Bond Strength and Etching Pattern of Adhesive Systems to Enamel: Effects of Conditioning Time and Enamel Preparation

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ABSTRACT

Statement of the problem: The performance of self-etch systems on enamel is controversial and seems to be dependent on the application technique and the enamel preparation.

Purpose of the Study: To examine the effects of conditioning time and enamel surface preparation on bond strength and etching pattern of adhesive systems to enamel.

Materials and Methods: Ninety-six teeth were divided into 16 conditions ($N = 6$) in function of enamel preparation and conditioning time for bond strength test. The adhesive systems OptiBond FL (Kerr, Orange, CA, USA), OptiBond SOLO Plus (Kerr), Clearfil SE Bond (Kuraray, Osaka, Japan), and Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) were applied on unground or ground enamel following the manufacturers' directions or doubling the conditioning time. Cylinders of Filtek Flow (0.5-mm height) were applied to each bonded enamel surface using a Tygon tube (0.7 mm in diameter; Saint-Gobain Corp., Aurora, OH, USA). After storage (24 h/37°C), the specimens were subjected to shear force (0.5 mm/min). The data were treated by a three-way analysis of variance and Tukey's test ($\alpha = 0.05$). The failure modes of the debonded interfaces and the etching pattern of adhesives were observed using scanning electron microscopy.

Results: Only the main factor "adhesive" was statistically significant ($p < 0.001$). The lowest bond strength value was observed for OptiBond FL. The most defined etching pattern was observed for 35% phosphoric acid and for Adper Prompt L-Pop. Mixed failures were observed for all adhesives, but OptiBond FL showed cohesive failures in resin predominantly.

Conclusions: The increase in the conditioning time as well as the enamel pretreatment did not provide an increase in the resin–enamel bond strength values for the studied adhesives.

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CLINICAL SIGNIFICANCE

The surface enamel preparation and the conditioning time do not affect the performance of self-etch systems to enamel.

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INTRODUCTION

The contemporary adhesive systems available in the market employ either the etch-and-rinse or the self-etch techniques to interact with dental substrate.[1] While the etch-and-rinse approach requires a separate acid-etch step to promote dentin and enamel demineralization before monomer infiltration, demineralization and infiltration occur simultaneously in the self-etch approach,[2] although with no perfect synchronism.[3] The self-etch adhesives can be classified into mild (pH > 2), moderate (1 < pH < 2), or strong (pH < 1),[4] depending on their composition and concentration of polymerizable acids and/or acid resin monomers.

The elimination of the separate etching and rinsing steps has made the bonding protocol easier to perform, which explains why these materials have become very popular among clinicians. According to some authors, the reduction in the number of steps involved in the bonding protocol minimizes the contamination risks[5] and the sources of errors. Besides that, the wet bonding technique is no longer required,[6] making them less technique- and operator sensitive. As the smear layer is not removed but is incorporated into the hybridized complex,[7] these materials are believed to result in less post-operative sensitivity; however, this assumption has not been supported by recent clinical studies.[8,9]

In spite of the handling advantages of self-etch systems, one cannot deny that enamel etching with phosphoric acid is still considered the gold standard against which new products should be tested. The available articles investigating the performance of self-etch systems in enamel are controversial because several factors such as the acidity of the adhesives, the method of application, and the bond strength test vary among studies. While some authors have reported superior performance of more acidic systems,[10,11] others have shown that only mild self-etch systems can provide bond strength values as high as those of etch-and-rinse adhesives.[12]

Another confounding factor among studies is the condition of enamel prior to bonding with self-etch systems. The grinding of enamel is usually performed with SiC paper or diamond burs in order to eliminate the less-reactive aprismatic enamel layer.[12] No significant difference in resin–enamel bond strengths between etch-and-rinse and self-etch adhesives was reported in one study when these materials were applied either in unground or in ground enamel.[13] However, this finding is not consensual. High resin–enamel bond strength values have been found for self-etch systems applied in ground enamel[7,14,15] although not as high as those achieved by etch-and-rinse adhesives.[16]

It is also worth mentioning that there are cases where bonding should be made on intact enamel, such as bonding of orthodontic brackets or conservative and preventive restorative measures. In these cases, an increase in the conditioning time of self-etch solutions could be a way to improve the self-etch adhesives' performance on unground enamel. However, so far, few studies have evaluated this clinical approach.[10,17]

Among the bond strength tests employed to measure the interfacial strength between resin and enamel, the conventional shear and tensile test followed by the microtensile test are the most common testing approaches. However, the recently developed microshear testing[18–21] can be another option because it takes the advantage of employing shear forces that are present in most clinical scenarios.[22]

Based on that, the aim of the this investigation was to evaluate the resin–enamel bond strength and the micromorphology of adhesive systems as function of enamel preparation and conditioning time. The null hypothesis to be tested is that the bond strength values and the etching pattern of all adhesives systems will be similar, regardless of enamel surface preparation and conditioning time.

MATERIALS AND METHODS

This study was approved by the Institutional Review Board from the Dental School under protocol number 228/04. One hundred sixteen extracted third molars were immersed in 0.5% chloramine at 4°C for 7 days[23] before the beginning of the laboratorial setting. Ninety-six teeth were used for bond strength measurements and 20 teeth were used for etching pattern evaluation.

Microshear Bond Strength

Ninety-six teeth were randomly divided into 16 experimental conditions ($N = 6$) that resulted from the combination of the factors "adhesive" (four levels), "enamel treatment" (two levels), and "conditioning time" (two levels). Each tooth was divided into buccal-to-lingual and mesio-to-distal directions with a diamond saw in an Isomet 1,000 machine (Buehler, Lake Bluff, IL, USA) in order to obtain 24 fragments per group. Each fragment was embedded in a polyvinyl chloride (PVC) tube (10-mm height × 13-mm diameter) using a chemically cured acrylic resin (Jet Clássico, São Paulo, Brazil). The buccal or lingual surfaces were maintained perpendicular to the horizontal plane.

Four adhesive systems with different bonding approaches were evaluated in this study. OptiBond FL and OptiBond SOLO Plus (Kerr, Orange, CA, USA), three- and two-step etch-and-rinse adhesive systems, respectively, were used. Clearfil SE Bond (Kuraray, Osaka, Japan), a two-step self-etch system, and Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA), a one-step self-etch system, were also selected. Clearfil SE Bond is a mild self-etch system, while Adper Prompt L-Pop is a strong self-etch system.[24,25] The composition, mode of application, and batch number of these materials are described in Table 1.

The adhesive systems were applied on ground and unground enamel surfaces either following the manufacturer's directions or after duplicating the conditioning time of the etchant. The grinding of enamel was performed with a wheel medium-grit diamond bur (#4142, particle size ca. 100 µm, KG Sorensen, Barueri, São Paulo, Brazil) using a high-speed handpiece with a water coolant. This procedure created 0.5-mm deep grooves[14] on the surface, which were flattened with a tapered round-end fine-grit diamond bur (#4138, particle size ca. 46 µm, KG Sorensen). Both ground and unground enamel were subjected to tooth prophylaxis with pumice slurry before the bonding protocol.

After applying the adhesive to the enamel, a microbore tube (Tygon tubing, S-54-HL, Saint-Gobain, USA) 0.7 mm in diameter and 0.5 mm in height was placed on the flattest area of the enamel fragment and the adhesive was light cured (10 seconds), thereby fixing the tube to the enamel surface and defining the bonding area.[19,20,26] Resin composite (Filtek Flow, shade B2, 3M ESPE) was placed into the tubes[19] and was light cured for 40 seconds (QHL75 Curing Light, Dentsply, Rio de Janeiro, Brazil) with 600 mW/cm^2 of irradiance. The specimens were stored in water at 37°C for 24 hours.[18,27–29] The Tygon tubes were carefully removed and the flash of resin extending beyond the base of the resin cylinder was removed

TABLE 1. ADHESIVE SYSTEMS, COMPOSITION, AND DIRECTIONS FOR USE.

Material (Batch Number)	Composition	Manufacturers' Directions
OptiBond FL (405161/407886)	37.5% phosphoric acid gel Primer: HEMA, GPDM, camphorquinone, ethanol, water Bond: Bis-GMA, HEMA, GDM, camphorquinone, Ba-Al borosilicate glass, disodium hexafluorsilicate, fumed silica (48% by weight)	1. Etch with 37.5% phosphoric acid (15 seconds) 2. Rinse for (15 seconds) and dry (5 seconds) 3. Apply primer and rub for 15 seconds. Dry for 5 seconds 4. Apply adhesive in a uniform thin layer 5. Light cure for 30 seconds (600 mW/cm^2)
OptiBond SOLO Plus (014088)	37.5% phosphoric acid gel Primer: HFGA-GDM, GPDM, ethanol, MEHQ, ODMAB, camphorquinone Bond: Bis-GMA, HEMA, GDM, GPDM, camphorquinone, ethanol, barium glass, sodium hexafluorsilicate, fumed silica	1. Etch with 37.5% phosphoric acid (15 seconds) 2. Rinse (15 seconds) and dry (5 seconds) 3. Apply the adhesive and rub for 15 seconds. Dry for 3 seconds 4. Light cure for 20 seconds (600 mW/cm^2)
Clearfil SE Bond (525AA/614AA)	Primer: MDP, HEMA, camphorquinone, hydrophilic dimethacrylate, N,N-diethanol p-toluidine, water Bond: MDP, bis-GMA, HEMA, hydrophobic aliphatic dimethacrylate, camphorquinone, N,N-diethanol p-toluidine, silanated colloidal silica (filled 10% by weight)	1. Apply SE primer to enamel and leave it for 20 seconds 2. Dry throughout with a mild air flow (10 seconds) 3. Apply bond (10 seconds) 4. Light cure for 10 seconds (600 mW/cm^2)
Adper Prompt L-Pop (195301)	HEMA phosphates provide the acidic component, with HEMA, bis-GMA, and a modified polyalkenoic acid providing the resin components; water	1. Apply adhesive to the entire surface, rubbing with moderate finger pressure for 15 seconds 2. Gently air-dry with an air stream (10 seconds) 3. Repeat as necessary 4. Light cure for 10 seconds (600 mW/cm^2)

HEMA = 2-hydroxyethyl methacrylate; Bis-GMA = bisphenolglycidyl methacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; GPDM = glycerophosphate dimethacrylate; GDM = glycerodimethacrylate; HFGA-GDM = hexafluoroglutaric anhydride-glycerodimethacrylate adduct; MEHQ = 4-methoxyphenol; ODMAB = 2-(ethylhexyl) 4-(dimethylamino) benzoate; SE = Clearfil SE Bond.

with a blade. Then, the resin/enamel adaptation was checked with a stereomicroscope (×10 magnification) to discard specimens with air bubbles or gaps evident at the interface.

A universal testing machine (model 5565, Instron, Canton, MA, USA) was used for the test. Each PVC tube containing the bonded specimens was attached to the testing device, which, in turn, was placed in the testing machine. A thin wire (0.2-mm diameter, Morelli Ortodontia, São Paulo, Brazil) was looped around the composite resin cylinder, around half its circumference, and was gently held flush against the enamel, at the resin–enamel interface. The resin–enamel interface, the wire loop, and the center of the load cell were aligned as straight as possible to ensure the correct application of the shear force. The force was applied to each specimen at a crosshead speed of 0.5 mm/min until failure. The value required to cause the

TABLE 2. MEANS AND STANDARD DEVIATIONS (MPa) OF RESIN–ENAMEL MICROSHEAR BOND STRENGTH FOR ALL EXPERIMENTAL GROUPS.

	Unground Enamel		Ground Enamel	
Adhesive	Recommended	Double	Recommended	Double
FL	14.6 ± 1.6	14.9 ± 4.7	13.9 ± 2.5	14.2 ± 4.8
SO	23.3 ± 2.0	23.3 ± 3.9	24.6 ± 3.5	23.2 ± 3.0
SE	20.3 ± 4.2	22.4 ± 5.1	24.3 ± 3.6	23.1 ± 3.0
AD	25.1 ± 3.3	21.6 ± 3.2	21.7 ± 4.0	21.0 ± 3.8

FL = OptiBond FL; SO = OptiBond Solo Plus; SE = Clearfil SE Bond; AD = Adper Prompt L-Pop.

debonding was then divided by the bonded area of the tygon tube, and the bond strength values were expressed in MPa. The data were subjected to a three-way analysis of variance[30] and Tukey's test ($\alpha = 0.05$).

Etching Pattern

Twenty molars were sectioned in order to obtain 40 enamel fragments, in the same way described for the microshear test. Two fragments were assigned for each experimental condition and for the prophylaxis and bur treatment for comparison purposes. A deep lingual slit was prepared with a diamond bur (7020, KG Sorensen) to facilitate subsequent fracture of the etched surfaces. All enamel fragments were subjected to prophylaxis with pumice and water. Half of them were ground as described for the microshear test.

Enamel surfaces were then conditioned either with phosphoric acid or with the self-etch adhesives. The former was rinsed with water for 15 seconds. In the latter, after application of the self-etch primers, the enamel surfaces were rinsed with ethanol and acetone[31,32] to remove the monomers. After that, the same specimens were gently split with a hammer and scapel blade along the preformed slits to provide a sagittal view of the etched enamel. The specimens were stored in a desiccator containing silica gel for 12 hours. After that, they were mounted on aluminum stubs with colloidal silver and were sputter coated with gold/palladium (Bal-Tec SCD 050 Sputter Coater, Bal-Tec, Balzers, Liechtenstein) to be observed under a scanning electron microscope (Philips XL30, Koninklijke Philips Electronics N.V., Eindhoven, the Netherlands) at 15 kV of accelerating voltage. The buccal or lingual etched surfaces as well as the sagittally fractured surfaces of the same tooth were examined.

Failure Mode

After debonding, all fractured samples were sputter coated with gold/palladium and were analyzed under a scanning electron microscope (JEOL 5600LV, JEOL Ltd., Tokyo, Japan) at 15 kV of accelerating voltage, and the failures were classified as adhesive (at the enamel/resin interface, but the substrates were observed individually at different surfaces), cohesive (within enamel or resin substrates), and mixed (when enamel and resin appeared in the same analyzed surface).

RESULTS

The overall bond strength means and standard deviations are depicted in Table 2. The three-way analysis of variance showed that there was no significant effect for the interactions ($p = 0.215$), enamel surface preparation ($p = 0.945$), and conditioning time ($p = 0.501$). Only the main factor adhesive was statistically significant ($p < 0.001$). Therefore, the data for each adhesive were joined to obtain an overall mean for each adhesive regardless of enamel treatment or conditioning time. The mean bond strengths values of Clearfil SE Bond, Adper Prompt L-Pop, and OptiBond SOLO Plus were statistically similar ($p > 0.641$) and different from the mean bond strength of OptiBond FL ($p < 0.0001$). Based on these results, a model including adhesive as a factor, where Clearfil SE Bond, Adper Prompt L-Pop,

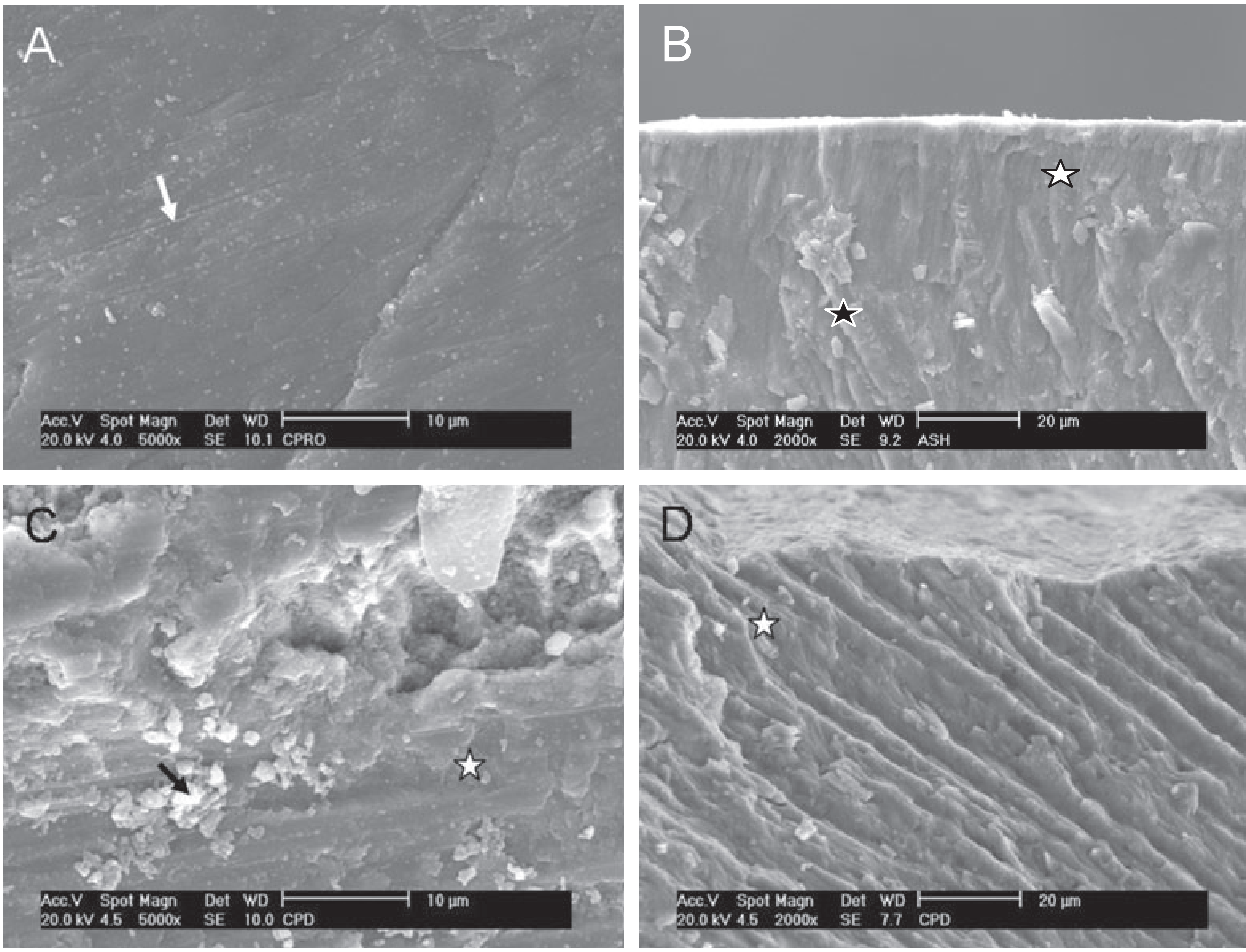

Figure 1. A,B, *Unground enamel after cleaning with slurry of pumice and water.* A, *Surface view (bar = 10 µm): smooth, with some grooves created by the slurry of pumice and water (white arrow).* B, *Sagittal view (bar = 20 µm): aprismatic (white star) and prismatic (black star) enamel.* C,D, *Diamond bur-treated enamel.* C, *Surface view (bar = 10 µm): enamel grooves caused by the diamond bur can be seen (white star); smear layer (black arrow).* D, *Sagittal view (bar = 20 µm): prismatic enamel reaching the surface (white star).*

and OptiBond SOLO Plus were joined, was fitted, and the mean bond strength values and standard error for the studied adhesives were 14.4 ± 0.7 MPa for OptiBond FL and 22.8 ± 0.4 MPa for Clearfil SE Bond, Adper Prompt L-Pop, and OptiBond SOLO Plus.

Etching Pattern

The surface of the enamel after prophylaxis is predominantly smooth (Figure 1A,B). The diamond bur treatment resulted in an exposure of enamel prisms (Figure 1C,D). The phosphoric acid applied either on unground (Figure 2A,B) or ground enamel (Figure 2C,D) resulted in similar etching patterns, which seemed to be more defined when the conditioning time was duplicated.

Different etching patterns were observed for the self-etch systems applied either on unground (Figures 3A,B and 4A,B) or

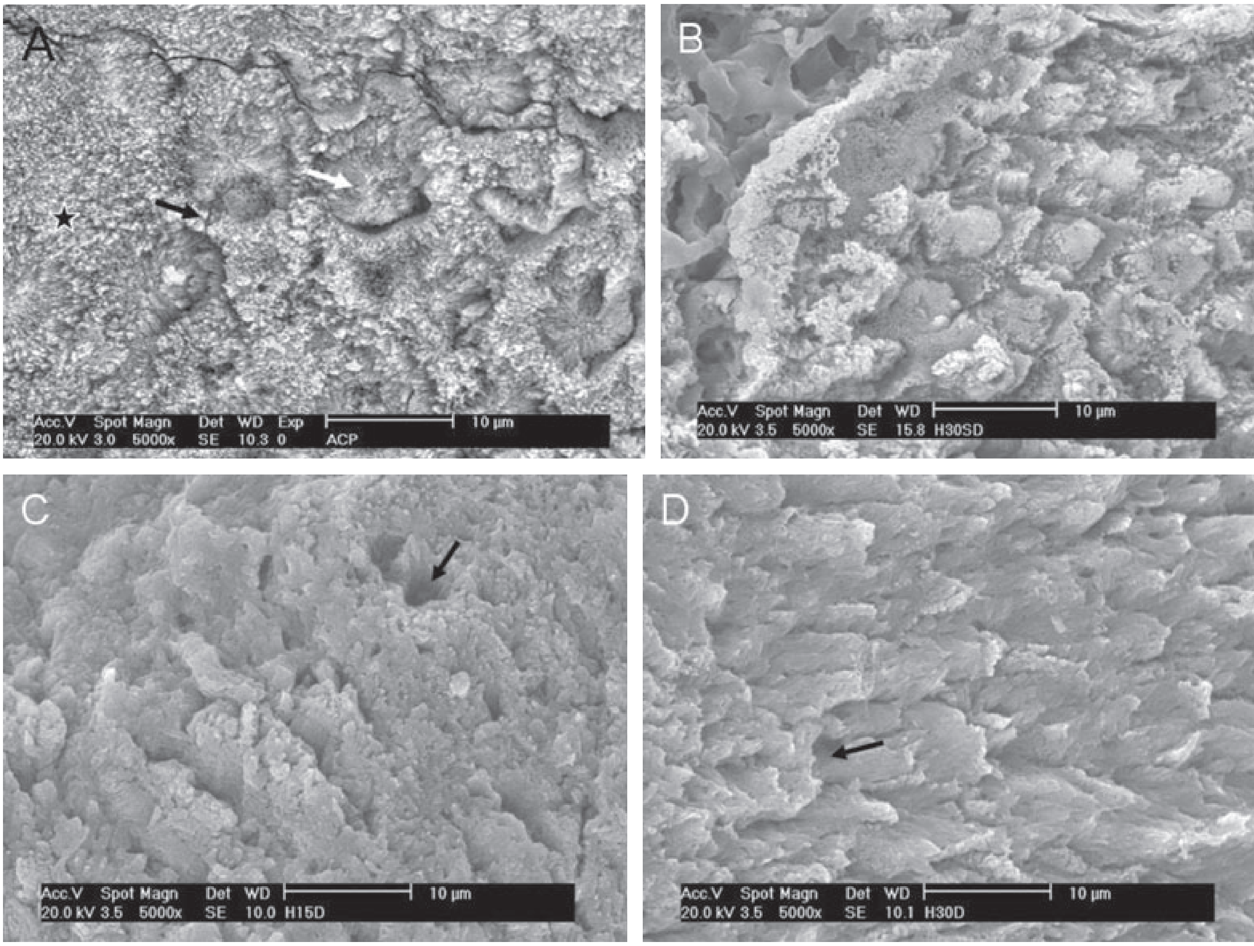

Figure 2. A,B, *Unground enamel following treatment with 35% phosphoric acid.* A, *Surface view of enamel treated with phosphoric acid for 15 seconds (bar = 10 µm): selective type III (black arrow) and type IV (black star) enamel demineralization.* B, *Surface view of enamel treated with phosphoric acid for 30 seconds (bar = 10 µm): selective enamel dissolution with a uniform pattern (mixed).* C,D, *Diamond bur-treated enamel following treatment with 35% phosphoric acid.* C, *Surface view of ground enamel treated with phosphoric acid for 15 seconds (bar = 10 µm): irregular area, with prisms reaching the surface (black arrow);* D, *Surface view of ground enamel treated with phosphoric acid for 30 seconds (bar = 10 µm): more expressive dissolution of enamel prisms, which reaches the surface (black arrow).*

ground (Figures 3C,D and 4C,D) enamel. A more defined etching pattern was observed on ground enamel for Clearfil SE Bond (Figure 3C,D) and Adper Prompt L-Pop (Figure 4C,D). For the other adhesives, no significant difference was observed between ground versus unground enamel.

Adper Prompt L-Pop showed a more defined etching pattern than Clearfil SE Bond, which was very similar to that observed after conditioning with phosphoric acid on ground enamel (Figure 4C,D).

Failure Mode

Table 3 presents the frequency distribution of failure modes according to the experimental conditions. Mixed failures were observed for

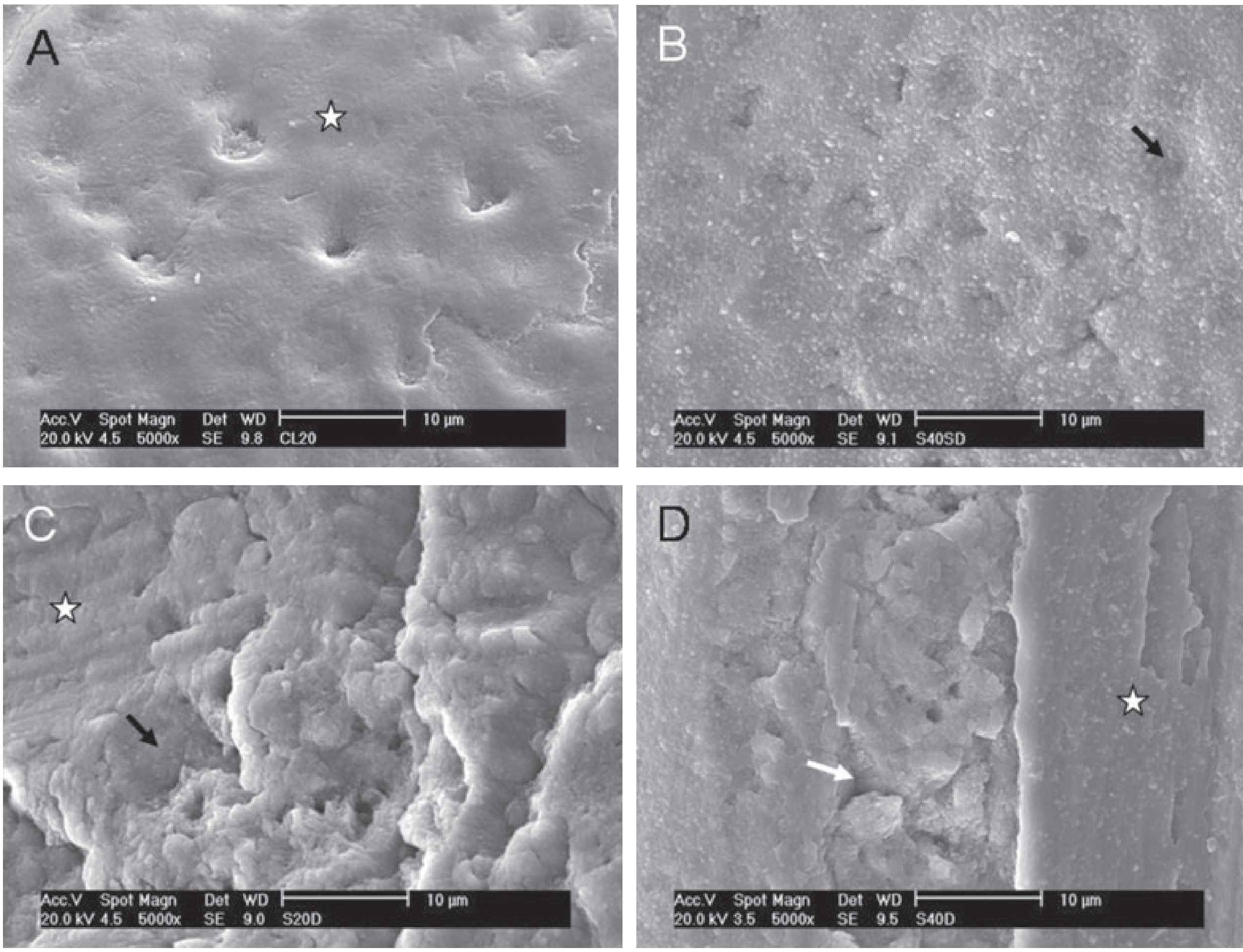

Figure 3. A,B, *Clearfil SE primer on unground enamel.* A, *Surface view after a conditioning time of 20 seconds (bar = 10 µm): note the predominately smooth surface (white star),* B, *Surface view after a conditioning time of 40 seconds (bar = 10 µm): demineralized areas where some prisms can be seen. No defined etching pattern (black arrow) but with a more irregular etching pattern.* C,D, *Clearfil SE primer on diamond bur-treated enamel.* C, *Surface view after a conditioning time of 20 seconds (bar = 10 µm): enamel prisms reach the surface (black arrow). Some grooves created by the diamond bur can be clearly seen (white star).* D, *Surface view after a conditioning time of 40 seconds (bar = 10 µm): shallow depressions (white arrow) within an area are predominately smooth (white star).*

all adhesives, but OptiBond FL showed cohesive failures in resin predominantly. Figures 5 and 6 show images of the failure modes.

DISCUSSION

The present study demonstrated that the bond strength values and the etching pattern depended on the adhesive employed, which led us to reject the null hypothesis of this study. Contrary to our previous expectations, the bond strength mean of OptiBond FL was the lowest. This adhesive has high filler loading (48% wt), and therefore the resulting bonding layer was likely thicker than that formed with nonfilled adhesive. This might have resulted in a different stress distribution under shear forces. A recent study has evaluated the stress distribution in shear and microshear test setups using finite

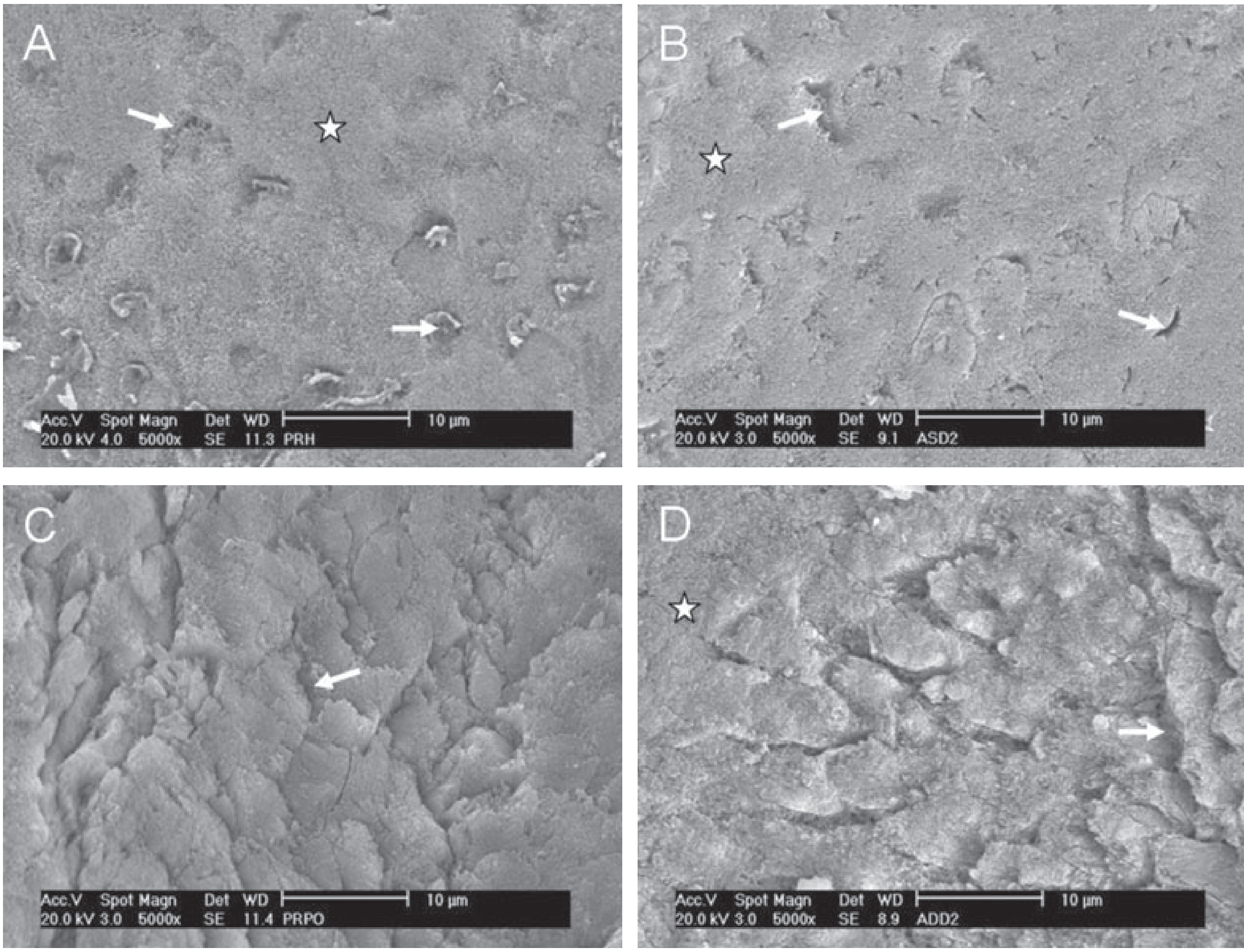

Figure 4. A,B, *Adper Prompt L-Pop on unground enamel after tooth prophylaxis.* A, *surface view after a conditioning time of 15 seconds and* B, *surface view after a conditioning time of 30 seconds (bar = 10 µm): demineralization exposing some enamel prism (white arrows), within an area with fine surface roughening (white stars).* C,D, *Adper Prompt L-Pop applied on diamond bur-treated enamel.* C, *surface view after a conditioning time of 15 seconds (bar = 10 µm): irregular surface, prisms reaching the surface (white arrow);* D, *surface view after a conditioning time of 30 seconds (bar = 10 µm): surface predominately irregular (white arrow) within areas of fine roughening (white star).*

element analysis.[33] The authors reported that stress concentration values farther exceeded the nominal strength and that a relatively thicker adhesive layer as well as the use of low-modulus composites can lead to relevant stress intensification, which could have accounted for the lower bond strength values observed by the OptiBond FL system. In the present study, the frequency distribution of cohesive failures was higher in OptiBond FL, suggesting that the lower bond strength was related to different stress distributions. When the same bonding approach (etch and rinse) was evaluated with a more fluid adhesive from the same manufacturer (OptiBond SOLO Plus), significantly higher resin–enamel bond strengths were detected and mixed failure mode was predominant.

TABLE 3. FREQUENCY DISTRIBUTION OF FAILURE MODES AND EXPERIMENTAL CONDITIONS.

Adhesive	Enamel Surface Treatment + Conditioning Time (T/A)	Mixed (%)	Cohesive resin (%)
FL	G + R (17/10)	40.0	60.0
	G + D (13/07)	28.6	71.4
	U + R (15/10)	40.0	60.0
	U + D (22/14)	57.1	42.8
SO	G + R (22/14)	64.3	35.7
	G + D (23/17)	70.6	29.4
	U + R (22/15)	73.3	26.7
	U + D (23/17)	82.3	11.7
SE	G + R (22/13)	76.9	23.0
	G + D (23/16)	75.0	25.0
	U + R (22/17)	88.2	11.8
	U + D (21/12)	91.7	8.3
AD	G + R (21/17)	88.2	11.8
	G + D (21/17)	82.4	17.6
	U + R (22/18)	88.9	11.1
	U + D (19/16)	93.8	6.2

G = ground; U = unground; R = recommended; D = double; T = total sample; A = number of analyzed samples; FL = OptiBond FL; SO = OptiBond Solo Plus; SE = Clearfil SE Bond; AD = Adper Prompt L-Pop.

Although no study has so far reported the limitations of the microshear bond strength testing,[19,21,34] the effect of fluid capillarity of the fluid adhesive can be considered a chief disadvantage of this method, particularly because the Tygon tube is usually placed on the tooth substrate before the adhesive light-curing step. In order to avoid such inconvenience, recent articles have attempted to light cure the adhesive before the placement of the Tygon tube.[35] Although this approach practically eliminates fluid capillarity, it results in an excess of adhesive layer beyond that in contact with the composite material. Whether or not this variable affects the bond strength values is yet to be addressed.

Different from what was observed in the present study, the performance of OptiBond FL (34.5 ± 2.2 MPa) was superior to OptiBond SOLO Plus (19.1 ± 2.3 MPa)[36] in a microtensile bond strength study. The kind of bond strength test used for the measurement of the interfacial strength could have been responsible for the opposite findings. A recent study has revealed that the conventional shear bond test lacks the sensitivity that is required to detect subtle differences between bonding agents or procedures,[37] which also seems to be the case for the microshear test.

Scanning electron microscopy examination of the enamel surface treated with Clearfil SE Bond revealed that this self-etch adhesive produced a very mild etching pattern, interacting only superficially with enamel. Therefore, the good performance of the mild self-etch Clearfil SE Bond in the present study and in other investigations[38,39] cannot be solely attributed to the etching pattern produced by the acidic 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer. The additional chemical interaction of the functional monomer 10-MDP with hydroxyapatite might play a very important role on the good performance of this material[15] on enamel, because this substrate consists of nearly only mineral substance, with which 10-MDP can chemically react.

An inferior performance of one-step self-etch systems in regard to bond strength measurements[1,7] has been reported. The high amount of solvents, especially water,[40,41] presented in these systems interferes with the polymerization of the resin monomers.[42] The hydrophilic and hydrophobic monomers from one-step self-etch systems usually separate into phases as the solvent

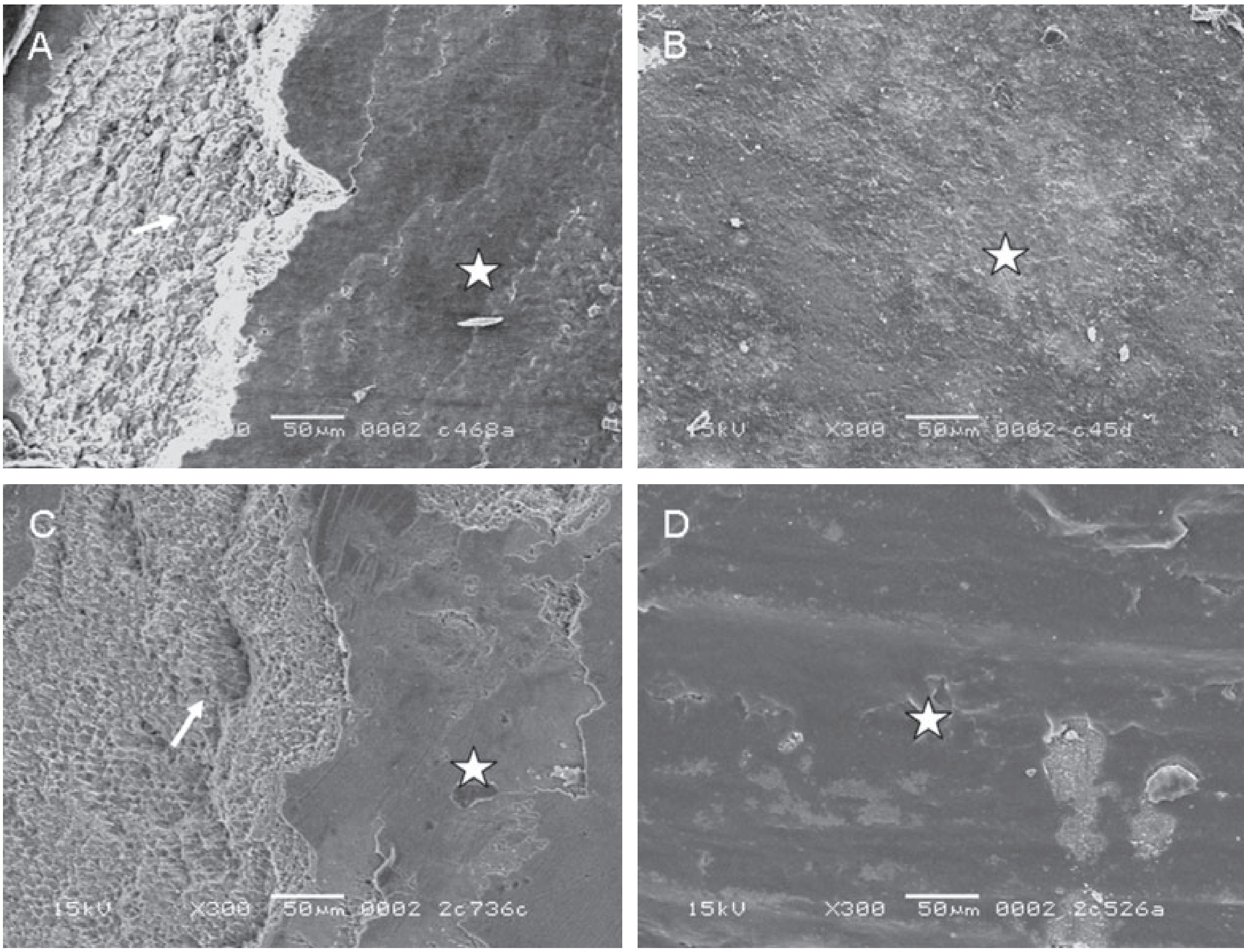

Figure 5. A *to* D, *Failure modes of OptiBond FL and OptiBond Solo Plus. Mixed failures (*A,C*) showing enamel (white arrow) and resin (white stars) in the same surface and cohesive resin failure (*B,D*) showing only resin (white stars). Bar = 50 µm.*

evaporates from the tooth substrate interfaces.[43] However, in the present investigation, the acidic one-step self-etch Adper Prompt L-Pop[1] showed bond strength values similar to the self-etch Clearfil SE Bond and the etch-and-rinse OptiBond Solo Plus. Although we cannot rule out the fact that the defined etching pattern achieved by this system could have played a role on this good performance, other factors can also be responsible. Maybe the phase separation, commonly observed with one-step self-etch systems, does not occur with such adhesive. However, this hypothesis should be further investigated.

The duplication of the enamel conditioning time did not result in an increase in the resin–enamel bond strength values, regardless of the adhesive employed. This finding is contrary to a previous study[17] which reported that an increase of 30 to 60 seconds in the application time of Clearfil Liner Bond 2 provided a better seal in class V cavities both in vitro and in vivo. Recently,[11] it has been demonstrated that doubling

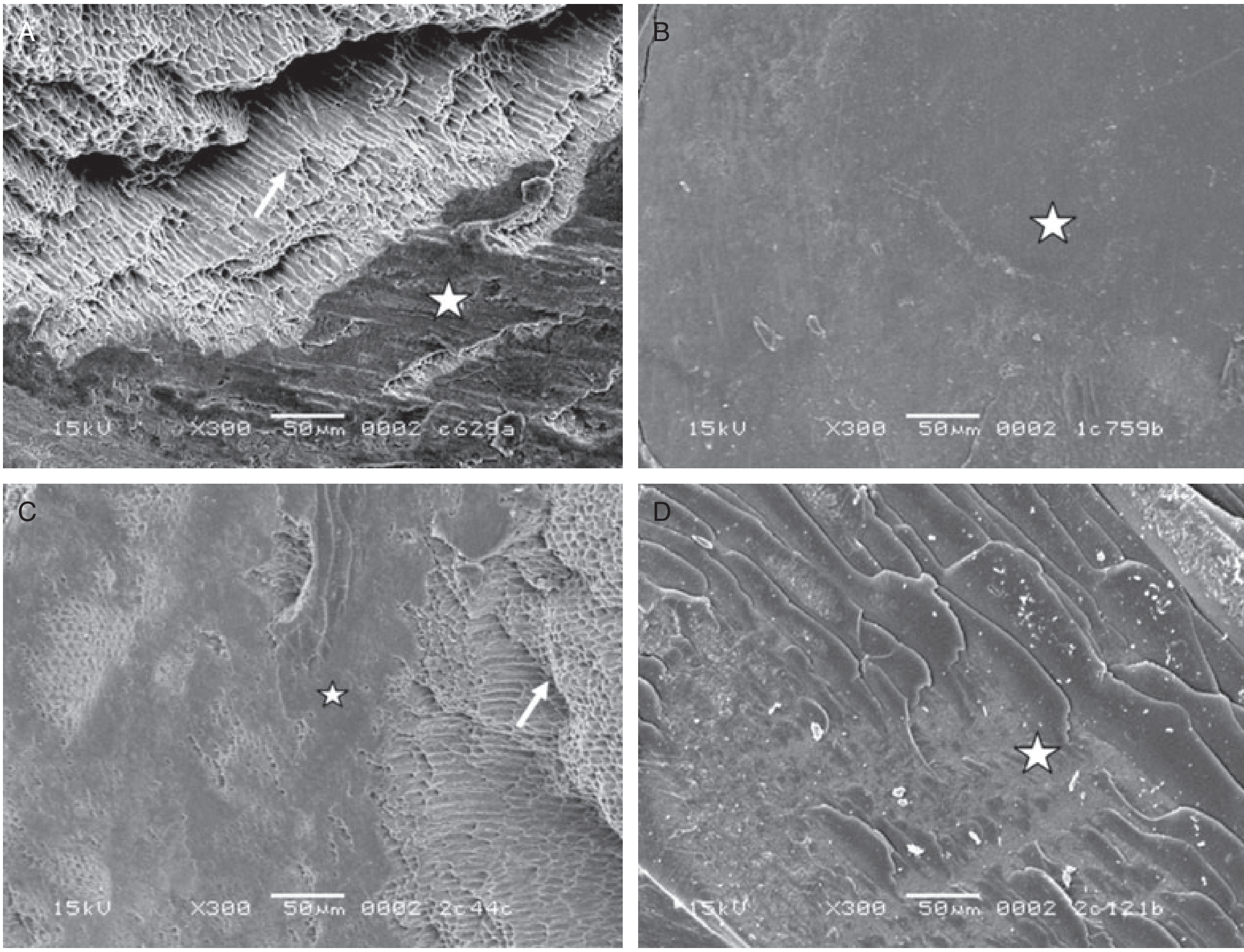

Figure 6. A *to* D*, Failure modes of Clearfil SE Bond and Adper Prompt L-Pop. Mixed failures (*A,C*) showing enamel (white arrow) and resin (white star) in the same surface and cohesive resin failure (*B,D*) showing only resin (white stars). Bar = 50 μm.*

the enamel application time of self-etch systems can increase the bond strength values for some self-etch systems. These authors[11] showed that the duplication of the conditioning time did not produce any increase in the resin–enamel bond strengths of Tyrian SPE and Adper Prompt L-Pop, while the opposite occurred for Clearfil SE Bond. These findings are not in agreement with the current investigation.

Although the extrapolation of the results from the laboratory setting to the clinical scenario is rather difficult, materials should be screened first under laboratory evaluations in order to exclude those with a performance lower than the average. Based on that, the materials with the lowest microshear bond strength values under the present study and in other in vitro studies should be avoided in clinical situations.

Although the surface enamel treatment allowed for the achievement of a more defined etching pattern for Clearfil SE Bond and Adper Prompt L-Pop, this morphological feature did not translate into higher resin–enamel bond strength values, as already reported by other studies.[7,13] However, some authors have concluded that the enamel surface preparation can increase the bond strength values of self-etch systems.[7,10,28] These controversial results could be attributed to the kind of adhesive systems employed, the method of adhesive application, the bonding test employed, etc. The etching pattern produced by Adper Prompt L-Pop on ground enamel was similar to that achieved by the phosphoric acid, which is in agreement with the findings of Hipólito et al.[32]

The variable that most influenced the etching pattern was the factor adhesive. In fact, the more acidic the system, the more defined the etching pattern in the enamel, as already confirmed by previous studies. More interesting is the lack of relationship between the etching pattern and the bond strength values. For instance, Clearfil SE Bond and Adper Prompt L-Pop provided similar bond strength values; however, the etching pattern produced by these materials was completely different. The former produced a surface apparently smooth, while the latter produced an etching pattern similar to that achieved by phosphoric acid. This lack of correlation was also observed in other studies,[15,44] which suggests that the composition of the material may be much more important than the application method and the condition of the enamel substrate before bonding. Further studies with experimental self-etch systems rather than with commercial materials should be conducted in order to identify the aspects of composition that result in good laboratorial and clinical performance.

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