed statistically significant differences when Prompt L-Pop (23.46 MPa) was used as etching material in comparison to phosphoric acid (8.85 MPa) at level $P < 0.05$ (Student t-test).

Using the ANOVA test showed there were interactions between fissure sealant materials and the etching system at the 0.05 level of significance (Table 2). There is substantial evidence the distribution of PFS scores (test of significant for ANOVA is a ratio of F) is different between the etching and sealant groups. The two-type of sealant materials etched with Prompt L-Pop showed significant value.

Scanning Electron Microscopy (SEM)

Each specimen was examined by SEM to determine the type of interfacial bonding failure. The specimens showed a resin replica of etched enamel created by sealant penetration into enamel microporosities exposed by etching. Some morphological differences were apparent between materials treated with the same etching system. When Prompt L-Pop was used, the fracture surface was less distinct between the two sealant materials.

Table 1. Shear bond strength for the fissure sealants after tooth etching by phosphoric acid (PA) or Prompt L-Pop

	Concise		Dyract Seal	
	PA	Prompt L-Pop	PA	Prompt L-Pop
Mean (MPa)	8.85	23.46	9.99	20.34
± SD	1.72	2.76	3.50	2.45

Mean values PA vs. Prompt L-Pop significant at $P < 0.05$

Table 2. Analysis of Variance (ANOVA).

Source of Variability	df	Sum of Square	Mean Square	F	Significant
Fissure sealants	3	1613.750	537.917	74.407	0.05
Etching system	1	1558.440	1558.440	215.570	0.05
Interaction between Etching & Fissure sealants	1	45.482	45.482	6.291	0.05
Error	36	7.229			
Total	40	11687.348			

Mean values phosphoric acid vs. Prompt L-Pop significant at $P < 0.05$

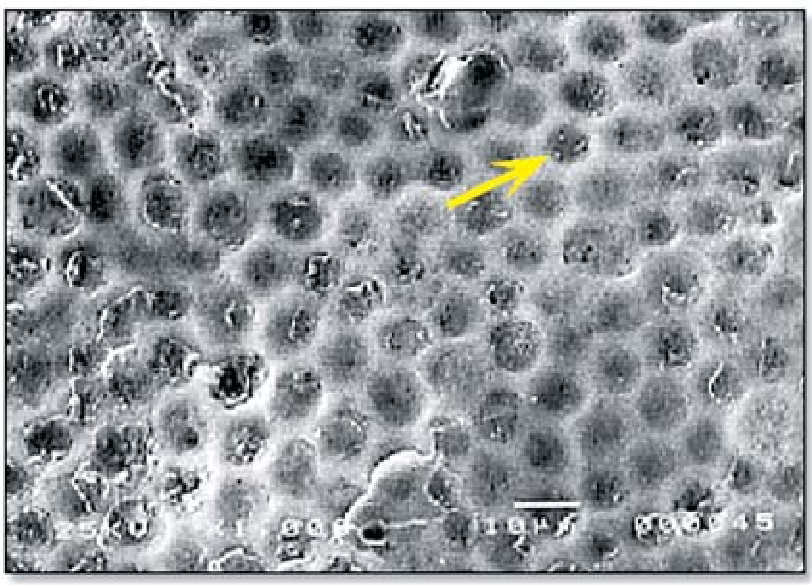


Figure 1. SEM micrograph of the enamel bonded with phosphoric acid and Concise showing the sealed interface and interfacial gap (magnification: x1000). Arrow shows fractured enamel with air bubbles.

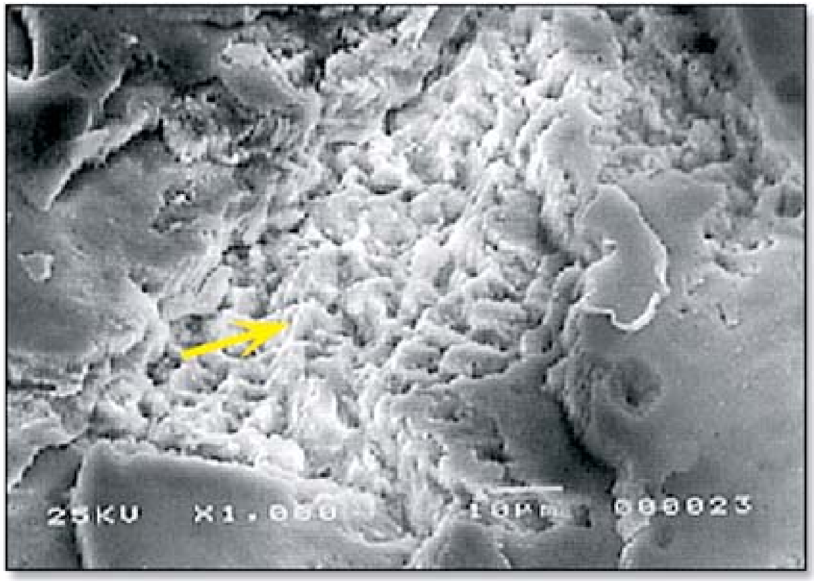


Figure 2. SEM micrograph of the enamel bonded with Prompt L-Pop and Concise showing a well-sealed interface (magnification: x1000). Arrow shows a fracture in the sealant material interface.

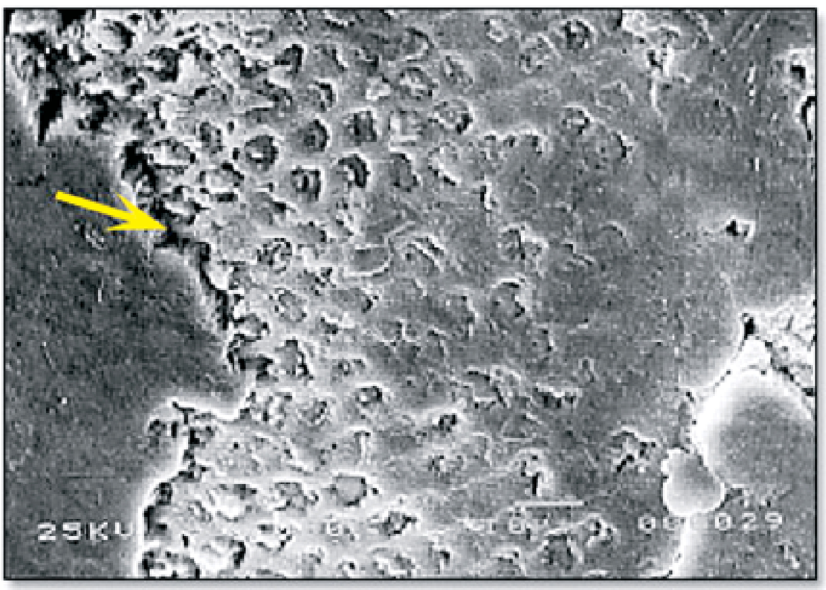


Figure 3. SEM micrograph of the enamel bonded with phosphoric acid and Dyract Seal showing sealed interfaces with irregular penetration of the sealant (magnification: x1000). Arrow shows a few prisms and an etched surface fracture.

Figure 1 shows a fracture of Concise sealant material, which is often associated with small air bubbles trapped in the sealant when etched with phosphoric acid. Also, a small number of enamel fractures were observed.

In other specimens of Concise material etched with Prompt L-Pop many fractures occurred in the sealant's material surface, which produced rougher fractured areas (Figure 2). The fracture surface indicates cohesive failure occurs between sealant material interfacials.

A few specimens of Dyract Seal sealant material showed small fractures within the enamel involving a few prisms and etching fracture when phosphoric acid was used as the etching system (Figure 3). The failure predominantly occurs in

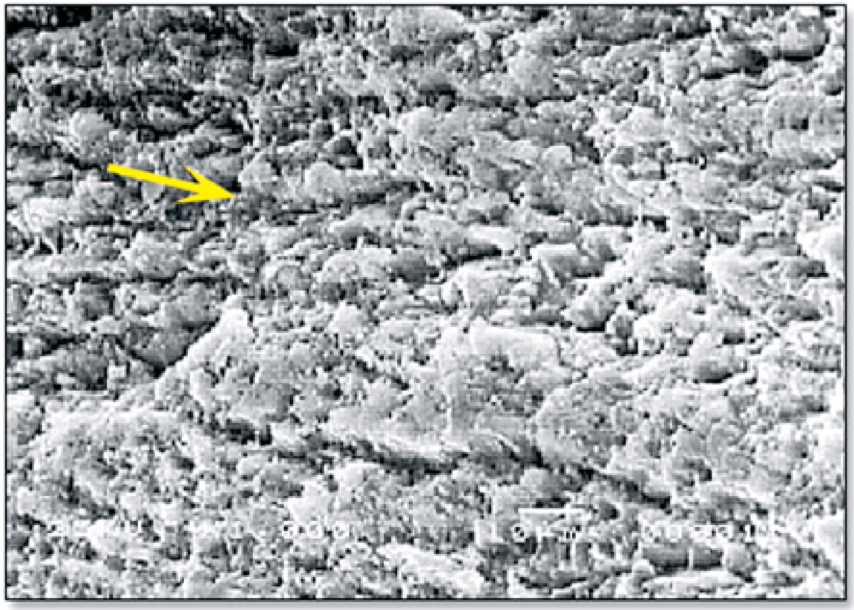


Figure 4. SEM micrograph of the enamel bonded with Prompt L-Pop and Dyract Seal showing well-sealed interfaces with penetration of the sealant (magnification: x1000). Arrow shows linear wave-like pattern in sealant interface.

the etching layer away from the enamel surface leaving the enamel surface covered with sealant.

The failure of Dyract Seal using Prompt L-Pop adhesive within the material or cohesive fractures produced a linear wave-like pattern in the sealant, which radiated from the area of material failure rather than etching failure (Figure 4).

Discussion

In both pre-school and disabled children moisture elimination is almost impossible.[9] This factor limits the mechanical properties of adhesion of fissure sealants to enamel and predisposes their early failure. In view of this the use of Prompt L-Pop as an etching material would be beneficial due to excluding the washing step of regular etching system.

Self-etching primers have been developed to simplify bonding procedures and to decrease the sensitivity of the technique for bonding to tooth structures. The present study is an *in vitro* study which was designed to examine the bonding strength and interfacial micromorphology failure of sealants bonded with Prompt L-Pop to enamel. The analysis of data obtained demonstrates significant differences between the same materials when etched with different etching systems.

Bonding to the intact natural surface of enamel is fundamental to achieve marginal sealing and for retention of pit and fissure sealants.[10] A study including Prompt L-Pop and total etch two- and 3-step adhesives showed bond strengths to human enamel was stable after one year water storage.[11] Also, being one of the most acidic self-etching adhesives, Prompt L-Pop has been found to produce (1) an etching effect on unground enamel approaching total etch adhesives and (2) a thick continuous hybrid layer.[12]

Prompt L-Pop is claimed to work as a self etching primer, thus, eliminating the phase of substrate etching rinsing and drying steps. The results of the published studies investigating the effectiveness of self-etching primers on ground enamel are not in agreement. Some authors have stated the bonding of self-etching adhesives to enamel is inferior to that achieved with total etch systems utilizing phosphoric acid as a separate conditioner.[13] Some other investigations have shown while self-etching primers produce more shallow etching patterns and resin penetration, the levels of bond strength achieved on enamel are satisfactory.[14,15] The stronger bond strength is in agreement with other studies when Prompt L-Pop was used in conjunction with composite or compomers.[16,17] Issa and Watts[13] as well as

Friedi et al.[18] reported the use of Prompt L-Pop under composite as bonding agent to enamel significantly increased bond strength (35 MPa) compared to those bonded with phosphoric acid etching (13 MPa). When there was no contamination, as in ideal conditions, using the bonding agent under the sealant yielded a significantly stronger bond.[19] Also, another study has found significant morphological adaptation of sealants to the fissure wall when self-etching primers are used.[20] Enamel bond strengths are adequate to prevent opening of margins by polymerization shrinkage.[21] It has been estimated bond strengths of 20-23 MPa may be required to resist contraction forces sufficiently to produce gap-free restoration margins. In the present study the bond strength values were approximately the same range when Prompt L-Pop was used, therefore, they may be sufficient to resist the contraction stresses.

The SEM micrographs of Dyract-Seal and Concise etched with phosphoric acid revealed these sealants appeared to have a fracture within etching due to the low bond strength of these materials. Since the shear bond values associated with these two materials were in the range of 2-10 MPa, most fractures are a combination of adhesive and cohesive fractures. However, when Prompt L-Pop is used, the micro-mechanical bonds obtained by these two sealants may, in part, be due to the flow characteristics of the sealant's materials or the phosphonated monomer of the adhesive. This pattern of bonding yielded bond strength values of 20-23 MPa. The fracture of the enamel could be due to the use of non-vital teeth as reported earlier.[22]

Conclusion

Self-etching primers have been developed to simplify bonding procedures and to decrease the sensitivity of the technique for bonding to tooth structure. Prompt L-Pop is a ground-breaking material that incorporates all the elements of contemporary adhesive systems in one solution, resulting in a one-step agent for enamel bonding. Its formulation resulted in a considerable decrease in the time necessary for bonding procedures. This might be beneficial for bonding teeth that cannot be adequately isolated in pediatric patients and it is certainly appealing to the busy practitioner.

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