

SEM study of a self-etching primer adhesive system used for dentin bonding in primary and permanent teeth

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Abstract

Purpose: The purpose of this study was to evaluate the interfacial micromorphology of direct esthetic restorations bonded to primary or permanent tooth dentin with a self-etching primer adhesive system.

Methods: Superficial dentin at the occlusal surface of 15 primary and 15 permanent molars was exposed with a carbide bur. Prompt-L-Pop was applied in one half of each surface. A control bonding system, Single Bond or Vitremer Primer, was used in the other half. Teeth were restored either with a composite resin (Filtek Z250), a compomer (Hytac), or a resin-modified glass ionomer (Vitremer). Twenty-five scanning electron microscope fields from 5 teeth were evaluated blindly by two investigators for each condition.

Results: In this study, a significant difference in quality of the interfacial seal was not observed when restorations performed in primary teeth were compared to restorations in permanent teeth. Interfacial gaps were observed in most restorations bonded with Prompt-L-Pop and restored with Filtek Z250 (9/10), Hytac (9/10), or Vitremer (5/10). No interfacial gaps were observed in teeth bonded with Single Bond and restored with Filtek Z250 (0/10) or Hytac (0/10), while all teeth bonded with Vitremer Primer and restored with Vitremer presented gaps (10/10). To understand the reason for the interfacial gaps observed with Prompt-L-Pop, we examined if this system generated a hybrid layer at the dentin/restorative material interface. All surfaces bonded with Single Bond and restored with Filtek Z250 or Hytac presented a visible hybrid layer. In contrast, 0/10 (Z250) and only 3/10 (Hytac) restorations bonded with Prompt-L-Pop showed signs of a hybrid layer.

Conclusion: The self-etching primer adhesive system Prompt-L-Pop failed to generate sealed interfaces consistently between the dentin of primary and permanent teeth and the composite resin or the compomer evaluated in this study. (*Pediatr Dent* 23:315-320, 2001)

Introduction

Recent advances in the chemistry of dentin bonding systems have improved short-term bond strength, however the long-term clinical outcome of esthetic restorations bonded to dentin is still unclear. Dentin is a challenging substrate for bonding due to its heterogeneity. Regional differences,¹ response to caries and other stimuli such as bruxism or mechanical abrasion,² presence of moisture³⁻⁵ all affect directly the quality and strength

of dentin bonding. A key factor for the clinical outcome of bonded esthetic restorations is the ability of the adhesive system to prevent the opening of interfacial gaps, since this is an irreversible process that frequently leads to microleakage.^{6,7}

Self-etching primers have been developed in attempt to simplify bonding procedures, and to prevent discrepancies between the depth of dentin demineralized by the acid and the ability of the primer to penetrate this demineralized layer.^{8,9} They utilize weaker acids that have been shown to remove partially the smear layer, maintain the smear plugs, and to create thin hybrid layers.^{8,9} The use of weaker acids may influence the ability of these systems to provide consistently good interfacial seal, since the thickness of smear layer generated at the dentin surface is influenced by variables such as the type of bur, the speed, and the amount of water irrigation used during cavity preparation.¹⁰⁻¹²

A further development of the concept of self-etching primers, the self-etching adhesives or self-etching primer adhesives was recently introduced.^{9,13} These materials have incorporated all the components of bonding systems (acidic conditioner, hydrophilic primer, and hydrophobic adhesive resin) into one bottle and are the first true "one-step agents". This simplifies the technique for dentin bonding, reducing operating time and perhaps making dentin bonding a less sensitive technique. Prompt L-Pop is a self-etching primer adhesive^{14,15} that was initially developed for use with compomers. The manufacturer has recently expanded the indications of Prompt-L-Pop suggesting its use for bonding composite resin restorations too. Data from the manufacturer indicates that this system allows for adequate bond strengths to both enamel and dentin. However, little is known about the mechanism of dentin bonding, as well as the quality of the interfacial seal between esthetic restorative materials and dentin substrate. Therefore, we designed this study to evaluate the quality of the interfacial seal and the micromorphology of the hybrid layer in composite resin, compomer, and resin-modified glass ionomer restorations bonded with Prompt-L-Pop to the dentin of primary or permanent teeth.

Methods

Fifteen permanent and 15 primary caries-free molars were used in this study. All teeth were thoroughly cleaned immediately after extraction and stored in an aqueous solution of 0.2% thymol for up to 3 months. The crowns were separated from the roots 2–3 mm apical from the CEJ, and the pulp tissue was

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removed with a dental explorer. The superficial dentin (within 1-2 mm of the DEJ at the central groove) of the occlusal surfaces was exposed by a flat cut perpendicular to the long axis of the tooth with a carbide bur # 331L in high speed with copious water spray. The occlusal surfaces were then divided in 2 halves by a 1 mm deep groove in the labial-lingual orientation to allow for evaluation of the experimental adhesive system (Prompt-L-Pop, ESPE America, Norristown, PA), and a control system in the same specimen.¹⁶

Primary and permanent teeth were divided in 3 groups and restored with either a composite resin (Filtek Z250, 3M, St. Paul, MN), a compomer (Hytac, ESPE), or a resin-modified glass ionomer (Vitremmer, 3M). Each tooth was bonded in one half of the occlusal surface with the self-etching primer adhesive system Prompt-L-Pop; and in the other half with the adhesive system that is recommended by the manufacturer for the corresponded restorative material used. The only exception was the group restored with Hytac that was bonded with Single Bond as the control system, due to the fact that Prompt-L-Pop is recommended by ESPE as the adhesive system for Hytac. The following was the protocol used for each group:

Group 1: One half of the occlusal surface was conditioned with 35% phosphoric acid (3M) for 15 s, thoroughly washed for 20 s, and air dried for 5 s. One layer of Single Bond was applied with a microbrush and polymerized for 10 s. Then, a second layer of Single Bond was applied. Prompt-L-Pop (ESPE) was brushed for 15 s to the other half of the occlusal surface. Both, the second layer of Single Bond and the Prompt-L-Pop were polymerized together for 10 s. Filtek Z250 was applied in 2 x 2 mm increments and polymerized for 40 s per increment until all occlusal surface was restored with a flat, 4 mm thick layer of composite resin.

Group 2: Teeth received the same bonding regimen as group 1 but were restored with the compomer Hytac (ESPE).

Group 3: One half of the occlusal surface was treated with Vitremmer Primer (3M), gently air dried for 15 s, and polymerized for 20 s. Prompt-L-Pop was brushed for 15 s to the other half of the occlusal surface, and polymerized for 10 s. Vitremmer was applied in 2 x 2 mm increments and polymerized for 40 s per increment until all occlusal surface was restored with a flat, 4 mm thick layer of resin-modified glass ionomer.

All teeth were then embedded in acrylic resin (L.D. Caulk Co., Mildford, DE), and the restorative material-dentin interface was exposed by fracturing the specimens with a surgical blade¹⁶. Teeth were prepared for scanning electron microscopy as previously described.^{16,17} Specimens were analyzed in a scanning electron microscope (model 1000B, Amray, USA) with acceleration

voltage of 15.0 kV. Two calibrated examiners (PDST and JEN) analyzed the interfaces and a consensus was reached for each measurement. The examiners were blinded for group assignment at the time of microscopic evaluation. The thickness of the hybrid layer and the width of interfacial gaps were measured in 5 sites of 5 independent teeth per condition and tooth type. The first measurement of the interfacial gap was made at about 300 μm from the center of the crown, and the remaining were made in intervals of approximately 100 μm from the initial measurement.¹⁶ The qualitative assessment of the interfaces was performed in the same sites that were analyzed for measurement of the gap width and hybrid layer thickness. To control for changes in gap width caused by the positioning of the specimen inside the SEM chamber, all samples were analyzed in a standard angulation of 15-18 degrees (base of the stubs in relation to horizontal plane).¹⁶

Statistical Analysis

Repeated-measures ANOVA was used for the analysis of dentin bonding system (Prompt-L-Pop or controls) and its relation to tooth type (primary or permanent teeth). The need for this analysis was determined by the use of ANOVA that demonstrated a significant effect of the type of dentin bonding system used on the width of the interfacial gap. Once the significance of the bonding system had been established, repeated-measures ANOVA was performed to test the hypothesis of main group effects (tooth type) on the width of the interfacial gap. Paired t-tests were used to compare the width of the gap generated by Prompt-L-Pop and control bonding system for each restorative material (i.e. Filtek Z250, Hytac, or Vitremmer). The significance of the data was determined at p<0.05.

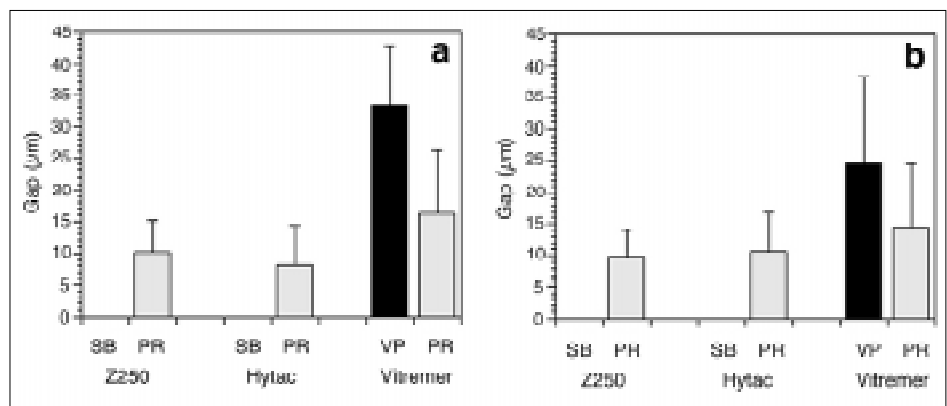


Fig 1. Width of the interfacial gaps in permanent (a) and primary (b) teeth. Values represent the average (\pm s.d.) of interfacial gaps measured under SEM (at 1,500x) in 25 fields from 5 independent teeth per condition. Restorations were bonded with Single Bond (SB), Prompt-L-Pop (PR), or Vitremmer Primer (VP).

Table 1. Qualitative Assessment of the Interfacial Seal. Partially Sealed Interfaces Were Characterized by Areas of Gap Intercalated by Areas of Sealed Restorative Material-Dentin Interface.

	Filtek Z250		Hytac		Vitremmer	
	Single Bond	Prompt-L-Pop	Single Bond	Prompt-L-Pop	Vitremmer Primer	Prompt-L-Pop
Opened	0	8	0	7	9	5
Partially Sealed	0	1	0	2	1	0
Sealed	10	1	10	1	0	5
Total	10	10	10	10	10	10

Table 2. Width of the Interfacial Gaps in Permanent and Primary Teeth According to Dentin Bonding System. Values Represent the Average(+ s.d.) of Interfacial Gaps Measured under SEM (at 1,500x) in 25 Fields

	Z250		Hytac		Vitremer	
	Single Bond	Prompt-L-Pop	Single Bond	Prompt-L-Pop	Vitremer Primer	Prompt-L-Pop
Permanent Teeth	0 (± 0) ^a	10.2 (± 6.9) ^b	0 (± 0) ^a	8.0 (± 8.3) ^b	33.2 (± 11.4) ^c	16.3 (± 19.0) ^c
Primary Teeth	0 (± 0) ^a	9.9 (± 4.6) ^b	0 (± 0) ^a	10.6 (± 7.3) ^b	24.4 (± 17.7) ^c	14.5 (± 19.9) ^c

^{a,b,c} The width of the gap in groups with the same letter in superscript were not statistically different at $P < 0.05$.

Results

Each specimen was examined by scanning electron microscopy to measure the interfacial gap between restorative material and dentin (Fig. 1), and to determine the quality of the interfacial seal (Table 1). The quantitative analysis of the interfacial gap demonstrated the presence of gaps (Table 2) when Prompt-L-Pop was used for bonding a composite resin (Z250), a compomer (Hytac), and a resin-modified glass ionomer (Vitremer). For Z250 and Hytac, the average gap observed with Prompt-L-Pop was larger ($p < 0.05$) than with the adhesive system Single Bond in permanent (Fig. 1a and Table 2) and

Hytac (Fig. 5). There was a tendency for thicker hybrid layers in primary teeth compared to permanent teeth restored with Single Bond, however this difference was not significant ($p > 0.05$). Only one resin-modified glass ionomer restoration bonded with Vitremer Primer presented a visible hybrid layer that had an average thickness of $3.72 \mu\text{m}$ (Fig. 5b).

Discussion

Self-etching primers have been developed to simplify bonding procedures and to decrease the sensitivity of the technique for bonding to tooth structures. One of the basic concepts behind self-etching primers is that the demineralization of tooth structure and the diffusion and embedding of the bonding agent around dentinal collagen fibers happens at the same time and to the same depth. Prompt-L-Pop has been recently introduced in the market as a promising material that combines the conditioner, primer, and adhesive resin in one single solution, allowing for a true one-step bonding technique for compomers and composite resins. This *in vitro* study was designed to examine the interfacial micromorphology of esthetic restorations bonded with Prompt-L-Pop to primary and permanent dentin substrates. This study found that compomer and composite resin restorations bonded with Prompt-L-Pop present larger and more frequent interfacial gaps than control restorations bonded with the conventional adhesive system Single Bond.

signs of a hybrid layer. Prompt-L-Pop did not create a visible hybrid layer in teeth restored with Filtek Z250, and only in few teeth (3/10) restored with Hytac (Fig. 5). When present, the thickness of the hybrid layer originated by Prompt-L-Pop was similar in primary and permanent teeth ($p > 0.05$). In contrast, Single Bond originated hybrid layers in all teeth restored with Filtek Z250 and

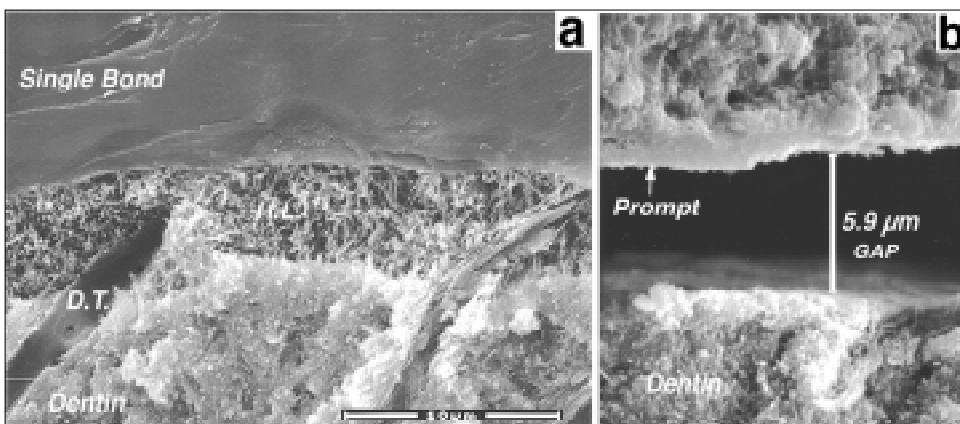


Fig 2. Micromorphology of representative Filtek Z250-dentin interfaces bonded with Single Bond (a) or Prompt-L-Pop (b). Well-diffused hybrid layer (H.L.) is observed in specimen bonded with Single Bond (a). Observe lack of visible hybrid layer in specimen bonded with Prompt-L-Pop. SEM photomicrographs are at 4,000x magnification. Dentin tubule (D.T.).

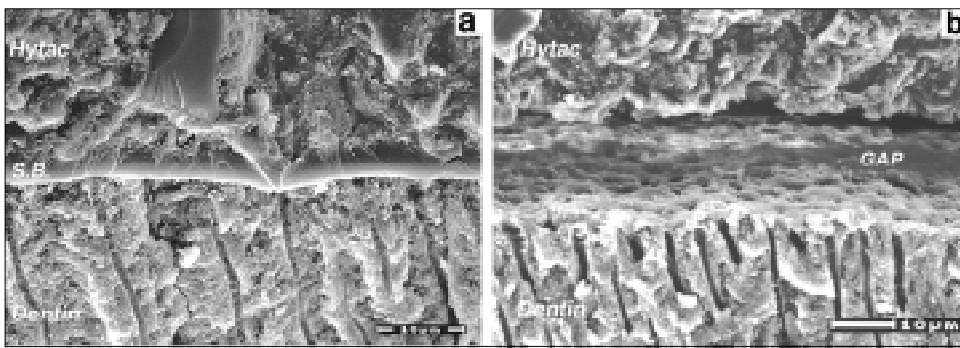


Fig 3. Micromorphology of representative Hytac-dentin interfaces bonded with Single Bond (a) or Prompt-L-Pop (b). Well-sealed interface is observed in specimen bonded with Single Bond (SB) (a). The specimen restored with Prompt-L-Pop presents a wide interfacial gap, without visible hybrid layer (b). Openings of dentinal tubules are visible inside the Prompt-L-Pop-dentin interface (b). SEM photomicrographs are at 2,000x magnification.

The role of dentin bonding for the successful outcome of esthetic restorations goes much beyond retention of restorative material to the cavity preparation. Perhaps more importantly, dentin bonding has to be strong and stable

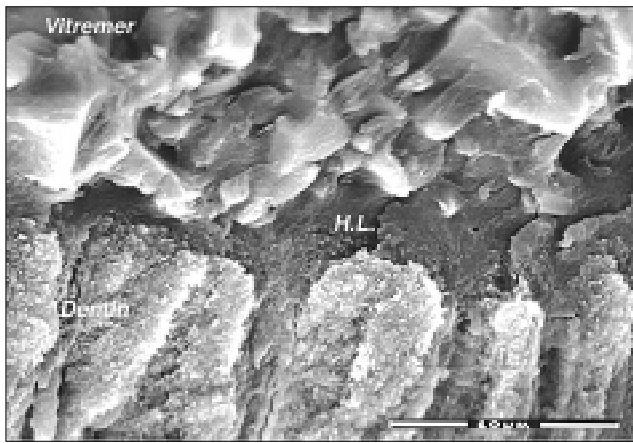


Fig 4. Micromorphology of a Vitremer-dentin interface bonded with Prompt-L-Pop. Well-diffused hybrid layer (H.L.) with a sealed interface can be observed in this specimen. SEM photomicrograph is at 4,000x magnification.

enough to counteract polymerization shrinkage of resin-containing dental materials and to provide adequate interfacial seal for these restorations.^{20,21} This is necessary for adequate pulp protection, reduction of microleakage and post-operative sensitivity,²² and to prevent the incidence of recurrent decay when the cavo-surface margin is in dentin.²³

The first criteria that was examined in this study was the ability of Prompt-L-Pop to generate sealed interfaces of esthetic restorative materials and dentin. The design of this study included a “traditional” dentin bonding protocol for each restorative material as an internal control for Prompt-L-Pop-bonded interfaces. Therefore, each tooth received two different bonding protocols, allowing for the direct comparison of the experimental material (Prompt-L-Pop) against a “gold-standard”. This design reduced the influence of tooth-related biases such as dentin depth and tooth age, and methodological biases such as effects of forces generated during fracture of specimens or their exposure to the vacuum necessary for SEM analysis.

The analysis of the data obtained for the incidence and size of interfacial gaps demonstrated no significant differences between primary and permanent teeth. Therefore, was pooled the data from primary and permanent teeth for the qualitative assesment of the bonded interfaces. The results obtained in compomer and composite resin restorations demonstrate that Single Bond generates significantly better sealed interfaces than Prompt-L-Pop. In each tooth restored with composite resin, the half of the interface that was bonded with Single Bond was completely sealed. When we evaluated the opposite half of the same teeth (bonded with Prompt-L-Pop), we found that the interface was opened in the

majority of the teeth evaluated. A similar trend was observed with the compomer Hytac. Only one tooth presented the interface bonded with Prompt-L-Pop sealed, while in all teeth the interface bonded with Single Bond was found to be sealed.

The average size of the gap in composite resin and compomer restorations in the interfaces bonded with Prompt-L-Pop was 10 μm . The same teeth presented no visible gap in the area bonded with Single Bond when examined by SEM. The authors are aware of the consequences to bonded interfaces of desiccating the specimens and using vacuum for SEM. While the absolute number obtained for these gaps may be exacerbated and not reflect the *in vivo* situation, the comparative analysis of both materials placed in the same tooth and seen at the same time under SEM allows us to conclude that the Prompt-L-Pop generates poorer seal of the composite resin or compomer-bonded interfaces than Single Bond.

Prompt-L-Pop has not been indicated for bonding of resin-modified glass ionomer restorations. However, it is still unclear which is the technique that would result in sealed resin-modified glass ionomer-dentin interfaces, since several studies have shown significant microleakage in these restorations.²⁴⁻²⁶ We have previously reported that Vitremer Primer does not provide a consistently sealed Vitremer-dentin interface.¹⁸ Here we investigated the ability of Prompt-L-Pop to improve the quality of Vitremer-dentin interfaces. The use of Prompt-L-Pop resulted in 5 interfaces considered sealed and 5 that were considered opened. In contrast, none of the surfaces conditioned with Vitremer Primer was considered sealed in this *in vitro* study. Therefore, restorations with Vitremer represented the best comparative results for Prompt-L-Pop despite the fact that this material has not been indicated for resin modified glass ionomers.

The development of a hybrid layer, an intermixture of dentin collagen and diffusible components of the adhesive system, is a determinant of the clinical outcome of restorations bonded to dentin.¹⁸ Therefore, we examined the presence and thickness of the hybrid layer in attempt to understand the reasons for the poor results obtained with Prompt-L-Pop. We found that all teeth restored with composite resin had a visible hybrid layer in the area bonded with Single Bond and no hybrid layer in the area bonded with Prompt-L-Pop. Only one out of five primary molars and two out of five permanent molars

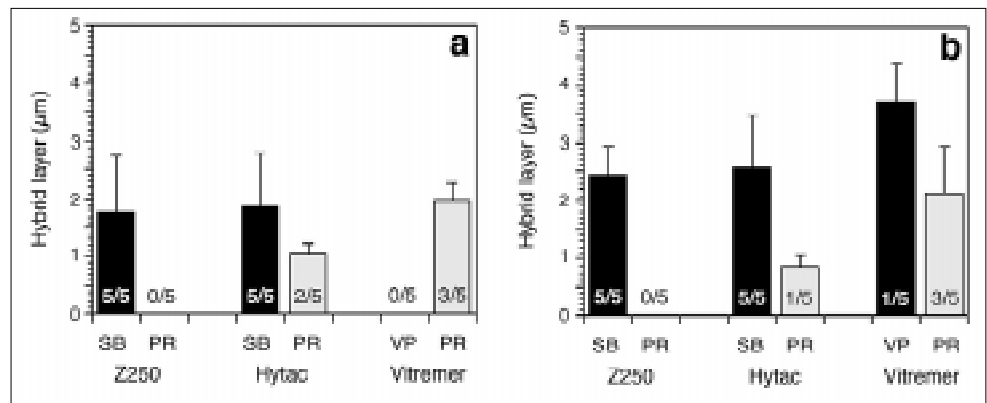


Fig 5. Width of the hybrid layer in permanent (a) and primary (b) teeth. Restorations were bonded with Single Bond (SB), Prompt-L-Pop (PR), or Vitremer Primer (VP). Values represent the average (\pm s.d.) of hybrid layer width measured under SEM (at 4,000x) in 5 fields per tooth. The numbers at the bottom of the columns indicate the number of teeth evaluated per condition (only interfaces with visible hybrid layers were included here).

bonded with Prompt-L-Pop and restored with compomer had visible hybrid layer. It is unclear why hybrid layers were not observed in most teeth restored with Prompt-L-Pop. The intrinsic nature of a self-etching primer adhesive system, where resin monomers are expected to be completely polymerized in an acidic environment, may have contributed for these findings. We speculate that the low pH of Prompt-L-Pop, which is required for the etching of tooth structure, may have impaired the polymerization of the resin monomers and therefore not allowed for the development of a strong and stable hybrid layer to prevent the opening of interfacial gaps. The hybrid layers obtained with Single Bond were thicker in primary teeth compared to permanent teeth, but this difference was not statistically significant. These results are in line with what we have previously reported for All-Bond 2 (Bisco) and Scotchbond Multi-Purpose (3M),¹⁶ however the mixing of the hydrophilic primer and hydrophobic adhesive resin in one single solution may have attenuated the effect of dentin type on the thickness of the hybrid layer. Further investigations are necessary to confirm this hypothesis.

Prompt-L-Pop originated visible hybrid layers more frequently when Vitremer was used as restorative material, which may explain why the interfaces were better sealed in these teeth. Six out of ten teeth had hybrid layers that measured in average 2 μm . In contrast, a hybrid layer could only be seen in one interface bonded with Vitremer Primer. Adequate sealing of interfaces between resin-modified glass ionomers and dentin is necessary to reduce microleakage. This should be achieved without interference on the ability of these materials to exchange fluoride ions with tooth structure and confer protection against the progression of demineralization. The protocols used today for dentin bonding with resin-modified glass ionomers do not seem to fulfill these objectives. While Prompt-L-Pop seems to provide better interfaces than Vitremer Primer in this study, it did so in only a half of the specimens evaluated. Therefore, further *in vitro* and *in vivo* investigations seem timely to determine which protocol for dentin bonding results in adequate interfacial seal and long-term clinical outcome for resin-modified glass ionomers.

Prompt-L-Pop is a ground breaking material that incorporated all elements of contemporary adhesive systems in one solution, resulting in the first true "one-step" agent for enamel and dentin bonding. Its formulation resulted in considerable decrease in the time necessary for bonding procedures. This might be beneficial for bonding teeth that can not be adequately isolated, for pediatric dentistry cases, and it is certainly appealing to the busy practitioner. In theory, the combination of the demineralizing agent with the hydrophilic primers should allow for a completely diffused hybrid layer that provides a strong and stable bonding to dentin. While conceptually sound, Prompt-L-Pop did not perform as well as the control adhesive system (Single Bond) used for composite resin and compomer restorations in this study. We observed that most interfaces of composite resins and compomers bonded with Prompt-L-Pop did not present a visible hybrid layer under SEM, and reasoned that this might be a potential explanation for the frequent presence of gaps in these interfaces. These findings suggest that Prompt-L-Pop will have a poorer clinical outcome compared

to bonding systems that have the etchant and the primer/bonding solutions in two separate vials.

Conclusions

Within the parameters of this *in vitro* study it is concluded that:

1. The quality of the interfacial seal was similar in primary and permanent teeth when a self-etching primer adhesive system was used for dentin bonding.
2. Dentin conditioning with a phosphoric acid followed by the application of a conventional adhesive resin (Single Bond) provided consistently better sealed compomer- and composite resin-dentin interfaces as compared to the use of a self-etching primer adhesive system (Prompt-L-Pop).
3. The pre-treatment of dentin with the self-etching primer adhesive system (Prompt-L-Pop) for bonding resin-modified glass ionomer (Vitremer) restorations resulted in better sealed interfaces as compared to the use of Vitremer primer.

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ABSTRACT OF THE SCIENTIFIC LITERATURE



Marginal adaptation of Class V restorations

A major problem of light-cured restorations is lack of marginal adaptation that can be attributed to shrinkage of the restorative material during polymerization. The goal of this *in vitro* study was to test the influence of "softstart polymerization (prepolymerization at a low light intensity followed by a final cure at high light intensity) on the marginal integrity of Class V esthetic restorations. Cavities were restored with a composite resin (Spectrum + Prime & Bond), or with a polyacid-modified resin (Dyract + Prime & Bond, or Hytac + OSB Primer). The restorations were either conventionally cured (40 seconds, 800 mW/cm²), or they were cured with the "softstart technique", i.e. lower starting intensity (10 seconds, 150 mW/cm²) followed by full intensity (30 seconds, 800 mW/cm²). The authors found that "softstart polymerization" did not improve the marginal adaptation of composite resin or polyacid-modified resin (compomer) restorations. The best marginal adaptation at the enamel/restoration interface was obtained with the composite resin. In contrast, the two polyacid-modified resins showed superior marginal adaptation at the dentin/restoration interfaces.

Comments: This study demonstrates that "softstart-polymerization" using a very low start curing light intensity does not provide better marginal adaptation in Class V composite resin and polyacid-modified resin (compomer) restorations. JN

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Marginal adaptation of Class V restorations with and without "softstart-polymerization. Friedl KH, Schmalz G, Hiller KA, Märkl A. *Oper Dent* 25: 26-32; 2000.

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ABSTRACT OF THE SCIENTIFIC LITERATURE



Factors affecting shear bond strength of composite resin

Enamel fluorosis may affect the bond strength of composite resins. The objective of this *in vitro* study was to determine the effects of age, severity of fluorosis, and etching time on the shear bond strength of direct composite resins to human permanent tooth enamel. The data presented demonstrate that the severity of fluorosis had no effect on shear bond strength. The authors also found that the enamel-composite resin bond was stronger in teeth from patients <40 years old than in teeth from 40+ years old patients. Interestingly, in younger patients the bond strength was significantly higher when the enamel was etched for 120 seconds with 35% phosphoric acid as compared to enamel etched for 60 seconds.

Comments: This study demonstrates that increasing the etching time from 60 to 120 seconds results in a significant increase in bond strength of composite resins bonded to fluorosed enamel in younger patients. JN

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Factors affecting shear bond strength of composite resin to fluorosed human enamel. Ateyah N, Akpata E. *Oper Dent* 25: 216-222; 2000.

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