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Effect of thermocycling to adhesive interface of enamel analyzed by polarized light microscopy

Análise do efeito da termociclagem na interface adesivo do esmalte analisada por microscopia de luz polarizada

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ABSTRACT

Objective: The less aggressive demineralization of enamel by self-etching systems results in greater staining, marginal leakage, and failure in their restorations, so this study aimed to assess the silver nitrate infiltration and tag formation of the enamel/adhesive interface. **Material and Methods:** Two hundred enamel fragments were randomly assigned into 10 groups according to the adhesive system (Single Bond Adper Plus-(SB), Clearfil Tri-S Bond-(CF), or Scotchbond Universal-(SBU)) and enamel surface (ground-(ge), unground-(ue), phosphoric acid etching-(pha), or none) (n=10): (SB-ue), (SB-ge), (CF-ue), (CF-ge), (CF-ue/pha), (CF-ge/pha), (SBU-ue), (SBU-ge), (SBU-ue/pha), and (SBU-ge/pha). Half of the restored samples were submitted to thermocycling. Four slices of 1.0mm/sample were obtained to evaluate tag formation and silver nitrate infiltration. All of the specimens were examined with Polarized Light Microscopy, and the percentage of infiltration was quantified. **Results:** No interactions were found among the three factors. The adhesive and aging exhibited an interaction. Significant differences were found only after thermocycling: the SB and SBU-etched groups had decreased infiltration compared with the other groups. The tag length after etching was higher for ge compared with ue, regardless of the adhesive system. **Conclusion:** The self-etching techniques resulted in significantly less tag formation compared with the conventional technique.

RESUMO

Objetivo: A desmineralização menos agressiva do esmalte por sistemas adesivos autocondicionantes resulta em maior descoloração e infiltração marginal e, falhas em suas restaurações. Deste modo, este estudo teve como objetivo avaliar a microinfiltração por meio da técnica da infiltração de nitrato de prata e, formação de tags na interface esmalte/adesivo. **Material e Método:** Duzentos fragmentos de esmalte foram divididos aleatoriamente em 10 grupos (n = 10) de acordo com o sistema adesivo (Single Bond Adper Plus- (SB), Clearfil Tri-S Bond- (CF), ou Scotchbond Universal- (SBU)) e a superfície do esmalte (lixada (GE), não lixada- (UE), condicionada com ácido fosfórico 37%- (PHA), ou sem condicionamento): (SB-UE), (SB-GE), (CF-UE), (CF-GE), (CF-ue/pha), (CF-ge/pha), (SBU-ue), (SBU-ge), (SBU-ue/pha), e (SBU-ge/pha). Metade das amostras restauradas foram submetidas a 20,000 ciclos térmicos. Quatro fatias de 1,0 mm/amostra foram obtidas para avaliar a formação de tags e infiltração de nitrato de prata. Todas as amostras foram examinadas com microscopia de luz polarizada, e a percentagem de infiltração foi quantificada. **Resultados:** Não foram observadas interações entre os três fatores. O sistema adesivo e envelhecimento exibiram uma interação. Foram encontradas diferenças significativas somente após termociclagem: os grupos SB e SBU condicionados apresentaram menor porcentagem de infiltração comparados aos outros grupos, independente do tipo de esmalte. Quanto à analise qualitativa, o comprimento dos tags após condicionamento ácido foi maior para o GE comparado ao UE, independente do sistema adesivo. **Conclusão:** A aplicação dos sistemas adesivos na técnica autocondicionante mostrou uma formação de tags significativamente menor comparada à técnica convencional.

KEYWORDS

Adhesives; Aging; Dental enamel; Microscopy, polarization; Silver nitrate.

PALAVRAS-CHAVE

Adesivos; Envelhecimento; Esmalte Dentário; Microscopia de Polarização; Nitrato de Prata.

INTRODUCTION

A constant challenge in adhesive dentistry is obtaining a lasting restorative interface with enamel and dentin and the materials that can be simply implemented in the clinic. Of all of the types of simplified adhesive systems (ASs), self-etching ASs eliminate the etching step by containing in its formulation an acid monomer that allows the simultaneous demineralization and infiltration of monomers on the tooth surface [1-3] for effective penetration of the adhesive and a reduction in the formation of the gaps that are common with the conventional technique. [2,4,5]

The first self-etching all-in-one systems that emerged commercially were not very successful. In addition to the instability of their components, these ASs contained a large concentration of water, which affected the complete polymerization of the adhesive. [1,2,6] With the recent development of an improved generation of adhesives, a new one-bottle self-etching AS that consists of 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which is a bifunctional monomer, and the copolymer Vitrebond exhibits more stable and durable chemical adhesion with both enamel and dentin. [7] This new adhesive, which has been classified as multi-mode by researchers, is available commercially and has been used by professionals in addition to the three older adhesive techniques, including conventional, self-etching, and selective enamel etching.

Because of the less aggressive demineralization of enamel by self-etching systems, [1,3] their restorations exhibit greater staining, marginal leakage, and failure because the enamel is highly mineralized. [1] As a result of these complications, the selective etching of enamel with phosphoric acid (pha) has been suggested in order to obtain a microretentive standard that is similar to the surface that is created by the conventional technique [3] because the etching increases the wettability and the surface free energy of solids, thus favoring the interaction of the adhesive with the substrate. [8] Clinically, most restorations are performed on surfaces that are worn by cavity preparation, which improves the adhesion. Several studies have shown that ground enamel surfaces have more effective interactions because the wear exposes the prisms that facilitate the infiltration of the monomer. [3,5,8]

Few studies have been published on the quality of the bonded interface of multi-mode adhesives with enamel or the tag formation and infiltration in samples aged by thermocycling. Silver nitrate has been used to detect infiltration around composite restorations. [4] The low molecular weight of silver is similar to that of water, and this allows the diffusion of the tracer within the adhesive layer. [4] Therefore, it has been used to detect flaws within the bond interface. [4]

The quality of micromechanical imbrication that is formed by the infiltration of monomers into the enamel through tag formation is important and directly affects the longevity of a restoration. Therefore, the aim of this study was to compare the new multi-mode AS, a self-etching AS, and the conventional technique with respect to silver nitrate infiltration and tag formation on the adhesive interface with unground (ue) and ground (ge) enamel and the effects of etching with pha and thermocycling by polarized light microscopy (PLM). The null hypotheses that were tested were the following: (1) no significant difference occur in infiltration on enamel among the ASs with or without aging and (2) the condition of the enamel does not affect the performance of the ASs.

MATERIALS AND METHODS

For this study, we used 80 freshly extracted human molars that were cleaned and maintained in 0.1% thymol until the beginning of the experiment. The use of the teeth was approved by the Research Ethics Committee of Araçatuba Dental School/UNESP (No. 2011-03381). Before the study, the teeth were polished with pumice and water with a Robson brush (KG Sorensen, Cotia, São Paulo, Brazil). The cervical portion of each tooth was removed with a diamond disc that was mounted on a precision cutter (Isomet 2000, Buehler, Lake Bluff, Ilinois, The United States of America). The tooth crown was sectioned again in the vestibular/lingual direction in order to obtain two halves with an enamel surface. One half was kept intact, and the other half was sanded with silicon carbide (SiC) 600-grit sandpaper for 60 s during water cooling in order to simulate the formation of the smear layer and obtain a flat surface for adhesion. From the beginning of the experiment, the specimens were kept in distilled water and stored at 37 °C.

The fragments were randomly assigned to 10 groups according to the adhesive used and the surface of the substrate to be treated (ug or ge enamel), as listed in Table I.

Table I - Groups division according to the adhesive used and the surface of the substrate to be treated

Groups	Adhesive System / Enamel Surface
SB/eu	Single Bond Adper Plus / unground enamel
SB/ge	Single Bond Adper Plus / ground enamel
CF/eu	Clearfil Tri-S Bond / unground enamel
CF/ge	Clearfil Tri-S Bond (Kuraray) / ground enamel
CF/pha/ue	Phosphoric Acid 37% + ClearfilTri-S Bond / unground enamel
CF/pha/ge	Phosphoric Acid 37% + ClearfilTri-S Bond / ground enamel
SBU/eu	Scotchbond Universal / unground enamel
SBU/ge	Scotchbond Universal / ground enamel
SBU/pha/ue	Phosphoric Acid 37% + Scotchbond Universal / unground enamel
SBU/pha/ge	Phosphoric Acid 37% + Scotchbond Universal / ground enamel

ue: unground enamel; ge: ground enamel; pha: phosphoric acid; SB: Single Bond Adper Plus (3M ESPE, St. Paul, MN, USA); CF: Clearfil Tri-S Bond (Kuraray America Inc, New York, NY, USA); SBU: Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA).

The restorative procedure was performed according to the manufacturers' recommendations, except for the Clearfil Tri-S Bond (CF)/pha/ue, CF/pha/ge, Scotchbond Universal (SBU)/pha/ue, and SBU/pha/ge groups, which were subjected to etching with 37% pha for 30 s. Table II lists the composition, manufacturer, and application procedure of all of the products used in this study. Filtek Supreme XT (3M ESPE, St. Paul, MN, USA), which is a composite resin, was applied in 2-mm increments over the entire surface of the enamel, and each layer was light-cured for 20 s with a LED curing device (800 mW/cm2, EMITTER B, Schuster, Santa Maria, Rio Grande do Sul, Brazil). The application and curing times of the resin composite were standardized for all of the groups.

Half of the sample in each group was subjected to 20,000 cycles of thermal baths of 5 °C to 55 °C for 60 s. When they were not being cycled, the teeth were stored in distilled water at 37 °C. Twenty-four h after the restorative procedure, the fragments that were not subjected to thermocycling were cut into slices that were approximately 1 mm thick under water cooling.

Analysis of nanoleakage with silver nitrate

In accordance with the method described by Yuan et al. [9] two slices of each fragment were covered with two layers of nail polish (Colorama Ltd., São Paulo, Brazil), and 1 mm at the enamel/restoration interface was left unpolished. After the nail polish dried, the specimens were placed in distilled water for 10 min to hydrate. The specimens were then immersed in ammoniacal silver nitrate for 18 h and isolated from light with aluminum foil. Next, the specimens were placed in an ultrasonic tank (Ultrasonic Cleaner, model USC1400, Unique, São Paulo, Brazil) for 5 min to remove the waste. The specimens were then placed in radiographic developing solution (Dental

Table II - Composition, manufacturers, and application procedure of the products used in the study

Adhesive Systems/Code	Composition	Application Procedure	Recommended Techniques
SBU:Scotchbond Universal Adhesive 3M-ESPE; St Paul, MN, USA (Batch #504834)	10-MDP, HEMA, Vitrebond™ Copolymer, filler, ethanol, water, initiators, silane (pH Ö 2.7)	- Acid etching for 30 s - Apply adhesive (rubbing) for 20 s - Gently air dry for 5 s - Light cure for 10 s	1-step self-etch Selective etch enamel Total-etch
CF: Clearfil Tri-S Bond Kuraray Noritake Dental Inc.; Tokyo, Japan (Bond Batch #00167A)	Adhesive: 10 MDP, bis-GMA, HEMA, colloidal silica, dl-camphorquinone, etanol, water; accelerators; initiators.	- Acid etching for 30s - Apply bond (rubbing) for 20 s - Gently air dry for 5 s - Light cure for 10 s	1-step self-etch Selective etch enamel
SB: Adper Single Bond Plus 3M-ESPE; St Paul, MN, USA (Bond Batch #N2I1104BR)	ethyl alcohol, bisgma, silane treated silica (nano-filler), HEMA, copolymer of acrylic and itaconic acids, glycerol 1,3-dimethacrylate, water, UDMA, diphenyliodonium hexafluorophosphate, edmab	- Acid etching for 30 s - Rinse with water for 20 s - Apply primer for 20 s - Gently air dry for 5 s - Apply adhesive for 20 s - Gently air dry for 5 s - Light cure for 10 s	1-step total-etch adhesive
Filtek Supreme Ultra Universal Restorative A2Body 3M-ESPE; St Paul, MN, USA (Batch#N205761)	silane treated ceramic, silane treated silica, UDMA, bisphenol a polyethylene glycol diether dimethacrylate, bisGMA, silane treated zirconia, polyethylene glycol dimethacrylate, tegdma, 2,6-di-tert-butyl-p-cresol	- Application of increments (thickness of 2.0 mm) - Light cure for 20 s (each increments)	

Developer, Eastman Kodak Co., Rochester, New York, The United States of America) and exposed to fluorescent light for 6 h to reduce the silver ions along the interface. Both sides of the slices were then sanded by hand with 320-, 400-, 600-, 800-, and 1,200-grit SiC sandpaper to a thickness of 150 μm. The specimens were fixed onto slides and coverslipped. The silver nitrate infiltration into the gaps at the interface were examined with PLM at 40X before and after aging with thermocycling.

Three areas of the interface were evaluated in each specimen. The percentages of the area (μm2) infiltrated (% of nanoleakage) were calculated in the regions between the base of the hybrid layer, the hybrid layer, and the adhesive layer in each AS. ImageJ (ImageJ 1.42q, Wayne Rasband, National Institutes of Health, The United States of America) was used to assess the amount of silver nitrate present in the area of interest.[9] Therefore, the image was modified from RGB mode to 8-bit mode, which converted the white color of the silver nitrate to red (Figure 1). The particle analysis tool was used to calculate the area in μm2 that was colored in red. Thus, the infiltration value (%) of each slice was the arithmetic average of the measures obtained on 3 images. The percentage of area infiltrated by silver nitrate was calculated with this program. [9]

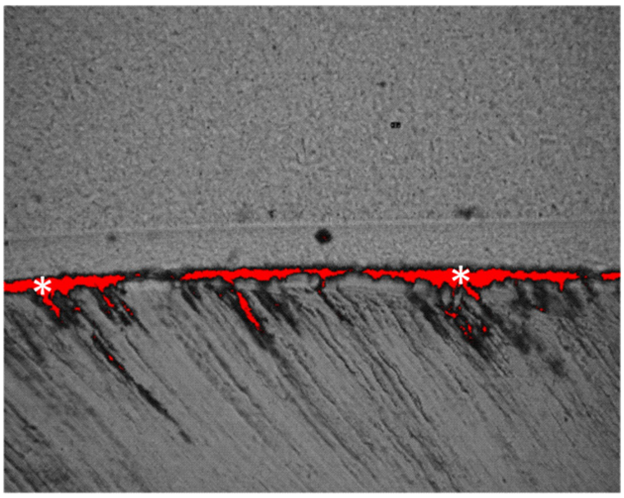

Figure 1 - Image analysis for the area infiltrated by silver nitrate. *The region of the interface is selected and the program interprets the points in red as infiltration area.

PLM analysis of the tag quality

The slices were sanded by hand with 320-, 400-, 600-, 800-, and 1,200-grit SiC sandpaper (T469-SF-Norton, Saint-Gobain Abrasives, Jundiaí, São Paulo, Brazil) under water cooling to a thickness of 150 μm. Subsequently, 40% nitric acid was applied to each specimen for 1 min for complete demineralization of the enamel, and the only portion that remained was the AS and composite resin. The specimens were carefully washed with water to remove the nitric acid, and excess moisture was removed with paper towels. After washing, the sections were fixed on slides and coverslipped and then examined with PLM.

RESULTS

Analysis of silver nitrate infiltration

The data were analyzed with a three-way analysis of variance, which showed no interactions ($p > 0.0001$) among the 3 factors tested (enamel, adhesive, and aging). However, an interaction was found between the adhesive and aging, which indicated that the adhesive technique was influenced by aging. The type of enamel surface did not affect the adhesive or aging factors (Figures 2A/B, 3A/C, and 4A/C).

The groups in which the self-etching (CF) and multi-mode (SBU) ASs were applied without prior acid etching showed a higher percentage of infiltration compared with the groups in which etching was performed (Figures 3A/E, 3C/G, 4A/E, and 4C/G). Thus, the previous use of pha in the application of the self-etching adhesive (SBU and CF) resulted in a lower percentage of infiltration area compared to that obtained with the conventional technique. Table III shows the average infiltration values in each group.

When the aged specimens were analyzed separately, the 24-h sample showed no statistical differences in any of the tested groups (Figures 2, 3, and 4). However, after aging, greater infiltration was detected in the CF, SBU, and CF/pha groups. The SBU/pha group had similar results as the SB group after aging.

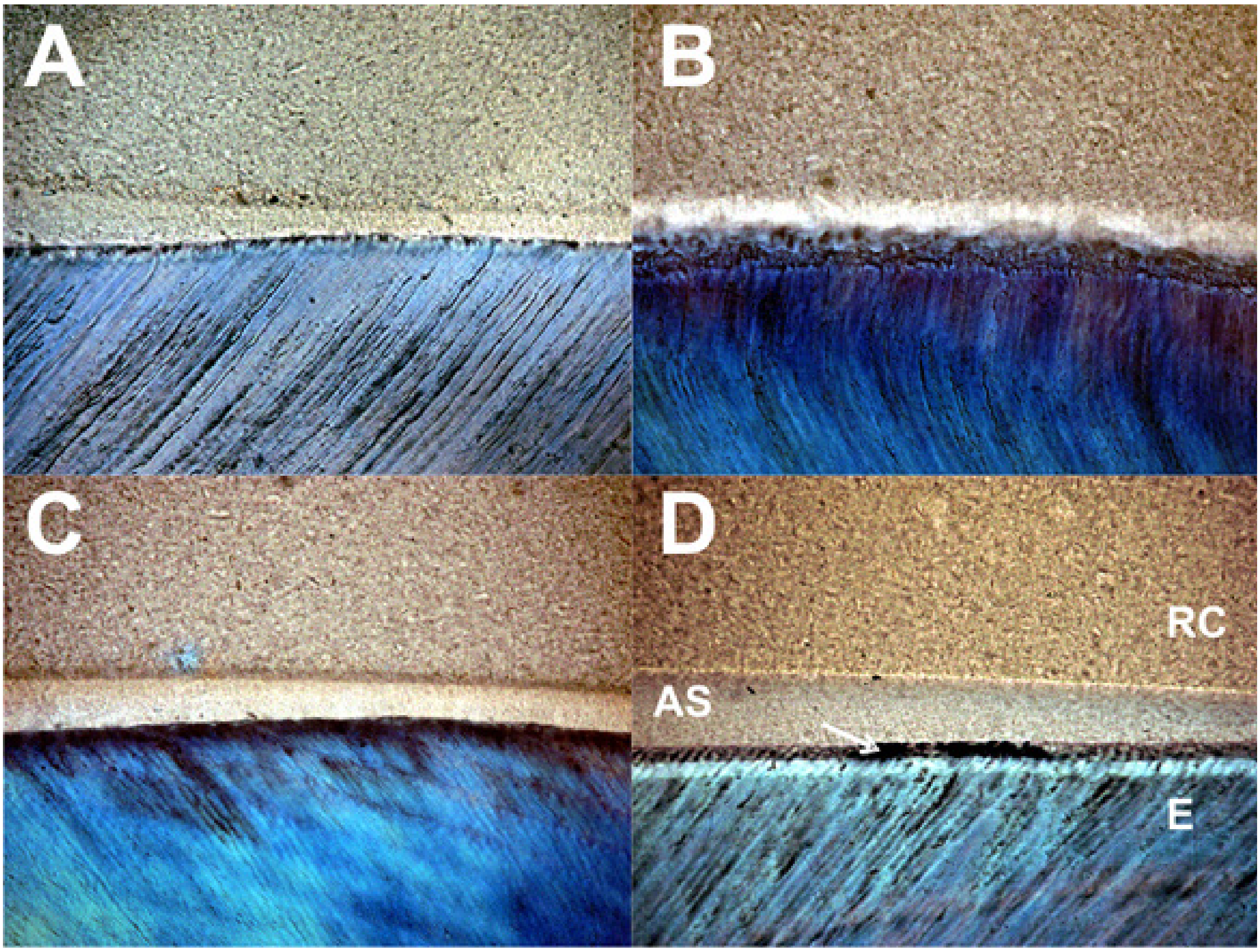

Figure 2 - Analysis of infiltration with silver nitrate in enamel/adhesive interface using Single Bond Adper Plus adhesive. A: ue; B: ge; C: ue/aged; D: ge/aged. AS: Adhesive system; RC: Resin composite; E: Enamel; Row: silver nitrate infiltration (adhesive interface).

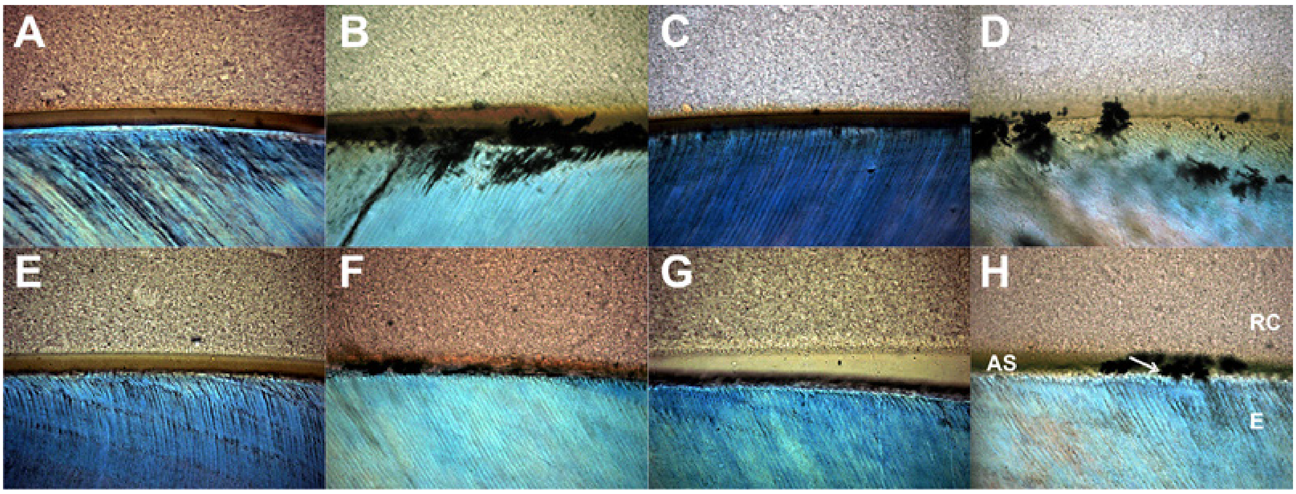

Figure 3 - Analysis of infiltration with silver nitrate in enamel/adhesive interface using Clearfil Tri-S Bond adhesive. A: ue; B: ue/aged; C: ge; D: ge/aged; E: pha/ue; F: pha/ue/aged; G: pha/ge; H: pha/ge/aged. AS: Adhesive system; RC: Resin composite; E: Enamel; Row: silver nitrate infiltration (adhesive interface).

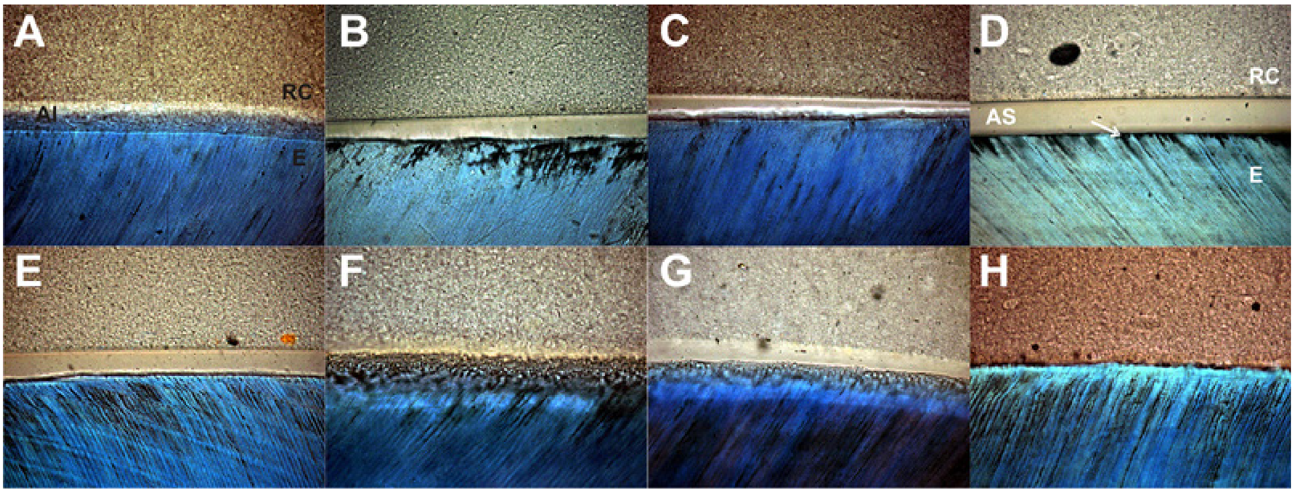

Figure 4 - Analysis of infiltration with silver nitrate in enamel/adhesive interface using Scotchbond Universal adhesive. A: ue; B: ue/aged; C: ge; D: ge/aged; E: pha/ue; F: pha/ue/aged; G: pha/ge; H: pha/ge/aged. AS: Adhesive system; RC: Resin composite; E: Enamel; Row: silver nitrate infiltration (adhesive interface).

Table III - Means (SD) of infiltration (%) per group

		SB	CF	CF/pha	SBU	SBU/pha
Ungroud Enamel	24h Thermocycling	0.01 (0.03) **a** 1.14 (0.82) **a**	14.35 (37.77) **a** 80.56 (33.23) **b**	8.48 (10.65) **a** 10.25 (13.14) **b**	28.71 (48.70) **a** 72.87 (46.33) **b**	14.47 (37.72) **a** 14.42 (37.74) **a**
Ground Enamel	24h Thermocycling	0.87 (1.68) **a** 10.63 (22.26) **a**	14.69 (37.62) **a** 50.11 (47.79) **b**	0.02 (0.02) **a** 2.52 (2.88) **b**	0.003 (0.01) **a** 75.70 (38.39) **b**	0.004 (0.01) **a** 0.84 (1.07) **a**

Different letters from it other indicate significant differences ($p < 0.05$). pha: phosphoric acid; SB: Single Bond Adper Plus (3M ESPE, St. Paul, MN, USA); CF: Clearfil Tri-S Bond (Kuraray America Inc, New York, NY, USA); SBU: Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA).

PLM analysis of tag quality

Figure 4 shows the differences in the length and quantity of tags relative to the adhesive technique and enamel surface. In the SB groups, the formation of resin tags was significantly increased (Figure 5 A/B), especially compared to the groups in which the self-etching technique (SBU and CF) was used. For the self-etching technique with the SBU and CF adhesives and unground/ground enamel, less tags were formed (Figure 5C/D). The groups in which the application of the self-etching adhesive (SBU and CF) was performed after etching with pha showed similar results as the group in which the conventional technique was used with the SB adhesive (Figure 5E/F). As for the substrate, the ground enamel surface interfered significantly in the tag quality, and longer and more numerous tags were formed for all of the adhesives tested. After the aging process was induced by thermocycling, the results were similar to those observed after 24 h (Figure 5).

DISCUSSION

This study investigated the quality of the adhesive interface before and after aging in PLM images in order to improve upon previous research on bond strength.[8] PLM tests that effectively analyze the bonding interface through assessments of infiltration[10] and tag formation [11] have been described previously. Good results with the SB adhesive have been reported,

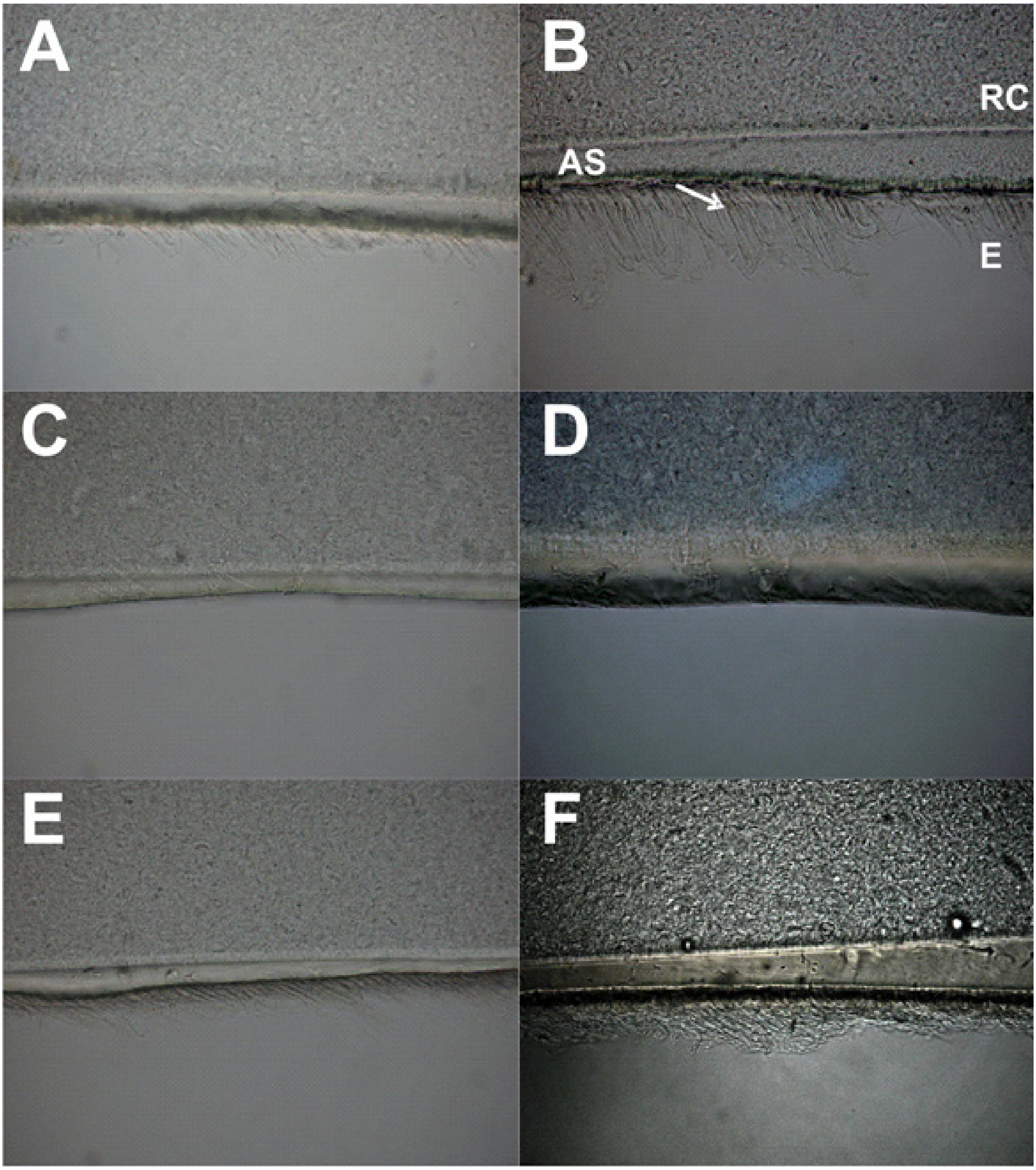

Figure 5 - Representative images of analysis of the quality of tags using Single Bond Adper Plus and Scotchbond Universal adhesive on enamel after aging. A: Adhesive system SB: ue/aged; B: Adhesive system SB: ge/aged; C: Adhesive system SBU: ue/aged; D: Adhesive system SBU: ge/aged; E: Adhesive system SBU: pha/ue/aged; F: Adhesive system SBU: pha/ge/aged; AS: Adhesive system; RC: Resin composite; E: Enamel; Row: tags formation.

[4,8] and it was therefore used as a control in this study for comparison with the self-etching adhesives.

With the advancements and improvements in the properties of restorative materials, a greater number of thermal cycles [3] are needed to evaluate the performance of the materials over time. The artificial aging effect that is induced by thermal cycling can stress the bond between the resin and the tooth and affect the integrity of the margin. [12] Furthermore, it can cause a microleakage phenomenon that may lead to staining and marginal breakdown. [12] Thus, 20,000 thermal cycles were performed in order to simulate the aging tooth/restoration interface.

Regardless of the adhesive technique, the type of ground/unground enamel surface influenced tag formation, and the ground enamel surface interfered significantly in tag quality, resulting in longer and more numerous tags in all of the adhesives tested. The unground enamel surface had less demineralization compared with surfaces that had been subjected to some kind of preparation because the adhesion occurred on the aprismatic enamel layer, which is hypermineralized. Therefore, tag formation may have been reduced. [1,3,8,13] According to Mine et al. [14] the surface preparation method significantly affected the nature of the smear layer and the interaction with the AS. They showed that preparation with 600-grit SiC sandpaper removed particles from the hydroxyapatite surface that interacted with the adhesive monomer, while the 1,200-grit SiC sandpaper promoted similar wearing of the extra-fine diamond tip that holds the hydroxyapatite particles. Thus, the 1,200-grit sandpaper promoted better interaction of the self-etching adhesive with the substrate.

Nonetheless, the surface condition did not influence the silver nitrate infiltration results, which led us to reject the second null hypothesis. This result possibly occurred because the micromechanical adhesion and the chemical interactions of the hydroxyapatite bifunctional monomer provided greater longevity to the adhesive restorations. [2,38] The chemical adhesion that was promoted by the adhesive and the micromechanical bond that was established by the formation of tags after etching the surface contributed significantly to the pha/SBU group exhibiting the best results. To illustrate this procedure, Figure 6A shows a clinical case in which the enamel was etched with 37% pha before the application of SBU and restored with the Filtek Supreme XT resin composite (Figure 6B). Figure 6C shows the restoration after 3 years, and a good marginal adaptation without infiltration/pigmentation is observed.

According to the Accession and Decalcification concept described by Yoshida et al. [15] the adhesion and decalcification phenomena of carboxylic acids that interact

Figure 6 - Clinical case of tooth restoration (11/21) with Scotchbond Universal and the Filtek Supreme XT composite resin. A: Application of Scotchbond Universal on the enamel after pre-etching with 37% phosphoric acid. B: Final appearance immediately after restoration with the Filtek Supreme XT composite resin. C: Composite resin restoration after 34 months.

with hydroxyapatite become less soluble and susceptible to hydrolytic degradation. Yoshida et al. [16] described the formation of a protective layer or nanolayer by the interaction of the smear layer with the adhesive that secures the hydrolytic degradation of the hybrid layer. [5,16,17]

The combination of the bifunctional monomer 10-MDP and the Vitrebond copolymer may have contributed to the satisfactory results that were obtained with the multi-mode SBU system. 10-MDP simultaneously binds through an ionic bond to calcium (Ca) and extracts Ca hydroxyapatite, thus forming a MDP-Ca salt. The remainder of the 10-MDP monomer remains attached to hydroxyapatite. [2,3,8] The Vitrebond copolymer interacts with Ca and is degraded less by contact with water. [18] However, even though the CF adhesive also contained 10-MDP, the system used in this study was not as effective compared with SBU and SB after aging, resulting in a significant increase in infiltration. These observations indicated that the presence and concentration of the components in the adhesive that were associated with the same etching technique were subjected more to degradation of the adhesive interface by aging. However, studies that compared Clearfil 3S with Clearfil SE showed better results when the two-bottle system was used. Reis et al. [19] evaluated the bond strength of 6 self-etching systems, including Clearfil 3S and Clearfil SE, to enamel and dentin, and they observed better performance and a lower number of specimens that were taken off prematurely when the two-bottle system was used.

Since Buonocore proposed etching to improve adherence, several studies [2,13] have shown that the etching of the enamel surface increases the adhesion surface, resulting in the formation of a better micromechanical bond in the composite resin restorations. The images that were obtained in this study showed that, after 24 h, the results after the use of pha were similar in all of the groups. After aging, enamel etching with pha increased the number and length of the tags for all of the tested adhesives and increased the infiltration values after aging when it was used with the conventional technique and self-etching adhesives. The use of the self-etching technique demonstrated superior infiltration results compared to the other groups, which was possibly due to the smaller amount of demineralization of the enamel adhesive systems. [1,3] However, the qualitative images showed little tag formation and larger areas of infiltration. Therefore, the chemical interaction with the hydroxyapatite enamel that is improved by the self-etching technique was not sufficient by itself because aging resulted in higher infiltration areas in the samples. The increase in infiltration after aging may result from the formation of cracks in the adhesive interface from hot water, which may accelerate the hydrolysis of unprotected collagen and extract poorly polymerized resin oligomers due to the repetitive contraction/expansion stresses that are generated at the tooth-biomaterial interface. [2,5]

Studies on dentin have reported a discrepancy between the demineralized area and the penetration of the adhesive, even for self-etching [7] adhesives, which suggests movement of water within the interface in areas that are not infiltrated by the hybrid layer and within the adhesives. [4,6] The presence of water in the adhesive matrix results in regions of incomplete polymerization and increasing permeability within the interface. [20] Clinically, stress that is generated at the interface during tooth function, together with chewing and mechanical/thermal stress, can trigger the deterioration of existing gaps or create new ones. [2,20]

Many of the specimens that were subjected to thermocycling were lost during the manipulation of the slices because of the detachment of portions of the resin, which was probably a result of weak enamel bonding. Thus, the first null hypothesis was accepted because aging affected the behavior of the various bonding techniques, and the groups in which the self-etching technique was applied showed superior infiltration results.

However, clinical studies [21] that have examined ASs with the same type of acid monomer (10-MDP) have described the effectiveness of the self-etching technique in composite resin restorations. According to Peumans et al. [22] after 8 years of use, the clinical effectiveness of the restorations that were created with the composite resin Clearfil SE were satisfactory compared with the restorations that were created with the conventional technique. [8,23] However, these results are controversial. Dermirci et al. [24] compared the use of different techniques for applying the same adhesive and found higher infiltration in the group in which the self-etching technique was performed. Therefore, it is still necessary to study other factors that affect the performance and longevity of restorations in order to better understand the technique and correct use of the material.

Thus, within the limitations of this study, it was observed that, after 24 h, there was no statistical difference in the infiltration analysis of all of the adhesive techniques tested. However, aging was found to interfere with the infiltration results as some groups had a higher infiltration area after thermal cycling. Although the current self-etching ASs have performed well on dentin, their performance on enamel is still unclear. It will be necessary to perform the acid etching for satisfactory adhesion because the etching was observed to result in the formation of longer and more numerous tags and decrease infiltration.

CONCLUSION

The acid pre-etching of enamel with the multi-mode adhesive was fundamental for reducing the degree of infiltration of the adhesive interface after aging.

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