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ORIGINAL ARTICLE

Interfacial and surface characterization of two self-etching adhesive systems and a total-etch adhesive after bonding to ground and unground bovine enamel—a qualitative study

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Abstract The purpose of the study was to evaluate the enamel surface and interface morphology of two self-etching adhesive systems (SAS) vs a total-etch control, after bonding to ground and unground enamel using field emission scanning electron microscopy (FESEM). Thirty bovine incisors were used in this study. The buccal enamel surface of 15 teeth was ground flat to resemble freshly cut enamel. The rest of the teeth were left intact. Two SAS, Clearfil SE Bond (CSE, Kuraray) and Prompt L-Pop (3M-ESPE), and a conventional adhesive system, Scotchbond Multipurpose (3M-ESPE, control), were used to condition the surface of unground and ground enamel on 12 teeth. A composite button was bonded to the remaining 18 teeth; a cross-section (1 mm thick) was obtained from each and the bonded interface was polished. All specimens were dehydrated in ascending grades of ethanol, gold-sputter-coated, and observed under FESEM (Hitachi S-4000) to evaluate the ultrastructural morphology of the enamel surface and the enamel–dentin interface. The etching patterns and adhesive penetration varied according to the aggressiveness of the SAS, with CSE being the mildest and H_3PO_4 being the most aggressive. There were no significant differences on the ultrastructural morphology of the enamel surface between unground and ground specimens. It appears that microporosities within enamel prisms provide sufficient enamel–resin hybridization in unground enamel. The enamel dissolution pattern and depth of infiltration depend on the type of SAS used, with no significant differences in unground and ground enamel.

Keywords Enamel · Total-etch adhesive · Self-etching adhesives · Interface · Scanning electron microscopy

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Introduction

Adhesion of dental adhesives to enamel was predictably obtained because Buonocore first introduced acid-etching in 1955 [24]. However, there is a need for improvement and specifically simplification of adhesive procedures to develop a predictable system that will be less time-consuming and less technique-sensitive [10, 16, 26].

Self-etching primers are adhesive systems that simultaneously acid-etch and prime both enamel and dentin. They penetrate, dissolve, and incorporate the smear layer into the adhesive interphase in a single step [6, 37]. The advantages of this approach include a simplified application technique by eliminating one step in the bonding process [41], cause infiltration of monomers to the same depth of demineralization [20], allows for monomer polymerization in situ [6, 18, 30], prevents the potential collapse of the collagen mesh after dentin conditioning because rinsing is not required [17], and results in decreased postoperative sensitivity [5].

However, the efficiency of these adhesives has not been consistent when used on enamel and dentin. Some of these new systems have demonstrated good results when used in dentin [13, 14, 17, 18, 21, 22, 25, 35, 37, 40], but not when applied on enamel [19]. Low bond strengths were found when unground enamel was used as the bonding substrate

[3, 15, 22, 41]. Nevertheless, similar bond strengths were obtained when testing ground vs unground enamel surfaces with conventional adhesives [1]. It is probable that chemical and micromorphological differences between ground and unground enamel may be one of the reasons why inconsistent results were obtained when bonding with self-etching primers and their diminished ability to etch the enamel surface because of their higher pH when compared to conventional etchants containing phosphoric acid [22].

In a previous study [11], microtensile bond strength values of self-etching primers to ground and unground bovine enamel showed no statistically significant differences. It is important to determine the correlation, if any, between the previously obtained bond strength values [23] and the ultrastructure of the bonded interface. Because both types of enamel are usually present in the clinical situation in the substrate area, it is necessary to attain efficient adhesion to each of these surfaces when using self-etching priming systems. Therefore, it is important to determine why these products gave similar bond strengths even though the patterns were very different.

The purpose of this study was to evaluate the ultramorphology of the enamel surface and bonded interface of two commercially available self-etching adhesive systems (SAS) to ground and unground bovine enamel using a conventional adhesive system with phosphoric acid as control. The null hypothesis was that there are no differences in the ultramorphology of SAS when compared to a total-etch control and that there are ultrastructural differences between unground and ground enamel surfaces.

Materials and methods

Evaluation of surface morphology

Twelve bovine incisors were used for this part of the study. After extraction, the teeth were cleaned of gross debris and stored in a 0.5% chloramine T solution at 4°C to prevent bacterial growth.

There were three adhesive systems applied to two enamel surface conditions (unground vs ground enamel), resulting in six groups (see Appendix). Two teeth were randomly assigned to each group for a total of 12 teeth. After the roots and the lingual half of the crown were removed, the facial enamel of the unground enamel samples was pumiced for 5 s using slurry of pumice and a white rubber cup with a slow-speed handpiece and then thoroughly rinsed. The facial enamel of the ground enamel specimens was ground flat with a 600-grit SiC paper. The enamel in both unground and ground specimens was conditioned with either one of the two SAS, Prompt L-Pop (LP, 3M-ESPE, Dental Products, St. Paul, MN, USA) or Clearfil SE Bond (CSE, Kuraray, Osaka, Japan), or with Scotchbond Multipurpose etchant (SBMP, 3M-ESPE, Dental Products) that was used as a control. The teeth conditioned with the SAS were rinsed with ethanol at the end of the etching time to remove any residual primers left on the surface. All teeth were then thoroughly rinsed with water and then fixed in 3% glutaraldehyde and 3% formaldehyde in 0.1 M Na-cacodylate buffer (pH of 7.3) for 2 h. After rinsing, the specimens were dehydrated in an ascending series of ethanol and critical-point-dried with hexamethyldisilazane (HMDS), mounted on aluminum stubs, and gold-sputter-coated to prepare them for analysis under a field emission scanning electron microscopy (FESEM, Hitachi S-4000). Specimens were examined at various magnifications and representative micrographs were obtained.

Evaluation of interfacial morphology

Eighteen additional teeth were divided into three adhesive groups and two enamel surface conditions (unground vs ground enamel) resulting in six groups (see Appendix). Six teeth were randomly assigned to each of the adhesive systems used. After removing the roots, the facial enamel of three teeth in each group was pumiced for 5 s as described above to obtain the unground enamel samples. In the rest of the teeth, the facial enamel was ground flat with a 600-grit SiC paper under running water to obtain a flat ground enamel surface. The same adhesive systems used before (products and batch numbers) were used according to the manufacturers' instructions. Composition, batch numbers, and application instructions for each adhesive system are provided in Table 1. All adhesives were light-cured for 10 s at 600 mW/cm^2 with a Demetron 401 light unit (Demetron/Kerr, Danbury, CT, USA). After bonding, a 2-mm layer of Herculite XRV composite resin (shade A-2; Kerr Dental, Orange, CA, USA) was bonded to the facial surface of each tooth and light-cured for 40 s at 600 mW/cm^2. The teeth were fixed for 2 h in 2.5% glutaraldehyde in 0.1 M Na-cacodylate buffer, rinsed and stored in water for 24 h. The crowns of the bonded teeth were attached to an acrylic block using sticky wax before sectioning them with a water-cooled slow-speed diamond saw (Buehler Isomet 1000™, Buehler, Lake Bluff, IL, USA) at right angles to the long axis of the crown. The cross-sections obtained had an approximate thickness of 1 mm (Fig. 1) and each section was sequentially polished with a 600- and 800-grit silicon carbide paper, 6 and 1 μm of diamond slurries, and slurry of 0.04 μm aluminum oxide. The specimens were dehydrated in an ascending series of ethanol and critical-point-dried with HMDS, mounted on aluminum stubs, and argon-ion-milled (Gatan Model 600 Dual Ion Mill) for

Table 1 Product information

	Manufacturer's instructions	Composition of dentin bonding systems		Manufacturer's batch numbers	
Clearfil SE Bond (Kuraray)	a) Dry enamel.	Primer	A) MDP	Lot Number	61112
	b) Apply primer with a sponge or brush tip. Avoid pooling.		B) HEMA		
	c) Leave for 20 s.		C) Hydrophilic DMA		
	d) Air-dry with mild air flow.		D) Water		
	e) Do not rinse.		E) Camphorquinone		
	f) Apply bond to entire surface with a sponge or brush tip.		F) *N*,*N*-Diethanol *p*-toluidine		
	g) Air-thin slightly.	Unfilled Resin	A) Bis-GMA	Primer	00101A
	h) Light-cure for 10 s.		B) HEMA	Adhesive	00032A
			C) MDP		
			D) Hydrophobic DMA		
			E) Camphorquinone		
			F) *N*,*N*-Diethanol p-toluidine		
			G) Microfiller		
Prompt L-Pop (ESPE)	a) Dry enamel.	Primer	A) Di-HEMA-phosphate	Lot Number	008
	b) Apply one layer to entire surface.		B) Photo initiator (phosphine oxide)	Primer	55739
	c) Rub for 15 s.		C) Water		
	d) Air-dry to disperse the material into a homogenous slightly shiny film. If surface does not appear shiny, reapply.		D) Stabilizer (butylhydroxytoluene)		
	e) Light-cure for 10 s.		E) Preservatives (methyl- and propylparabene)		
			F) Fluoride complex		
Scotchbond Multipurpose (3M)	a) Dry enamel.	Etchant	A) Phosphoric acid (35%)	Lot Number	19991004
	b) Apply etchant for 15 s; rinse for 15 s.		B) Silica		
	c) Dry thoroughly.		C) Water		
	d) Apply adhesive with a brush.	Primer	A) HEMA	Etchant	7523
	e) Light-cure for 10 s.		B) Polyalkenoic acid copolymer	Primer	7542
			C) Water		
		Adhesive	A) Bis-GMA	Adhesive	7543
			B) HEMA		
			C) Photoinitiator		

From [11]
DMA dimethylacrylamide, *MDP* membrane dipeptidase, *HEMA* hydroxyethyl methacrylate, *Bis-GMA* 2,2-bis[4-(2′-hydroxy-3′-methacryloxy-propoxy)phenyl]propane

10 min with a 2-kV DC gun voltage, 1.0-mA gun current, and 10–30 μA of specimen current to enhance the relief of the interface. Finally, they were gold-sputter-coated to prepare them for analysis under a FESEM (Hitachi S-4000), examined at various magnifications and representative micrographs were obtained.

Results

Surface morphology

The etching pattern obtained with phosphoric acid on unground bovine enamel can be seen at different magnifi-

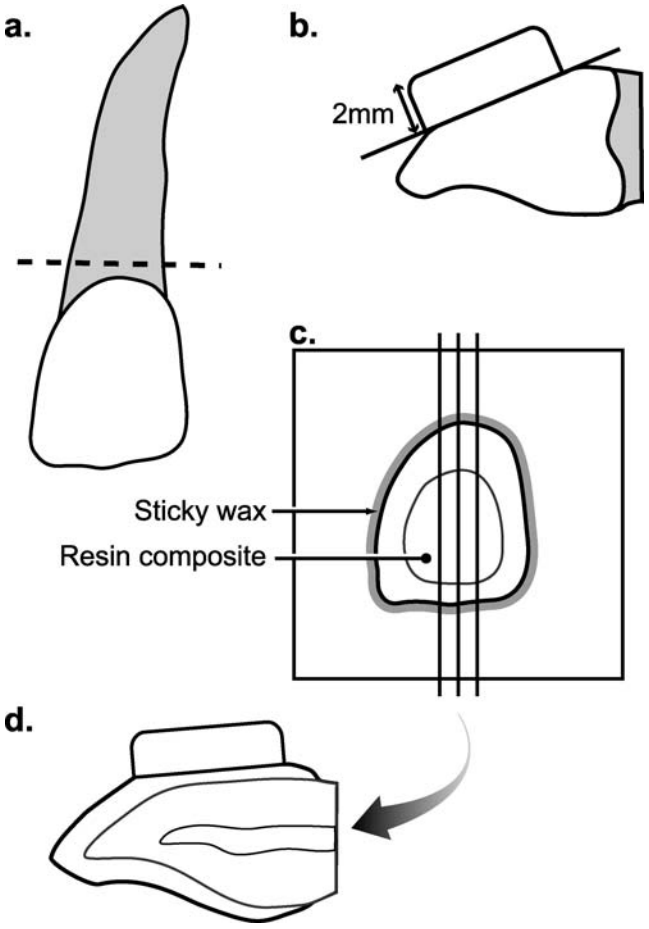

Fig. 1 Sample preparation: **a** after sectioning the root, the enamel was cleaned to prepare it for the bonding procedure; **b** after conditioning of the enamel surface, a 2-mm composite button was bonded to either ground or unground enamel; **c** crowns were attached with sticky wax to an acrylic block for sectioning; and **d** sample section ready to be prepared for SEM evaluation

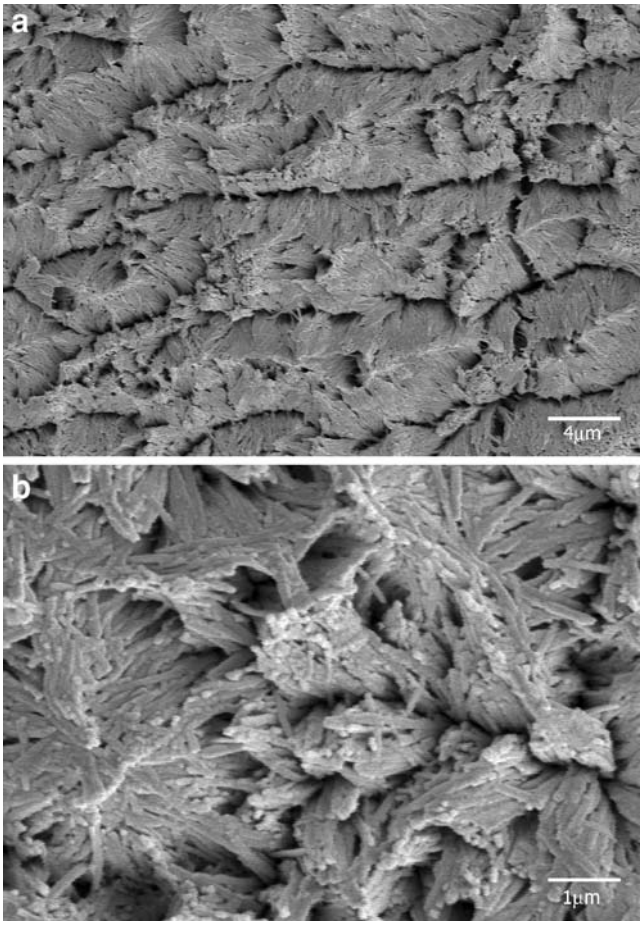

Fig. 2 **a** Unground bovine enamel surface after conditioning with H_3PO_4 for 15 s. Although the etching pattern does not resemble that observed in human enamel, mineral dissolution is clearly evident surrounding the enamel crystals (×5,000). **b** Mineral dissolution has created macro- and microporosities around the enamel crystals in unground bovine enamel after H_3PO_4 conditioning for 15 s (×20,000)

cations in Fig. 2a,b. There seemed to be a clear dissolution of the enamel prisms with the formation of microporosities. Ground enamel gave a similar appearance compared to unground acid-etched enamel (image not shown).

Figure 3a shows the unground enamel surface after conditioning with CSE self-etching primer. The surface presents fine polishing scratches and only slight dissolution of the mineral phase can be seen. When the surface is seen under higher magnification (Fig. 3b), the enamel surface shows a mild dissolution of surface crystallites to create microporosities within enamel prisms.

The mineral dissolution seen on the unground specimens treated with LP was far more aggressive than that seen with CSE (Fig. 4a,b). In lower magnification, the outline of enamel prisms can be seen (Fig. 4a). At higher magnification, the microporosities in enamel produced by LP are revealed.

Interfacial morphology

The interface obtained with SBMP on unground phosphoric acid-etched enamel can be observed in Fig. 5a,b,c. Macroretentive resin tags (white arrows) and microretentive resin tags (white asterisks) are clearly visible, as well as a 5- to 8-μm-thick layer of adhesive. Note that some enamel crystals migrated into the adhesive before it polymerized (Fig. 5b). When ground phosphoric acid-etched enamel samples were observed, the macro- and microretentive resin tags formed due to crystal dissolution of the enamel prisms were more prominent (Fig. 6a,b,c).

In the specimens etched with CSE, neither the unground (Fig. 7a,b) nor the ground enamel surfaces (Fig. 8a,b) seemed to be affected by the acidity of the self-etching primer. No prism dissolution could be seen, nor the presence of macro- or microretentive resin infiltration. The enamel resin interface appeared only as a flat, nonporous interface covered by a thick layer of silica-filled adhesive resin.

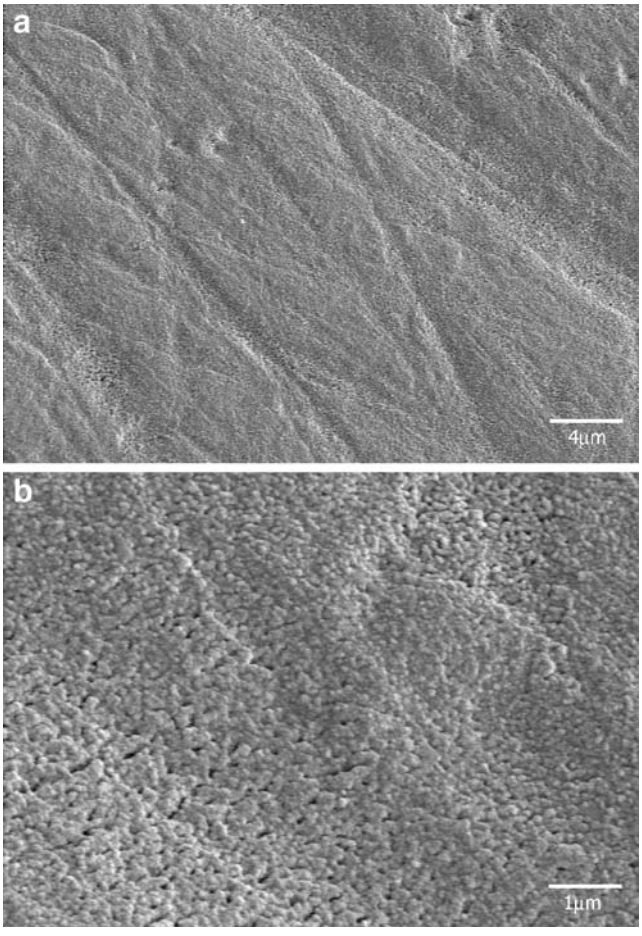

Fig. 3 **a** Unground bovine enamel surface after conditioning with CSE primer for 20 s. The etching pattern is very mild and the surface seems to be unaltered with the exception of the presence of *scratch-like marks* probably produced by pumice and a rubber cup before conditioning (×5,000). **b** At higher magnification, the seemingly unaltered enamel surface shows some mineral dissolution after conditioning with CSE primer (×20,000)

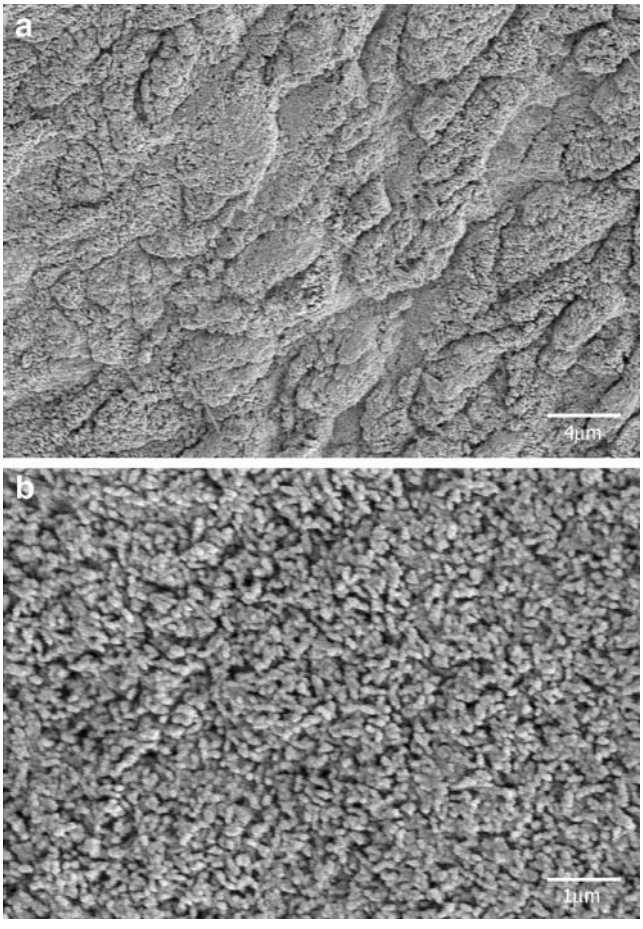

Fig. 4 **a** Unground bovine enamel surface after conditioning with LP for 15 s. The etching effect is more aggressive than the one created with CSE and prism demarcation can be readily seen (×5,000). **b** At higher magnification, the enamel surface shows mineral dissolution after conditioning with LP (×20,000)

The specimens etched with LP showed relatively mild prism dissolution when compared to the conventional phosphoric acid etching. Figure 9a,b,c shows the resin-bonded interface with unground enamel at different magnifications. Figure 10a,b,c shows the same interface on ground enamel. The ground enamel samples seemed to have slightly more mineral dissolution than the unground samples. In addition, there was no adhesive layer between the composite resin and the conditioned enamel in any of the samples treated with LP.

Discussion

Differences between unground and ground enamel were discussed previously in the literature. An aprismatic layer of enamel can be found on the surface of deciduous and permanent human teeth, and it was suggested that grinding of this superficial layer of enamel will improve bonding with acid etching [15, 22, 28, 38]. On the other hand, unground enamel has a hypermineralized surface and the resulting etching pattern after an acid conditioner is applied is frequently less homogeneous than that of ground enamel [1], although it is difficult to distinguish differences in the surface micromorphology of ground vs unground enamel after conditioning with 37% H_3PO_4 under SEM at high magnifications because both show the presence of partially demineralized hydroxyapatite crystals.

The etching pattern of unground enamel surfaces was evaluated in this study to asses if variations in the dissolution of the enamel prisms with the different adhesive systems could be observed and if it is related to the interfacial morphology and bond strength values from our previous study [11].

It was reported that the surface of unground, unpumiced, young bovine enamel may be covered by a layer containing primarily organic substances that is unaffected by acid

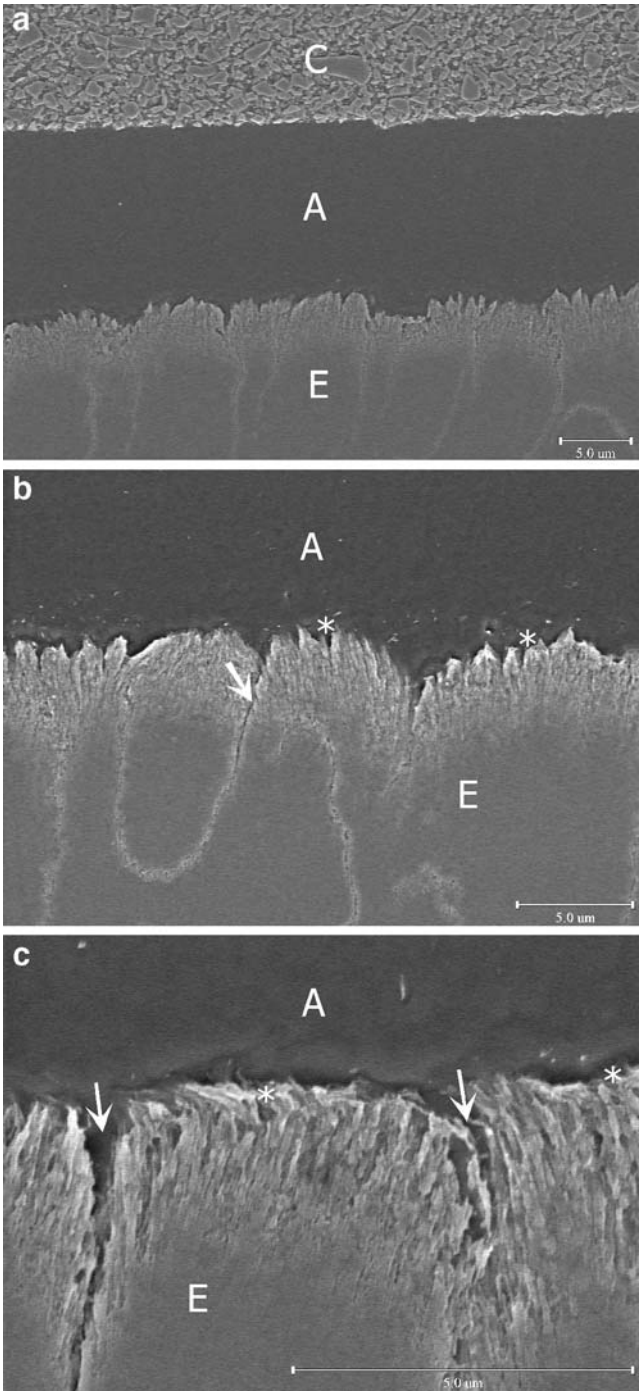

Fig. 5 **a** SEM showing a sagittal image of the unground bovine enamel interface conditioned with H_3PO_4 for 15 s. **b** Mineral dissolution around the enamel prisms boundaries and crystals can also be observed with the formation of macroretentive resin tags (*arrows*) and microretentive tags (*stars*). **c** At this magnification, little difference can be seen between the unground and ground surfaces (×3,000, 5,000, and 15,000).

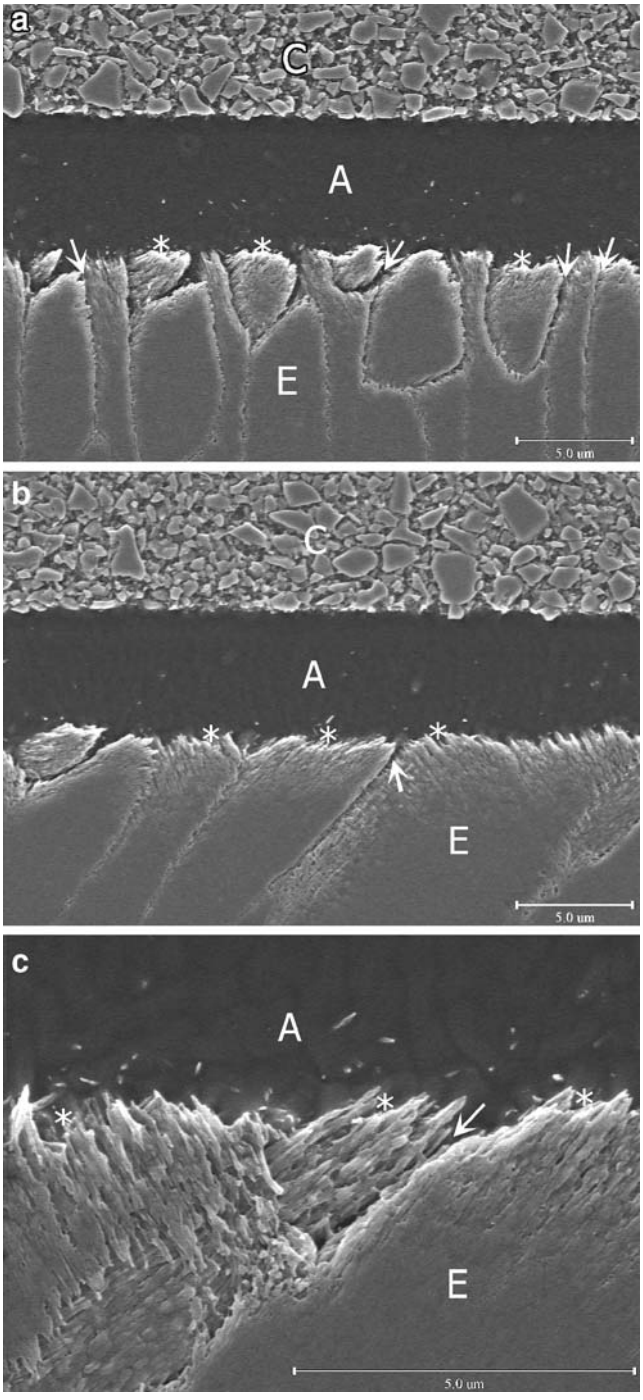

Fig. 6 **a** SEM showing an image of the polished cross-sectioned interface of ground bovine enamel conditioned with H_3PO_4 for 15 s. Mineral dissolution around the enamel prisms boundaries and crystals can be observed. **b** The clear formation of macroretentive resin tags (*arrows*) and microretentive tags (*stars*) can be seen, as well as a thick layer of adhesive resin in the samples treated with the SBMP adhesive system. **c** At higher magnification, the macroretentions (*arrows*) and microretentions (*stars*) are easily seen, as mineral dissolution affects the prisms. Argon-ion-milled surface preparation (×3,000, 5,000, and 15,000)

etching [29, 33]. This layer can be readily removed by either pumicing or grinding the superficial layer of enamel, thus making this surface susceptible to acid etching. In the present study, all teeth were pumiced before conditioning with either H_3PO_4 or the SAS. The microtensile bond strengths of human and bovine enamel was recently

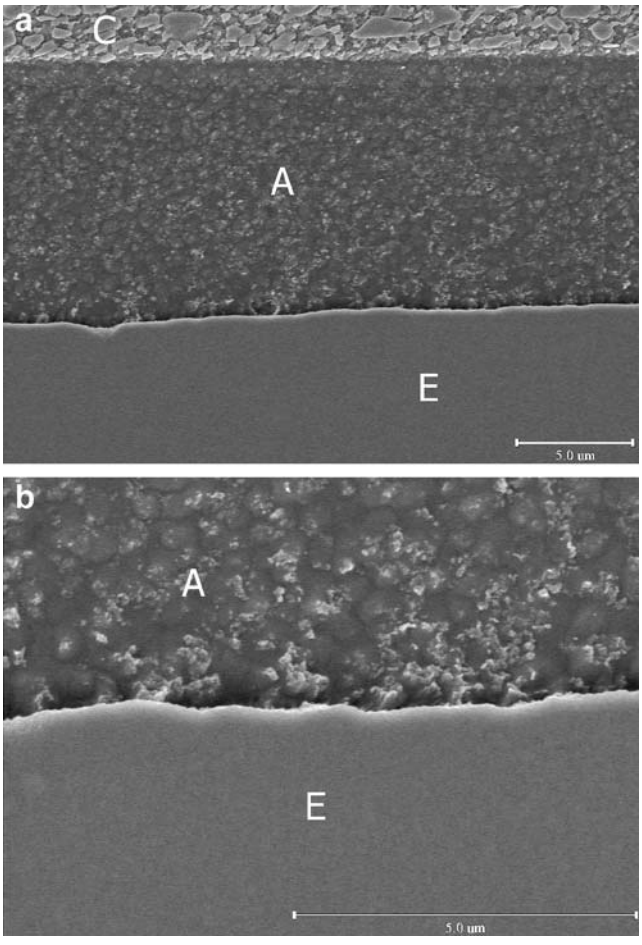

Fig. 7 **a** SEM images showing unground bovine enamel surfaces treated with CSE primer for 20 s. There seems to be no difference between surface treatment (ground vs unground) at this magnification (×5,000 and 15,000)

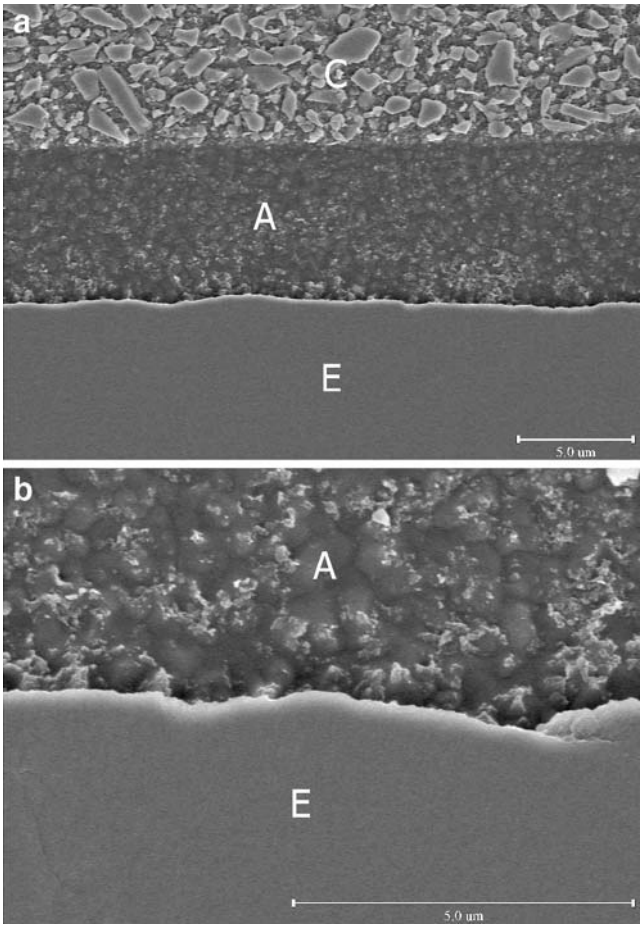

Fig. 8 **a** SEM showing ground bovine enamel surfaces treated with CSE primer for 20 s. No apparent mineral dissolution can be observed at either magnification and a thick layer of adhesive, similar to what was seen in the SBMP treated samples, can be observed (×5,000 and 15,000)

reported not to be statistically significantly different [27, 32]. Therefore, bovine enamel can be used as substitute for human enamel.

The control group was treated with a conventional adhesive system that uses 35% H_3PO_4. This strong acid clearly causes enough mineral dissolution to permit the formation of macro- and microretentive resin tags between and within enamel prisms. Polished cross-sections of resin-bonded enamel showed adequate infiltration of the adhesive resin into the surface porosities generated by this strong etchant.

Two different types of self-etching adhesives were used: a two-step system that combines conditioning and priming into one bottle but has a separate adhesive resin that is subsequently applied to complete the bonding procedure (CSE) and a one-step system that combines the conditioner, primer, and adhesive resin for a single application (LP).

CSE may be considered a mild self-etching adhesive because it has a pH of 2 and, therefore, its interaction with enamel seems to result in very mild mineral dissolution. The resulting etching pattern can barely be observed in SEM images (Fig. 3a,b). Shallow microporosities and an enamel etching pattern that was not as well-defined were previously reported after dissolution of enamel prisms with SAS compared with phosphoric acid etching, and it was suggested that this mild effect may ultimately alter adhesion [9]. In the current study, however, it is obvious that some mineral loss has occurred (Fig. 3b). It is interesting to note that a recent investigation showed that CSE bonding to enamel is stronger on phosphoric acid-etched surfaces compared to enamel that was not treated with H_3PO_4. However, bonding of CSE to dentin was significantly reduced after application of phosphoric acid. The recommendation in this paper was that enamel should be etched with phosphoric acid before the application of CSE, whereas dentin should not be pretreated with H_3PO_4 [36].

In a previous study by the authors where microtensile bond strength values of resin composite bonded to unground and ground bovine enamel surfaces were mea-

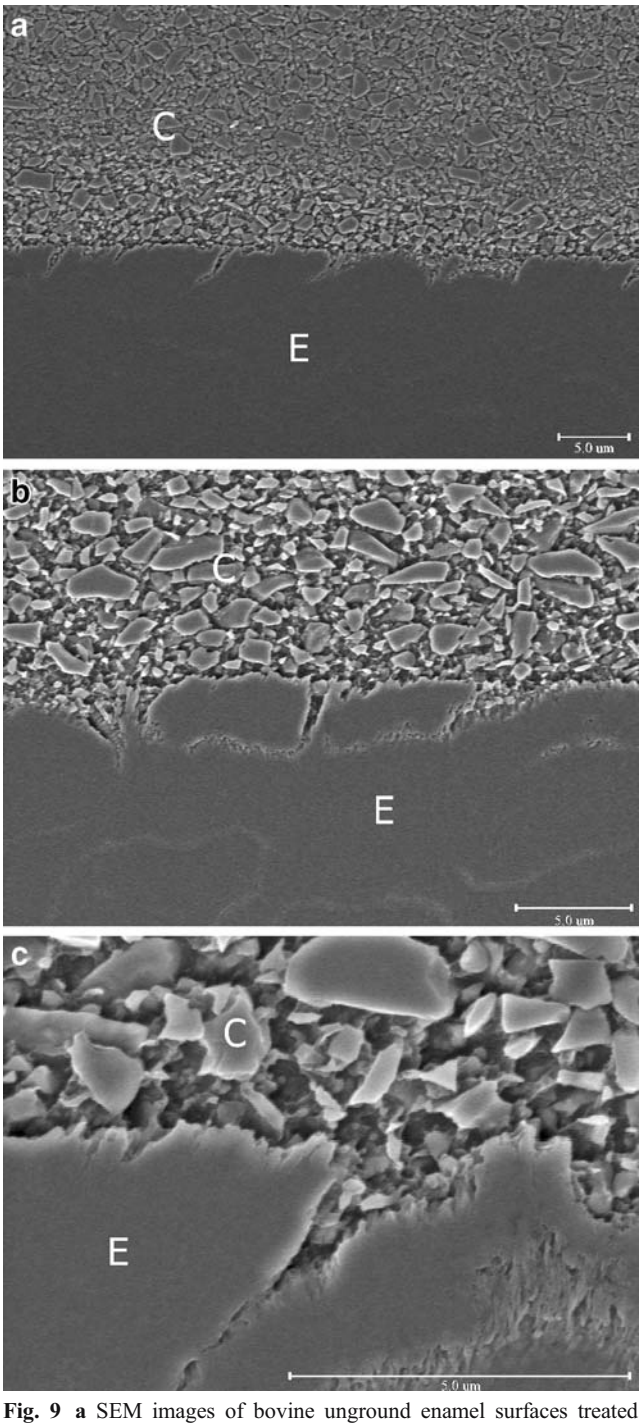

Fig. 9 **a** SEM images of bovine unground enamel surfaces treated with LP show almost no difference in the formation of macro- and microresin tags (×3,000). **b** Shallow depth of demineralization is evident and there is no adhesive layer between the enamel and the overlying composite (×5,000). High magnification reveals microporosity within the enamel prism surface (×15,000)

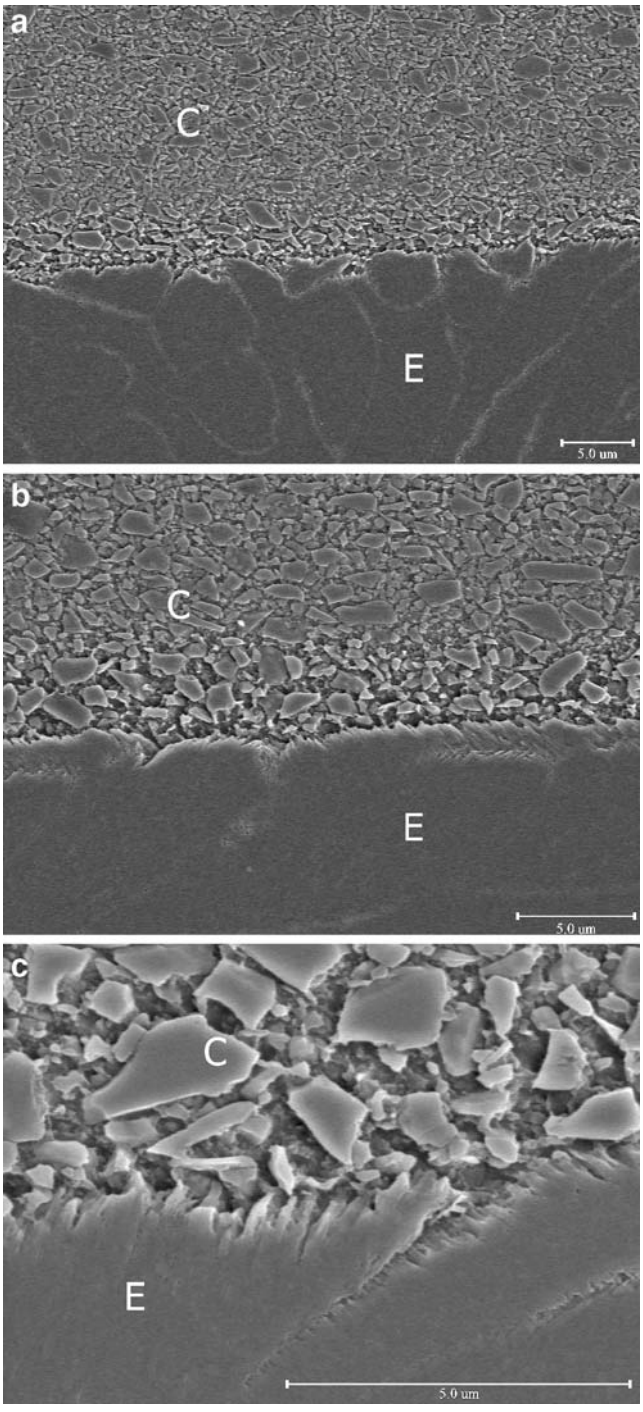

Fig. 10 **a** SEM images showing bovine ground enamel surfaces conditioned with LP for 15 s. Mineral dissolution among the prisms and between the crystals is clearly evident, although the depth of demineralization is 1/3 to 1/4 of that seen with the conventional adhesive system (×3,000). **b** The formation of macroretentive resin tags (*arrows*) and microretentive tags (*stars*) can be seen, although less dramatic than the ones seen with SBMP. There was no layer of adhesive resin between the conditioned enamel and the composite resin in the samples treated with LP (×15,000)

sured, the mean bond strength values were not statistically significantly different (Table 2) [11]. The adhesive interface for CSE does not show the formation of macrotags between enamel prisms. Therefore, there does not seem to be any apparent resin infiltration with the concomitant formation of a hybridized layer of enamel. However, recent transmis-

Table 2 Mean tensile bond strength (MPa) for resin–enamel bonds

Adhesives	Mean	SD	Min	Max	Number of samples
Prompt L-Pop (ug)	43.0a	7.9	35.4	60.5	11
Prompt L-Pop (g)	41.1a	12.1	21.6	58.2	10
Clearfil SE (ug)	41.7a	11.3	20.4	56.4	12
Clearfil SE (g)	38.6a	8.8	27.9	53	11
SBMP (ug)	37.6a	9.6	16.2	48	12
SBMP (g)	44.5a	6	34.7	55.9	11

From [11]
Groups identified with the same letter are not significantly different ($p>0.05$).
ug Unground enamel, *g* ground enamel

sion electron microscopy studies of such interfaces confirm the formation of a very thin (0.33 μm thick) layer of hybridized enamel [34] in unground enamel and 1.88 μm in ground enamel [8]. This may explain why the mean bond strength values for this adhesive are comparable to those obtained with the conventional system (Table 2). Apparently, resin–enamel bond strength relies both in interprismatic tag formation and hybridization of interprismatic enamel [8, 34] in a manner analogous to resin–dentin bonding being due to both resin tag formation and hybridization of intertubular dentin.

Shinchi et al. [31] evaluated the effect of phosphoric acid concentration on resin tag length and bond strength of composite to enamel. Their results showed that there was little correlation between the length of the tags and bond strength values, and they proposed that phosphoric acid concentrations of less than 10% might be satisfactorily used. Our results confirm the lack of correlation between resin tag length and bond strength as exemplified by the minimal etching effects of CSE (Fig. 3a,b) compared to their high bond strength from our previous study (Table 2). Similar high bond strengths with minimal enamel etching were reported in human teeth [14, 31]. Apparently, little mechanical retention is needed for good adhesion. Yoshida et al. [39] suggested that mild self-etching adhesives only demineralize dentin partially, which results in remaining crystals of hydroxyapatite surrounding the collagen. These crystals may serve as a receptor for chemical interaction with the functional monomer of the adhesive and contribute to the adhesive performance [39].

On the other hand, all of the CSE specimens show a thick layer of filled adhesive between enamel and composite. This may be an advantage of this system because it was previously reported that thicker adhesive layers withstand better the contraction stresses generated by resin composite polymerization [4].

LP may be considered an aggressive self-etching adhesive (pH of 1). The amount of mineral dissolution that was seen on the samples treated with this self-etching adhesive resulted in a mineral loss that more closely resembled that of phosphoric acid, although the enamel etching pattern was not as clearly defined. This self-etching adhesive is a one-step system that does not have a separate adhesive resin to complete the bonding process because the monomers are already incorporated into the mixture. It is of interest to note that in the interface images examined in this study, there was no adhesive layer between the composite resin and the conditioned enamel, and therefore, it is difficult to determine if there was an enamel hybrid layer present at the magnifications used. This adhesive system has a very low viscosity and tends to form very thin (i.e., less than 10 μm) films that become saturated with oxygen before polymerization. We speculate that the oxygen inhibition prevented adequate polymerization so that when the resin composite was applied to the adhesive layer, the

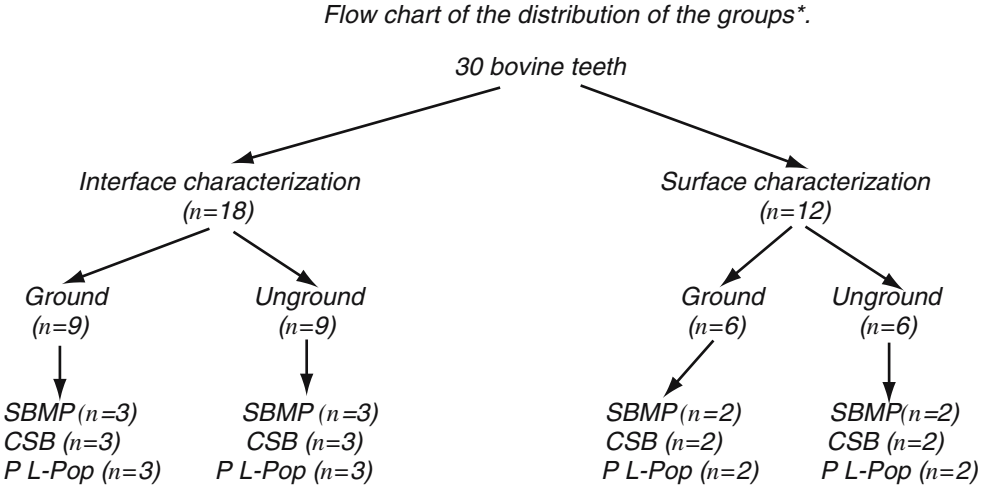

Fig. 11 Flow chart of the distribution of the groups

adhesive was displaced laterally. Similar observations were reported by Pashley et al. [23] on dentin. They obtained better results after two applications of adhesive. The manufacturer now recommends the use of two applications. The morphologic results of the current study require rejection of the null hypothesis that there are no differences in the ultramorphology of self-etching systems when compared to a total-etch control and that there are ultrastructural differences between unground and ground enamel surfaces.

The results of previous in vitro studies were variable in determining whether self-etching systems adequately condition enamel [7, 9, 15, 24, 41]. In the present study, the enamel surface in the different groups was treated in a similar manner before acidic conditioning and differences in the amount of mineral loss were obvious among the groups, which indicate that the amount of mineral loss is directly related to the acidity of the adhesive system. When comparing the apparent loss of mineral on the enamel surface with the bond strength values obtained in our previous study [11], there seems to be no adverse effect in bond strength when resin composite is bonded to enamel surfaces that present even mild etching patterns. In this study, the methodology to treat the teeth was the same as in the previous study [11]; the same operator handled the samples and treated them with the same batch of adhesive systems following the exact same protocol. Therefore, the author's feel that bond strength values obtained from our previous study seem to suggest that there is no relationship between the amount of mineral loss or hybrid layer thickness and bond strength values to ground or unground enamel (Table 2).

These findings do not agree with Kanemura et al. [15] who suggested that the shallow etching pattern obtained with the self-etching primers might not be deep enough to obtain good resin penetration to unground enamel. According to Cehreli and Altay [2], the etching patterns of aprismatic enamel are dependant upon the aggressiveness of the acids used, pH, protein kinase, and/or the etching time. Therefore, it should be expected that the use of a stronger acid, such as phosphoric acid, and a longer application time of the conditioner will result in a more dramatic dissolution and removal of the enamel mineral phase [12, 13]. Nevertheless, the depth of resin penetration might not be as important as the quality of these shallow microporosities and the degree of polymerization of the resin for obtaining a good bond to enamel. This agrees with Perdigao et al. [24] where they concluded that other factors, such as variations in surface energy, could be responsible for achieving adequate bonds to enamel.

Conclusions

Evaluation of the etching pattern and interface morphology of a conventional adhesive system (SBMP) and two SAS (CSE and LP) clearly demonstrated that there was a difference in the amount of mineral dissolution on the enamel surface that probably resulted from the different acidity and length of application time of the different adhesive systems. However, large differences between unground and ground bovine enamel surfaces were not seen either in their surface etching effect or in the ultrastructural morphology of the adhesive interface.

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Appendix

Figure 11

References

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