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Selective Enamel Etching: Effect on Marginal Adaptation of Self-Etch LED-Cured Bond Systems in Aged Class I Composite Restorations

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Clinical Relevance

Selective enamel etching was shown to be an effective approach to reduce gap formation in Class I composite restorations for one-step self-etch adhesives.

SUMMARY

The aim of this study was to evaluate the influence of previous enamel etch and light emitting diode (LED) curing on gap formation of self-etch adhesive systems in Class I composite restorations after thermomechanical aging (TMA). Thus, on 192 human molars, a box-shaped Class I cavity was prepared maintaining enamel margins. Self-etch adhesives (Clearfil SE and Clearfil S3) were used to restore the preparation with a microhybrid composite. Before application of the adhesives, half of the teeth were enamel etched for 15 seconds with 37% phosphoric acid; the other half were not etched. For the photoactivation of the adhesives and composite, three light-curing units (LCUs) were used: one polywave (Ultra-Lume LED 5, UL) and two single-peak (FlashLite 1401, FL and Radii-cal, RD) LEDs. After this, epoxy resin replicas of the occlusal surface were made, and the specimens were submitted to TMA. New replicas were made from the aged specimens for marginal adaptation analysis by scanning electron microscopy. Data were submitted to Kruskal-Wallis and Wilcoxon tests (α=0.05). Before TMA, when enamel was etched before the application of S3, no gap formation was observed; however, there were gaps at the interface for the other tested conditions, with a statistical difference ($p \leq 0.05$). After TMA, the selective enamel etching previous to the S3 application, regardless of the LCU, promoted higher marginal adapta-

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DOI: 10.2341/11-184L

tion compared to the other tested groups ($p \leq 0.05$). Prior to TMA, higher marginal integrity was observed, in comparison with specimens after TMA ($p \leq 0.05$). With regard to Clearfil SE and Clearfil Tri-S cured with FL, no differences of gap formation were found between before and after aging (5.3 ± 3.8 and 7.4 ± 7.5, respectively), especially when the Clearfil Tri-S was used in the conventional protocol. When cured with RD or UL and not etched, Clearfil Tri-S presented the higher gap formation. In conclusion, additional enamel etching promoted better marginal integrity for Clearfil Tri-S, showing it to be an efficient technique for Class I composite restorations. The two-step self-etch adhesive was not influenced by selective enamel etching or by the LED-curing unit.

INTRODUCTION

Composite restorations have been widely used in clinical practice, due to esthetic and some biomechanical properties similar to dental hard tissues.[1] For the bonding procedure of these adhesive restorations, self-etching bond systems were introduced to decrease the number of bonding technical steps, since the presence of acidic monomers in their composition yields simultaneous etching and priming of the dental hard tissues.[2,3] A partial removal of the smear layer is promoted and, consequently, the formation of smear plugs left undisturbed, decreasing tooth postoperative sensitivity and leaving the adhesive protocol less time-consuming.[3-6]

Self-etching adhesives are known to exhibit a good bonding performance to dentin and a poor bonding behavior to enamel.[3,4] In order to remove the smear layer created by instrumentation, demineralize the enamel substrate, and increase bond quality and durability, a selective enamel phosphoric acid etching before the application of a self-etch adhesive has been proposed.[7-11] Some studies demonstrated that this additional enamel etching decreases gap formation when self-etch adhesives are used.[5,6,12-16] However, this additive step for direct composite restoration procedures was only evaluated in Class II, III, and V restorations.[5,6,13-16] Since the Class I preparation exhibits a high Configuration factor with enamel surrounding the superficial margins, it is considered the best model for understanding the real effects of enamel etching and the stress development on the tooth/adhesive interface.[14,17]

An adequate bond system cure is another important factor to consider in restoring Class I cavities to ensure a good bond performance and marginal integrity. An efficient light-curing unit (LCU) should guarantee a satisfactory adhesive and composite degree of conversion, which may improve its physical and mechanical properties, yielding a good marginal seal.[18-22] Quartz-tungsten-halogen (QTH) lamps have been largely used in restorative procedures; however, they present some drawbacks like bulb, filter, and reflector degradation over time and a lifetime of approximately 40 to 100 hours.[18-20] In this sense, light emitting diodes (LEDs) have been shown to overcome these problems, promoting an adequate cure of resin composites and dental adhesives through emission of unfiltered blue light.[18-20] These LCUs generate a narrow spectral range that targets the absorption wavelength of camphorquinone, yielding low amounts of wasted energy and minimum heat generation, with a longer lifetime and less decrease of light intensity.[18-20] Thus, the curing potential of current LED curing units has been shown to be similar to that presented by conventional QTH light units.[23-25]

It is known that some resinous materials, like resin cements, and some adhesive systems are not well cured with conventional single-peak LEDs due to alternative photoinitiator content.[26] Because of this, LEDs with additional wavelengths (polywave third generation LEDs) were developed, emitting light wavelengths within a spectral region that targets the absorption peak of camphorquinone and within the UV-VIS region (400–415 nm).[23-25] This polywave behavior is expected to yield an adequate cure of adhesives and composites that contain alternative photoinitiator systems, such as phenyl propanedione, bis-alkyl phosphinic oxide, and trimethylbenzoyl-diphenyl-phosphine oxide.[25] Since manufacturers do not state all photoinitiator content, is it important to choose an adequate LCU that would polymerize all resinous materials,[25-27] enhancing the marginal seal and improving the composite restorations' durability.

Thus, this study aimed to investigate the influence of prior enamel etching and LED curing lights on gap formation of self-etch bond systems in Class I composite restorations. The first tested hypothesis was that selective enamel etching would improve the marginal adaptation of the adhesive systems in Class I composite restorations. The second hypothesis was that the third generation polywave LED would present lower gap formation for the tested bond systems. Moreover, the third hypothesis was that the thermomechanical fatigue would increase

Table 1: *Composition, Application Mode, and Manufacturers' Information for the Adhesive Systems Tested*

Adhesive Systems	Composition	Application Mode
Clearfil SE Bond (Kuraray Medical Inc, Tokyo, Japan)	Primer (batch 00896A): water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate. Adhesive (batch 01320A): MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica.	Apply primer for 20 seconds. Mild air stream. Apply bond. Gentle air stream. Light cure at an energy density of 11 J.
Clearfil Tri-S Bond (Kuraray Medical Inc, Tokyo, Japan)	Adhesive (batch 00116A): MDP, bis-GMA, HEMA hydrophobic dimethacrylate, dl-camphorquinone, silanated colloidal silica, ethyl alcohol, and water.	Apply the bond system for 20 seconds. Gentle air stream for 10 seconds. Light cure at an energy density of 11 J.
Charisma (Heraeus Kulzer, Hanau, Germany)	Composite (batch 010080): Bis-GMA, TEGDMA, barium aluminum boro silicate glass and pyrogenic silicon dioxide, photoinitiators (64% filler),	Light cure at an energy density of 22 J.

interfacial debonding, promoting higher gaps at the superficial margins.

MATERIALS AND METHODS

One hundred ninety-two healthy human third molars were selected. The teeth were collected after obtaining the patient's informed consent under a protocol approved by the State University of Campinas ethical review board (057/2009). The teeth were cleaned, embedded in polystyrene resin, and their occlusal surfaces were wet polished with 320-grit SiC paper under running water (Politriz, AROTEC, São Paulo, Brazil) to expose a flat enamel surface area without exposing dentin. Then, typical Class I cavities were prepared using no. 56L carbide burs (KG Sorensen, Barueri, Brazil) at high-speed, under air/water cooling. After five-cavity preparations, the bur was replaced. Preparations had a standard size, with cavity dimensions of 5 mm mesial-distally, 5 mm buccal-lingually, and a 3 mm depth, maintaining all cavity margins on enamel substrate. Two self-etch adhesive systems were used for the bonding protocols: Clearfil Tri-S Bond (one-step self-etch, pH = 2.7, Kuraray Medical Inc, Okayama, Japan) and Clearfil SE Bond (two-step self-etch, pH = 2, Kuraray Medical Inc). The composition, application mode, and batch number of the adhesive systems are presented in Table 1. After cavity preparation, teeth were assigned to 12 groups (n=16) according to the three studied factors: selective enamel etching, bond system, and curing light (two conditioning protocols × two adhesives × three LEDs).

For the photoactivation procedure three LEDs were tested: FlashLite 1401 (FL) (Discus Dental, Culver City, CA, USA), Radii-cal (RD) (SDI Limited, Victoria, Australia), and Ultra-Lume LED 5 (UL) (Ultradent, South Jordan, UT, USA). Prior to the restorative procedure, the output power (mW) of each LCU was measured with a calibrated power meter (Ophir Optronics, Har, Hotzvim, Jerusalem, Israel). Then, irradiance (mW/cm^2) was determined by dividing the output power by the tip end area. Spectral distributions were measured with a calibrated spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA). Beam distribution and irradiance data were integrated using the Origin 6.0 software (OriginLab, Northampton, MA, USA). The spectral range distribution of each LCU is shown on Figure 1, and the characteristics of the LCUs are presented in

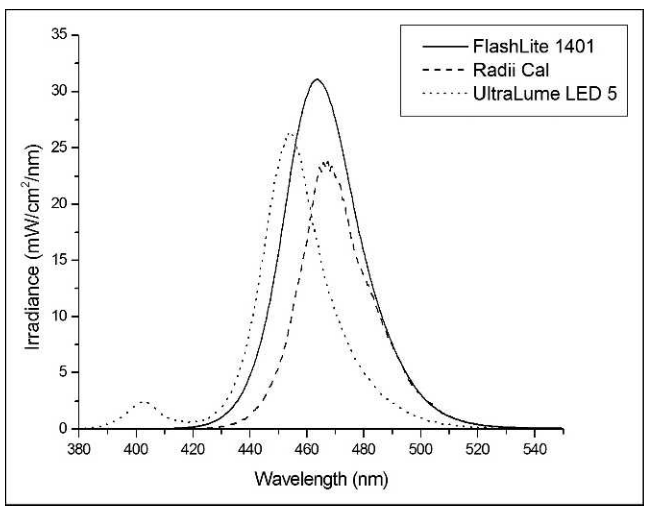

Figure 1. *The spectral range distribution of each LCU used in this study.*

Table 2: *Characteristics of the Light-Curing Units (LCUs) Used in This Study*

LCU	Manufacturer	Type	Tip Diameter, mm	Irradiance, mW/cm^2	Composite Radiant Exposure, J/cm^2	Adhesive Radiant Exposure, J/cm^2
FlashLite 1401	Discus Dental, Culver City, CA, USA	Single-peak	7	1077	22	11
Radii-cal	SDI Limited, Victoria, Australia	Single-peak	7	731	22	11
Ultra-Lume LED 5	Ultradent Products Inc, South Jordan, UT, USA	Polywave	11 × 7	800	22	11

Table 2. Also, energy density was standardized to approximately 11 J for curing the bonding system and 22 J for each composite increment photoactivation. For RD, due to the fact that it mandatorily operates in ramp mode for 5 seconds, these initial seconds were discarded, and only a continuous light was delivered to keep the irradiance standardized. Consequently, an equal energy density was obtained for each of the LCUs.

All groups were restored with B1-shade Charisma microhybrid composite (Heraeus Kulzer, Hanau, Germany), using an incremental oblique technique with six increments of approximately 2 mm thick. The first layer was applied horizontally and light cured, followed by two oblique layers. Next, another three layers were placed in the same way as described before, until the cavity was completely filled. Then, finishing and polishing procedures were performed with medium-, fine-, and extra fine-grit aluminum oxide disks (SoftLex, 3M/ESPE, St Paul, MN, USA), respectively. After polishing, impressions with a low viscosity vinyl polysiloxane material (Express XT, 3M ESPE) of the teeth were taken and a first set of epoxy resin replicas (Epoxicure Resin, Buehler, Lake Bluff, IL, USA) was made for scanning electron microscopy (SEM) evaluation. In sequence, specimens were submitted to 200,000 mechanical loading of 40N (2 Hz) and 500 thermal cycles (ranging from 5°C to 55°C with a dwell time of 60 seconds in each bath with an interval of 5 seconds) in a thermomechanical device ER-11000 (ERIOS, São Paulo, Brazil) to simulate aging of the composite restorations in oral environment conditions.

New impressions of the teeth were made and another set of replicas was made for each restoration. All replicas were mounted on aluminum stubs, sputter coated with gold, and evaluated with a scanning electron microscope (JEOL, JSM-5600LV, Tokyo, Japan) at 200× magnification. SEM analysis of the composite/enamel marginal adaptation was performed by one operator having experience with quantitative margin examination and who was blinded to the restorative procedures. The marginal integrity between resin composite and enamel was expressed as a percentage of the entire superficial margin length.

Enamel Etching Patterns

Eighteen half-teeth (n=3 for the two self-etch adhesives with and without prior enamel etch and negative and positive controls) were ground and randomly assigned into 12 groups. For the positive control, a 37% phosphoric acid treatment was realized and for the negative control, the enamel surface did not receive any treatment. The experimental groups were treated with the two tested bond systems using manufacturer's instructions with or without prior enamel etching. The treated surfaces were thoroughly rinsed with alternate baths of acetone (20 seconds) and ethanol (20 seconds) in an attempt to remove the self-etch primers and the monomer components. All specimens were dehydrated in ascending grades of ethanol (25%, 50%, 75%, and 90%) for 10 minutes each and immersion in 100% ethanol for 30 minutes. After dry storage at 37°C for 24 hours, specimens were sputter coated with gold and analyzed by SEM.

Statistical Analysis

For the statistical analysis, as the data did not exhibit normal data distribution (Kolmogorov-Smirnov test), nonparametric tests were used (Kruskal-Wallis for groups' comparison and Wilcoxon matched-pairs signed-rank tests for pairwise com-

Table 3: *Results of the Gap Formation Analysis (% and SD) of Enamel Margins Before and After Thermomechanical Aging)*†

Tested Conditions	Before Aging, % (SD)	After Aging, % (SD)
FlashLite/no etch/Clearfil SE	2.6 (4.7)B	5.6 (4.5)AB*
FlashLite/etch/Clearfil SE	0.6 (1.3)B	3.0 (2.8)AB*
FlashLite/no etch/Clearfil Tri-S	5.3 (3.8)B	7.4 (7.5)AB
FlashLite/etch/ Clearfil Tri-S	0A	2.4 (4.9)A*
Radii-cal/no etch/Clearfil SE	3.2 (3.9)B	13.1 (14.7)AB*
Radii-cal/etch/Clearfil SE	3.6 (8.2)B	6.2 (8.6)AB
Radii-cal/no etch/ Clearfil Tri-S	3.0 (3.1)B	12.4 (10.7)B*
Radii-cal/etch/ Clearfil Tri-S	0A	2.9 (5.7)A
Ultra-Lume/no etch/Clearfil SE	3.5 (4.0)B	3.9 (4.4)AB
Ultra-Lume/etch/Clearfil SE	3.7 (3.7)B	4.8 (4.9)AB
Ultra-Lume/no etch/ Clearfil Tri-S	3.1 (4.3)B	8.6 (7.5)B*
Ultra-Lume/etch/ Clearfil Tri-S	0A	0.9 (2.61)A

† *Same letters within column indicate no statistically significant difference (*$p \leq 0.05$*, Kruskal-Wallis and Dunn comparison). Asterisks stand for* $p \leq 0.05$*; Wilcoxon matched-pairs and signed-rank tests.*

parisons before and after thermomechanical aging) with a pre-set alpha of 0.05.

RESULTS

Marginal Adaptation Analysis

The results of the marginal adaptation analysis are shown in Table 3. No gap formation was observed before thermomechanical loading when enamel was etched before the application of Clearfil Tri-S; however, there were gaps at the interface of the Clearfil SE Bond regardless of the energy source, with statistical difference (Kruskal-Wallis test; $p \leq 0.05$). After aging, selective etching prior to the Clearfil Tri-S application, when RD or UL were used, promoted higher marginal adaptation compared to the other tested groups (Kruskal-Wallis test; $p \leq 0.05$). Figure 2 shows examples of marginal integrity and marginal gaps of Class I composite restorations that were observed in this study.

Prior to thermomechanical loading, higher marginal integrity was observed in comparison with after aging (Wilcoxon test; $p \leq 0.05$). When the bonding systems were cured with FL and only when Clearfil Tri-S was used in the conventional way were there no differences between before and after aging (5.3 ± 3.8 and 7.4 ± 7.5, respectively). When cured with RD or UL and not etched, Clearfil Tri-S presented the higher gap formation.

Enamel Etching Patterns

The SEM enamel etching patterns are shown in Figures 3 (A,B), 4 (A,B), and 5 (A,B,C).

DISCUSSION

An important factor to promote clinical success of Class I resin composite restorations is a satisfactory enamel marginal adaptation. The presence of gaps is considered as the first sign of restoration failure, clinically evidenced by marginal staining.[28] Also, when detectable marginal disruption is present, these interface defects could lead to interfacial leakage.[28,29] In the oral environment, many pulpal sensitivities and responses are related to when bacterial leakage occurs along the tooth/composite bonding interface.

In the present work, the first hypothesis was partially validated, since only the gap formation for the one bottle all-in-one self-etch adhesive Clearfil Tri-S was affected by the selective enamel etching procedure, regardless of the LCU. This may be explained by some characteristics of this mild one-step adhesive, like its pH and etching potential.[3,4] As shown by SEM evaluation of the enamel etching patterns , Clearfil Tri-S alone promotes a smooth enamel demineralization, not increasing the surface free energy and bond penetration. Its pH of approximately 2.6 did not promote an adequate demineralization of the enamel surface and resulted in poor bond strength. This weak bond interface is easily affected by composite shrinkage stress during the photocuring procedure.[7,8,28] In this sense, the selective enamel etch procedure could have promoted a deeper dissolution of prism cores and boundaries in a type III etching pattern, increasing the surface free energy of this substrate and consequently, increasing the percentage of gap-free margins.

For Clearfil SE Bond, the previous acid etching did not affect the gap formation of the composite restorations. This may have occurred because of

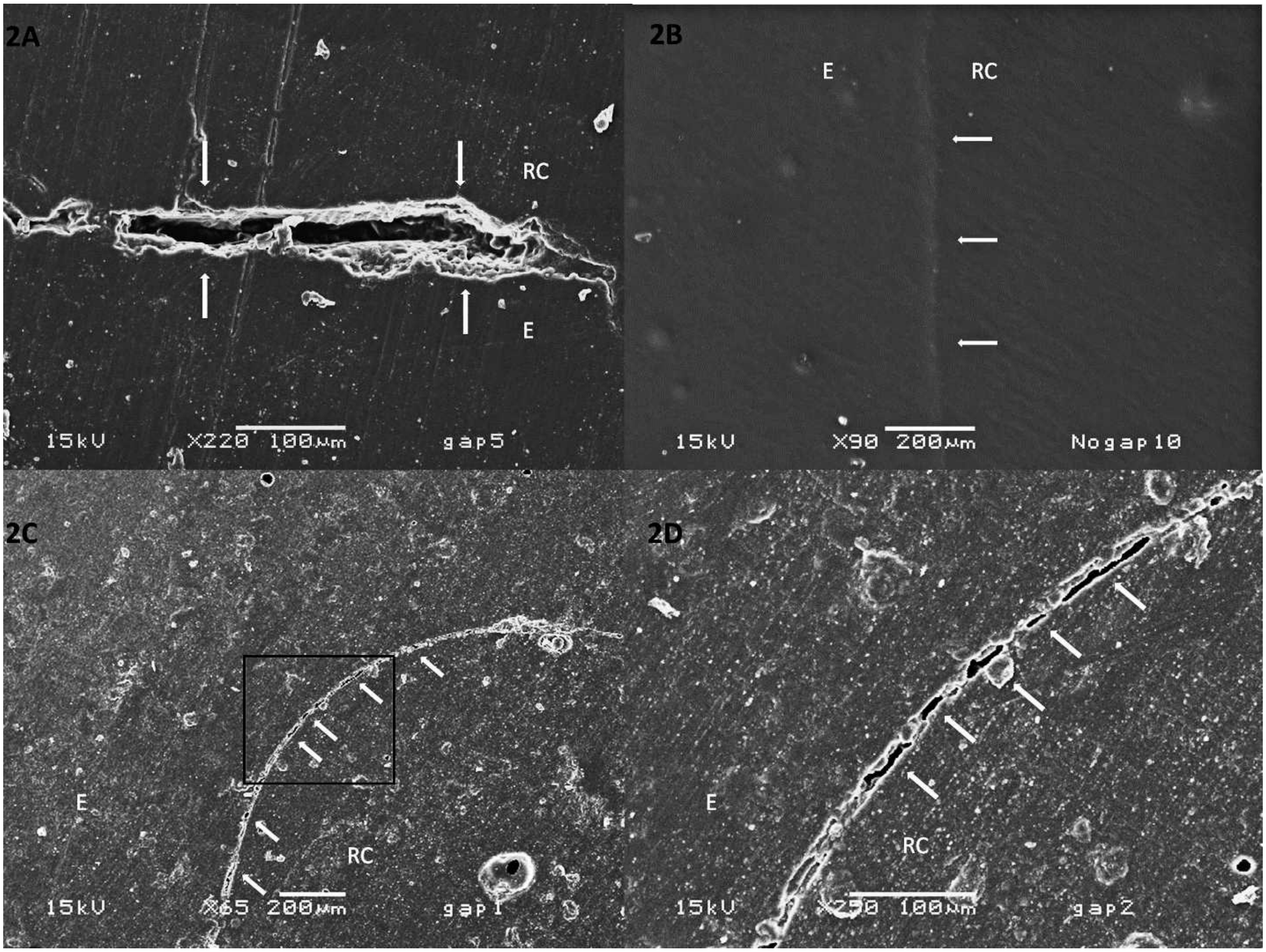

Figure 2. *(A): The white arrows point to interfacial gap formed between resin composite and enamel. (B): The white arrows indicate a perfect marginal seal between enamel and composite. (C): Note the composite/resin interface presenting some interfacial gaps. The white arrows point to the correct location of the marginal gaps. (D): A high magnification showing the interfacial gaps between composite and dental enamel.*

the lower pH of the adhesive system, approximately 2.0, which could have promoted higher enamel demineralization compared to Clearfil Tri-S alone, regardless of the curing light. Also, the studied two-step bond system contains a separate hydrophobic resin that is applied after the acidic primer. This hydrophobic resin coat can improve bond durability, especially due to the structural polymer network that is not hydrophilic and can maintain optimal bonding behavior after fatigue stress.[8,30,31]

The second hypothesis of this study was rejected because the polywave third-generation LED did not improve the marginal adaptation of the resin composite restorations. The photoinitiator content of the composite and adhesive resin formulations seems to be camphorquinone, as informed by manufacturers. Therefore, the polywave LED for camphorquinone-based resin exhibited a similar behavior as the single-peak second generation LED. This was demonstrated by the fact that both adhesive systems and the microhybrid composite with camphorquinone did not have interfacial integrity affected. Also, they may have presented a similar degree of cure and consequently, less marginal shrinkage stress, preserving the superficial marginal adaptation between composite and enamel.

Another fact to discuss is the morphology of the Ultra-Lume LED 5 tip, in which there is a central LED that emits visible light at the peak spectra of camphorquinone and four accessory LEDs in the corner of the tip that emit UV-VIS wavelengths. In a Class I preparation, with 25 mm^2, as the condition presented in this work, light emitted by the accessory LEDs would not reach the adhesive resin

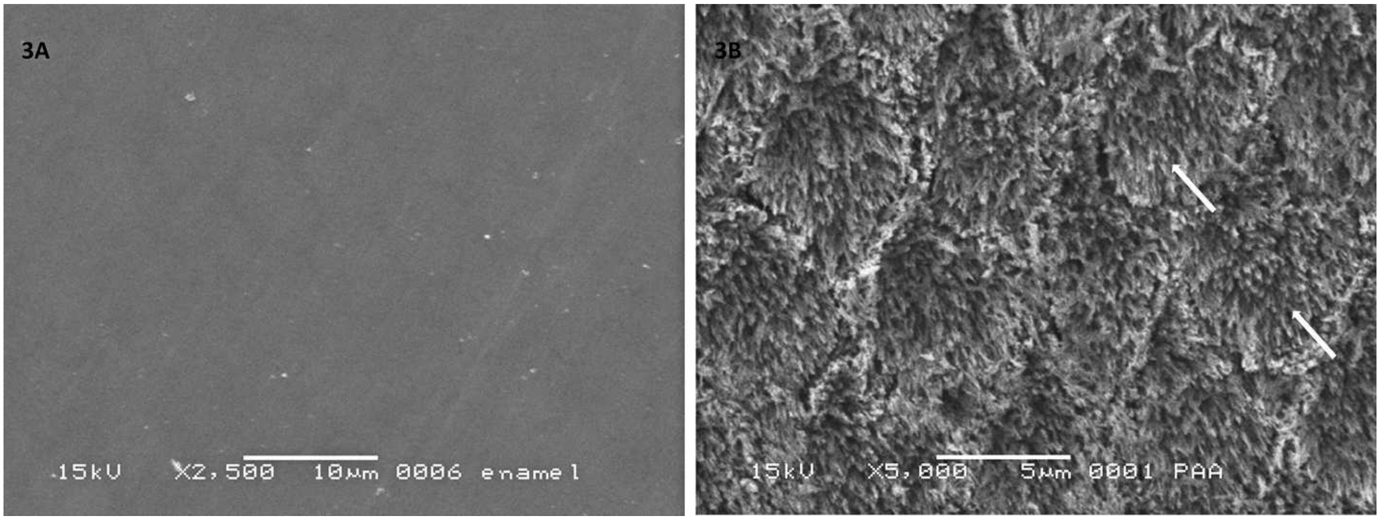

Figure 3. *(A): SEM photomicrograph showing the dental enamel without any acid treatment. (B): Phosphoric acid (37%) etched enamel, showing a type II etching pattern, with the dissolution of the prism cores.*

since the tip dimensions are higher than the area of the tooth preparation.[24] Consequently, only the central LED with the absorption peak of camphorquinone may have irradiated the adhesive resin, promoting similar gap formation as FlashLite and Radii-cal. This fact is not in agreement with a previous study, which found that polywave LEDs promote better resin mechanical properties compared to single-peak LEDs, even when the light curing tip end is located at a long distance.[22]

For half of the tested groups the thermomechanical loading promoted higher gap formation, partially validating the third hypothesis. When specimens were restored using Clearfil Tri-S without selective enamel etching, the thermomechanical aging promoted less marginal integrity, except when FL was used for photopolymerization. The previous enamel etch may have guaranteed a higher bond performance due to the increase in the enamel surface free energy caused by a deeper enamel demineralization.[4,5,14] The etching pattern of Clearfil Tri-S with previous acid treatment shows a deeper demineralization of enamel cores and boundaries, favoring the penetration of the adhesive resin. Consequently, the

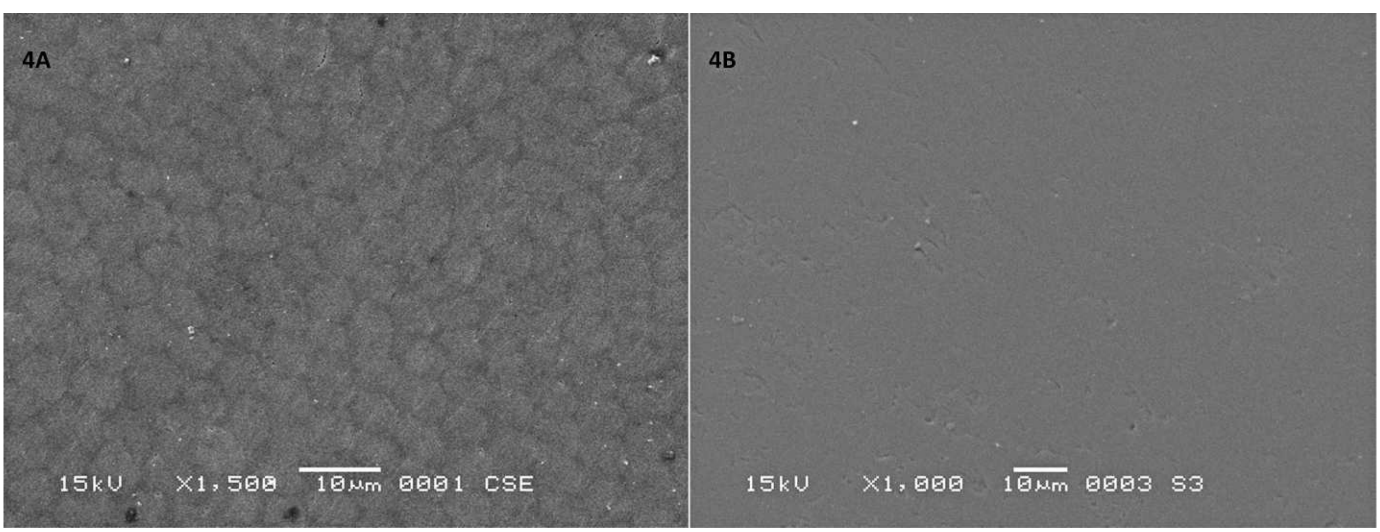

Figure 4. *(A): SEM photomicrograph showing the smooth enamel etching promoted by Clearfil Tri-S, with no dissolution of prism cores and boundaries. (B): SEM photomicrograph showing the smooth primer etching of Clearfil SE, with a type I etching pattern, with only dissolution of prism boundaries.*

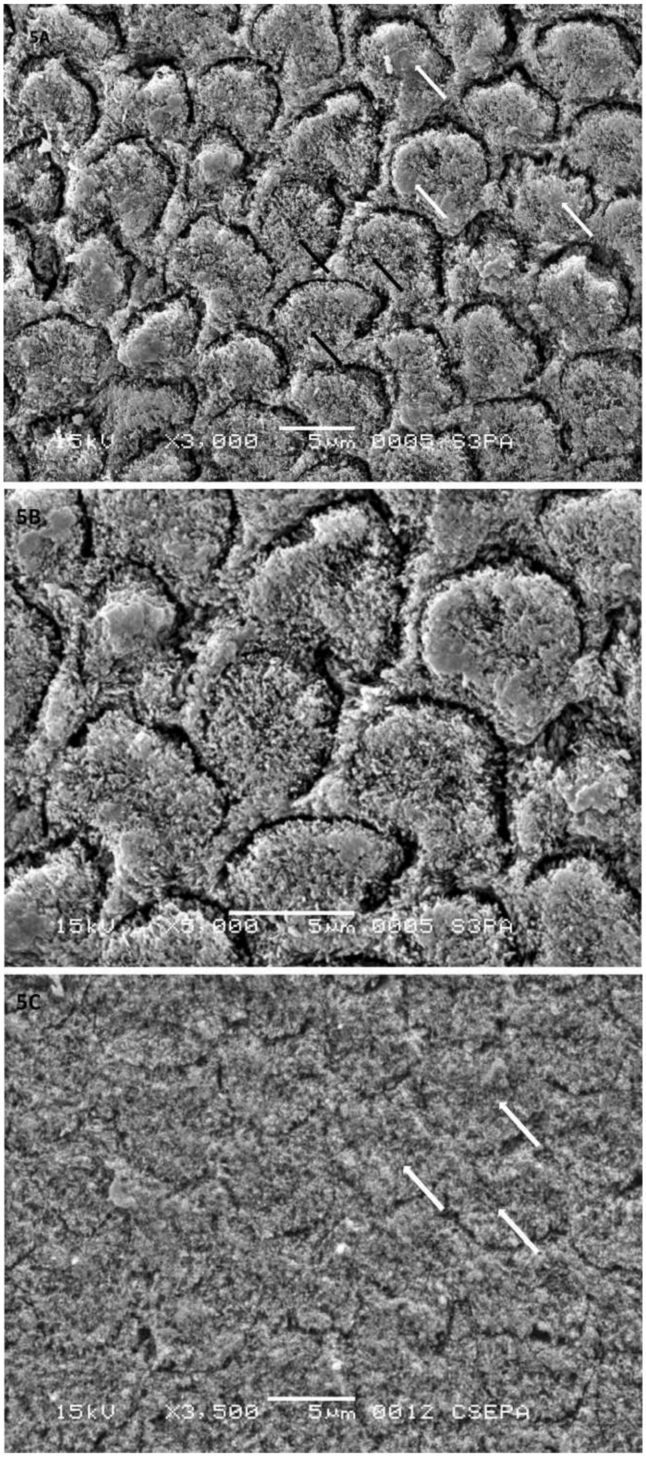

Figure 5. *(A): SEM photomicrograph of Clearfil Tri-S with previous phosphoric acid etching, showing a deep dissolution of prism cores and boundaries, with a type III etching pattern (characteristics of the type I and II). The white arrows point to the characteristics of type I etching pattern, with intact prism core, while the black arrows show characteristics of the type II etching pattern, with higher dissolution of prism peripheries. (B): SEM photomicrograph of Clearfil Tri-S with higher magnification 5000×. (C) SEM photomicrograph of Clearfil SE with previous acid etching, with a type III etching pattern, pointed out by the white arrows.*

thermomechanical effect was not capable of inducing higher gaps at the interface, preserving the enamel/composite bonding. For FL, the aging may not have affected bonding to enamel because this LCU emitted higher light irradiance compared to the other curing devices, even if the energy dose was standardized. This higher irradiance may have caused a bond disruption at the interface both before and after thermomechanical fatigue.[32-34]

For Clearfil SE, Ultra-Lume curing light promoted the maintenance of marginal integrity even after thermomechanical aging, regardless of additional enamel etching. This may be explained by the hydrophobic layer of this bond system which may have not been influenced by the composite shrinkage stress and consequently maintained the bond interface with no alterations. This additional layer can promote a higher monomer conversion and allow marginal integrity after thermomechanical loading. Also, when RD was used with enamel etching, the aging loading did not influence the percentage of marginal gaps.

Although some *in vitro* studies exist, final conclusions regarding the role of enamel selective acid etching for self-etch adhesives in Class I composite restorations will depend on the outcomes of clinical trials. Clinical long-term studies and investigations of the retention ability of this approach for bond systems in the oral environment can best evaluate the quality and durability of these restorations.

CONCLUSION

Selective enamel etching promotes better marginal integrity for Clearfil Tri-S, showing itself to be an efficient additional step for Class I composite restorations. The two-step self-etch adhesive was not influenced by selective enamel etching or by the LED-curing unit. In general, the mild one-step self-etch bond system preserved the marginal adaptation integrity when enamel was previously etched, except when the single-peak LED FlashLite, with higher irradiance, was used.

(Accepted 22 August 2011)

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