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Limited Etching Time Increases Self-adhesive Resin Cement Adhesion to Enamel

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Clinical Relevance

Enamel acid etching should precede the use of self-adhesive resin cements to obtain better adhesion and, thus, increased restoration longevity.

SUMMARY

Aim: To evaluate the influence of different enamel etching times on the bond strength of two self-adhesive resin cements (RCs) with and without thermocycling (TMC).

Methods: One hundred twenty bovine teeth were used. Blocks of enamel (8×4×2 mm) were obtained, polished, and randomly divided into two groups, according to the RC used: MaxCem Elite or RelyX U200. Groups were subdivided into four groups (n=16), according to the etching time: Control (0 seconds), 5 seconds, 10 seconds, and 20 seconds. Three RC cylinders (1-mm diameter) were built on each enamel block. The specimens were submitted to two storage conditions: 24 hours in distilled water or TMC (5000 cycles/5°C-55°C). Afterward, the specimens were submitted to the shear bond strength (SBS) test. The failure modes and adhesive interfaces were analyzed by scanning electron microscopy (SEM) and confocal laser scanning microscopy (CLSM). Data were analyzed with three-way analysis of variance (ANOVA) and Tukey test (α=0.05).

Results: Etching increased the SBS for both the RCs, especially for the groups etched for 5 and 10 seconds. TMC affected negatively the SBS of the control groups ($p<0.05$). No resin tags were observed in control groups, and the formation of tags was time dependent.

Conclusion: The 10 seconds etching time was more effective in increasing the enamel–resin bond strength. TMC negatively affected bond strength in specimens without acid etching.

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INTRODUCTION

The clinical success of indirect esthetic restorations depends on many factors, including appropriate material selection and use of a proper cementation technique.[1,2] The cementation is a sensitive and complex procedure that requires conditioning of restoration and tooth surfaces followed by the use of a resin cement (RC) as a luting agent.[3] RCs have the ability to bond the restorations with the tooth structure, guaranteeing durable and reliable bonding interfaces.[4]

However, the use of RCs requires multiple steps that might compromise their bonding effectiveness.[5,6] In order to simplify the number of clinical steps involved in the cementation procedure, self-adhesive RCs have been introduced.[3,6,7] Self-adhesive RCs combine features of the resin composites, dental cements, and self-etching adhesives. According to the manufacturers, self-adhesive RCs eliminate the need for pretreatments of the dental substrates.[2,8] Self-adhesive RCs contain acidic monomers (methacrylate phosphoric acids) that promote demineralization of the enamel and modification of the smear layer on the dentin surface.[3,6,7] Simultaneously, the conditioned surfaces are infiltrated by adhesive monomers, producing micromechanical retention; in parallel, chemical bonding occurs between the acidic monomers and on the tooth hydroxyapatite.[2,3,6,7] Thus, the necessity of the acid etching step was eliminated.[9]

Despite the advantages offered by self-adhesive RCs, the bond strength achieved by them on enamel is lower than those achieved by conventional RCs.[1,10] It is known that the acidic monomers contained in the self-adhesive RCs are not sufficiently strong to promote adequate conditioning of the enamel.[7] The weak acidity of the RCs might create a shallower etching pattern in the enamel that results in lower micromechanical retention.[1]

In order to improve the bond strength between the enamel and self-adhesive RCs, an additional pretreatment of the enamel with phosphoric acid has been proposed.[3] The increase of the bond strength in the enamel may prolong the clinical longevity of indirect restorations. Several studies have already demonstrated that acid etching with 37% phosphoric acid improves the adhesion between the enamel and self-adhesive RCs.[4,5,11,12] However, the most appropriate enamel etching time for self-adhesive RCs is still not clear.

On the other hand, no long-term data exists related to the stability of the adhesion between self-adhesive RCs and enamel treated with different etching times. In this respect, it is known that the adhesive interfaces could suffer degradation resulting in bonding failure. Thermocycling (TMC) is a widely used laboratory method to accelerate the degradation of bonding interfaces, producing dimensional changes in the substrates and affecting the adhesive interface.[13-15]

Therefore, the aim of this study was to evaluate the influence of phosphoric acid etching time on the bond strength between two self-adhesive RCs and enamel immediately and after TMC. The interfacial morphology was analyzed through Scanning Electron Microscopy (SEM) and Confocal Laser Scanning Microscopy (CLSM). The null hypotheses tested were that (1) there is no difference in the bond strength to enamel and in its morphology, regardless of the etching time; and (2) the TMC does not affect the bond strength stability to enamel.

MATERIALS AND METHODS

Tooth Preparation

One hundred twenty bovine incisors, without caries, fractures, or cracks were selected and stored in deionized water at 4°C. The bovine incisors were cut at the cementoenamel junction using a diamond saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) under running water. The roots were discarded, and the pulp chambers were thoroughly cleaned and washed.

Blocks of $8 \times 4 \times 2$ mm were obtained from the buccal surfaces of the crowns. The blocks were individually embedded in acrylic resin (VipiFlash, Pirassununga, SP, Brazil) using PVC molds with 12-mm diameter. The buccal surfaces were ground flat using 180-grit silicon carbide (SiC) sandpaper on a polishing machine (Aropol-E, Arotec Diagnostics, Cotia, SP, Brazil) under water cooling to achieve a flat enamel surface and then polished with 600-, 1200-, and 2000-grit SiC sandpaper. The final polishing was performed with a 1-μm diamond solution (MetaDi Supreme; Buehler Ltd., Lake Buff, IL, USA).[16] Immediately, the specimens were cleaned (Ultrasonic Cleaner USC 750; Unique Group, São Paulo, SP, Brazil) for 5 minutes. Afterward, the specimens were examined under 20× magnification (Leica MZ6; Leica Microsystems, Wetzlar, Germany), and the specimens with any enamel surface alteration were excluded and replaced. Finally, the specimens were stored in deionized water at 4°C.

Table 1: *Composition and Batch Number of the Materials Used in This Study*

Material	Name Shade/Batch Number Manufacturer	Composition
Self-adhesive resin cement (RC)	MaxCem Elite Clear/5365521 Kerr Corporation, Orange, CA, USA	*Bis*-GMA, UDMA, GPDM, glyceroldimethacrylate, mono-, di-, and multimethacrylate co-monomers, barium aluminum borosilicate glass, fluoraluminum silicate glass, stabilizer, CQ, others.
	RelyX U200 Translucid/562548 3M Oral Care, St. Paul, MN, USA	Base: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiators, stabilizers, rheological additives. Catalyst: Methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives. Zirconia/silica fillers
Phosphoric Acid (PA)	Etch-37 630001725 Bisco Inc, Schaumburg, IL, USA	Phosphoric Acid (37%), Benzalkonium chloride (1%)

Abbreviations: Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane; CQ, camphorquinone; GPDM, glycerol phosphate dimethacrylate; UDMA: urethane dimethacrylate.

Bonding Procedure

The specimens were randomly divided into two groups, according to the cement used: MaxCem Elite (Kerr Dental, California, USA) or RelyX U200 (3M Oral Care, MN, USA). Both the groups were subdivided into four subgroups according to the etching times: Control (0 seconds), 5 seconds, 10 seconds, and 20 seconds. Enamel surfaces were etched with 37% phosphoric acid (Etch-37, Bisco Dental Products, Schaumburg, IL, USA) for 5 seconds, 10 seconds, or 20 seconds, followed by water rinsing for 30 seconds and air-drying for 20 seconds.

Silicone molds (Oranwash L, Zhermack, Badia Polesine, RO, Italy) containing three cylinder-shaped orifices (2-mm high and 1-mm diameter) were placed onto the enamel surfaces. The cylindrical orifices were filled with the self-adhesive RCs, following the manufacturer's instructions and light cured for 20 seconds using a light-curing LED unit (BluePhase G2, Ivoclar Vivadent, Schaan, Liechtenstein) with radiant emittance of 1200 mW/cm^2. The silicone molds were sectioned and carefully removed, and the specimens were stored in deionized water at 37°C for 24 hours. Three cylinders were built on each enamel surface, totaling 48 cylinders per group. The composition of the materials used is in Table 1.

Artificial Aging Protocols

Each subgroup of specimens was further divided into two groups (n=8) according to the aging protocol: (i) specimens were stored for 24 hours in deionized water or (ii) specimens were thermocycled (MCT-2; MM Co., São Carlos, SP, Brazil) for 5000 cycles between 5°C and 55°C, with a 30 second dwell time and a 6 second transfer time.

Shear Bond Strength (SBS)

After the artificial aging conditions, the specimens were fixed in a universal testing machine (Instron 4411; Instron Corporation, Canton, MA, USA), and the SBS was performed using a steel wire (0.009" diameter) that was looped around each RC cylinder, then a load was applied at a crosshead speed of 1 mm/min until failure.

Fractographic Analysis

The specimens were sputter-coated with gold (Bal-Tec SCD 050, USA) and analyzed under a scanning electron microscope (SEM) (JSM-5600LV, JEOL Ltd, Tokyo, Japan). Representative photomicrographs were obtained from all the specimens. The failure patterns were classified as follows: adhesive failure (failure in the bonding interface), cohesive failure within enamel, cohesive failure within the RC, and mixed failure (combination of adhesive and cohesive failure).[17]

SEM Analysis of the Enamel–cement Interface

Sixteen blocks of enamel (8×4×2 mm) were cut and wet polished, as previously described. The blocks were randomly divided into two groups according to the RC used and subdivided into four groups according to the etching time (n=2). The blocks were treated as previously described. After etching, a layer of 1 mm of RC was applied on the treated enamel and light cured. Finally, the specimens were stored in deionized water for 24 hours at 37°C.

After this period of time, the specimens were longitudinally cut in half, and all of them were embedded in epoxy resin (EpoxiCure 2, Buehler Ltd, Lake Buff, IL, USA), using PVC tubes of 24-mm diameter and 10-mm higher. After that, the surfaces

Table 2: *Enamel Shear Bond Strength (SBS) Means and Standard Deviation (in MPa) of the Resin Cements (RC) Following Different Etching Times and Storage Conditions*

Etching time	MaxCem Elite		RelyX U200	
	24 hours	Thermocycling (TMC)	24 hours	Thermocycling (TMC)
Control	10.95 ± 3.9 Ba	0 Ca*	12.01 ± 2.3 Ba	0 Ba*
5 seconds	14.97 ± 4.1 Ab	10.55 ± 3.5 Bb*	22.58 ± 2.6 Aa	22.04 ± 3.0 Aa
10 seconds	16.76 ± 5.8 Aa	18.81 ± 3.3 Aa	19.30 ± 4.6 Aa	21.57 ± 3.5 Aa
20 seconds	10.63 ± 2.8 Bb	12.92 ± 4.6 Bb	22.61 ± 3.2 Aa	23.33 ± 5.5 Aa

Different letters indicate significant difference. Upper case letters compare etching times within the same cement and storage condition. Lower case letters compare RCs within the same etching time and storage condition. Asterisk indicates significant difference among storage conditions within the same cement and etching time. ($p<0.05$)

were polished with abrasive paper discs from 400 to 1200-grit, the final polishing was done with 1-μm diamond solution (MetaDi Supreme; Buehler Ltd, Lake Buff, IL, USA) and a polish-cloth disc. Specimens were demineralized in 20% phosphoric acid for 10 seconds and submitted to deproteinization by immersion in 10% NaOCl for 10 minutes. Specimens were dehydrated in ascending ethanol series (20%, 30%, 50%, 70%, 90%, and 100% for 20 minutes per step) and immersed in hexamethyldisilazane (Electron Microscope Sciences, Fort Washington, PA, USA) for 10 minutes. After chemical dehydration, specimens were sputter-coated with gold (Desk II, Denton Vacuum Inc, NJ, USA) and examined under SEM at 1500× magnification (JSM-5600LV; Japan Electronics Optics Laboratory, Tokyo, Japan).

CLSM Analysis of the Etching Pattern

Eight enamel samples (8×4×2 mm) were flattened and polished, as previously described, and divided in four groups (n=2) according to each etching time. After etching, the samples were rinsed for 1 minute with deionized water and then let in ethanol 100% for 5 minutes in an ultrasonic bath. Finally, the samples were stored overnight in 0.5 wt% of sodium fluorescein to reveal the etching pattern.

After immersion, the samples were assessed using CLSM (Leica TCS SP5, Leica, Mannheim, Germany) with an Argon laser of 488 nm with a band pass filter of 490-540 nm in oil immersion objective (63×, 1.4 NA). Three images of each specimen were obtained and qualitatively analyzed as the enamel etching pattern.

CLSM Analysis of the Enamel–RC Interface—Two samples of each subgroup were used for CLSM analysis and were prepared following the same protocol used for the SEM microscopy. For this analysis, 1 wt% fluorescent rhodamine B was added to the RCs, before the bonding process. After light-curing, the specimens were stored in deionized water for 24 hours at 37 °C.

The samples were then cut in slices of 1-mm thickness using a precision cutting machine (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) with a diamond saw to obtain three slides for each sample. The slices were cleaned (Ultrasonic Cleaner USC 750; Unique Group) in deionized water for 5 minutes to remove any debris. Then, the slices were examined using CLSM (Leica TCS SP5, Leica). A mixed helium–neon (He–Ne) gas laser was used as the light source at 543 nm wavelength. The images were recorded in fluorescent mode using an oil-immersed objective (63×, 1.4 NA). A representative area of each slice was scanned (11 sections of 2.5 μm each), and the adhesive cement tags were identified.

Statistical Analysis

Specimens that failed pretest or deboned spontaneously following the storage conditions were assumed as 0 MPa. Data of SBS were tested for normality and homogeneity of variances using Shapiro–Wilk and Levene Tests ($\alpha=0.05$), which indicated that the data set were normally distributed. Then, data were analyzed using three-way analysis of variance (ANOVA) and Tukey post hoc test ($\alpha=0.05$), using SPSS 23 Statistics software (IBM Inc., Chicago, IL, USA) for Mac operating system (OS).

RESULTS

Shear Bond Strength

The shear bond strength (SBS) values obtained in this study are presented in Table 2. Three-way ANOVA showed that the bond strength values were significantly influenced by the three factors: material, storage, and etching time ($p<0.001$); the double interactions material*etching time and storage*etching time were also significant ($p<0.001$). Tukey post hoc test revealed that MaxCem Elite showed the highest bond strength values when the

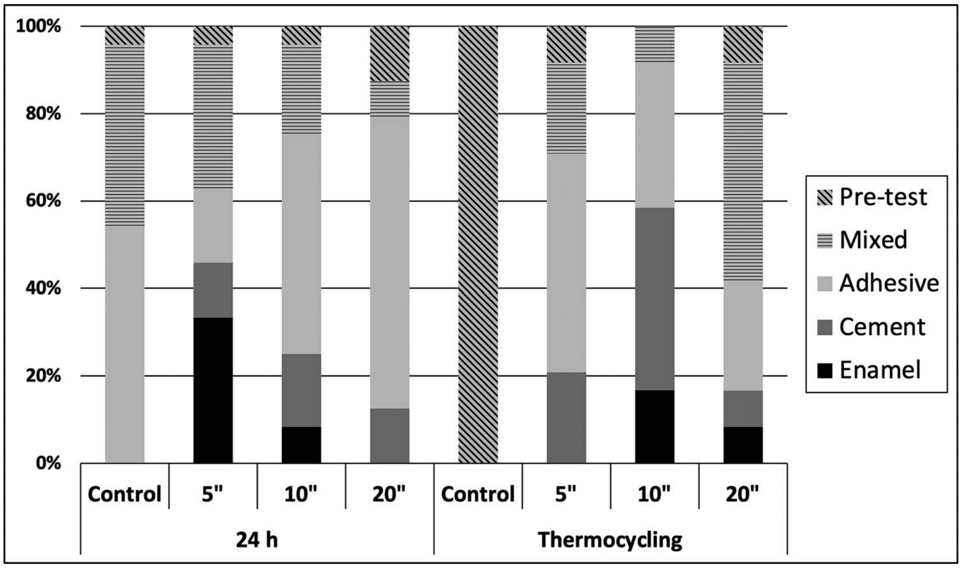

Figure 1. *Distribution of failure pattern mode for MaxCem Elite.*

enamel was etched for 10 seconds, and RelyX U200 showed the highest bond strength values when the enamel was etched regardless of the etching time used and the storage condition for both RCs. No statistically significant differences were found between storage conditions, with the exception of groups that were not treated with acid and MaxCem Elite etched for 5 seconds, in which a significant decrease in bond strength was observed after TMC compared with storage for 24 hours.

MaxCem Elite when bonded to enamel after 5-second etching time, dropped the SBS value significantly when thermocycled. However, when used with 10-second etching time, the SBS values increased even after TC. RelyX U200 SBS was not affected either by TMC or etching time. Both cements used without enamel etching were greatly affected by TMC, decreasing significantly the SBS values.

The failure mode percent distribution for the two RCs are summarized in Figures 1 and 2. Pretest failures were observed for all the groups with low incidence, with the exception of the control groups after TMC where all the specimens were lost. Specimens exhibiting pretest failure were counted as 0 MPa. Adhesive and mixed failures were observed in all groups for both the RCs, with the exception of the control groups that were submitted to TC. An increase in the cohesive failure types was observed in the groups treated with acid etching.

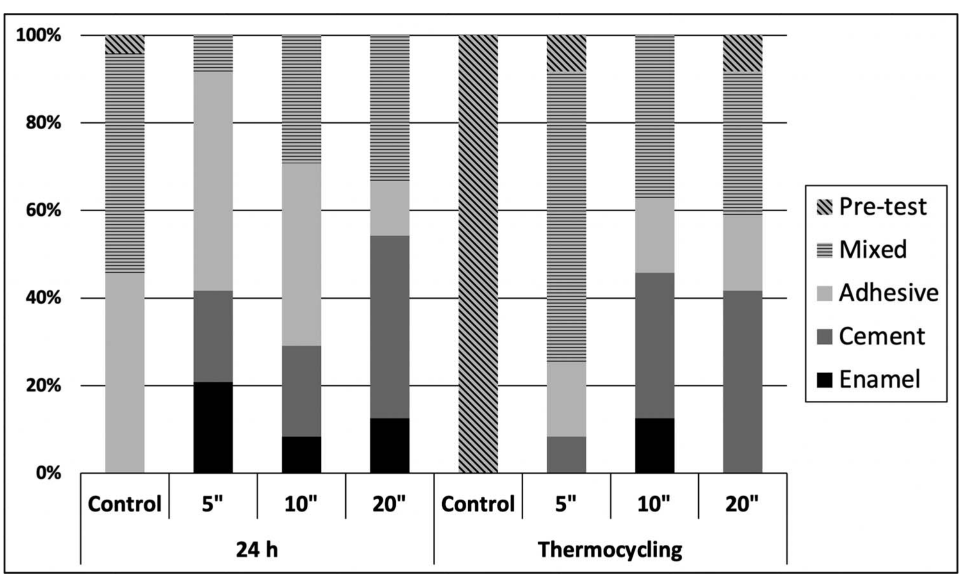

Figure 2. *Distribution of failure pattern mode for RelyX U200.*

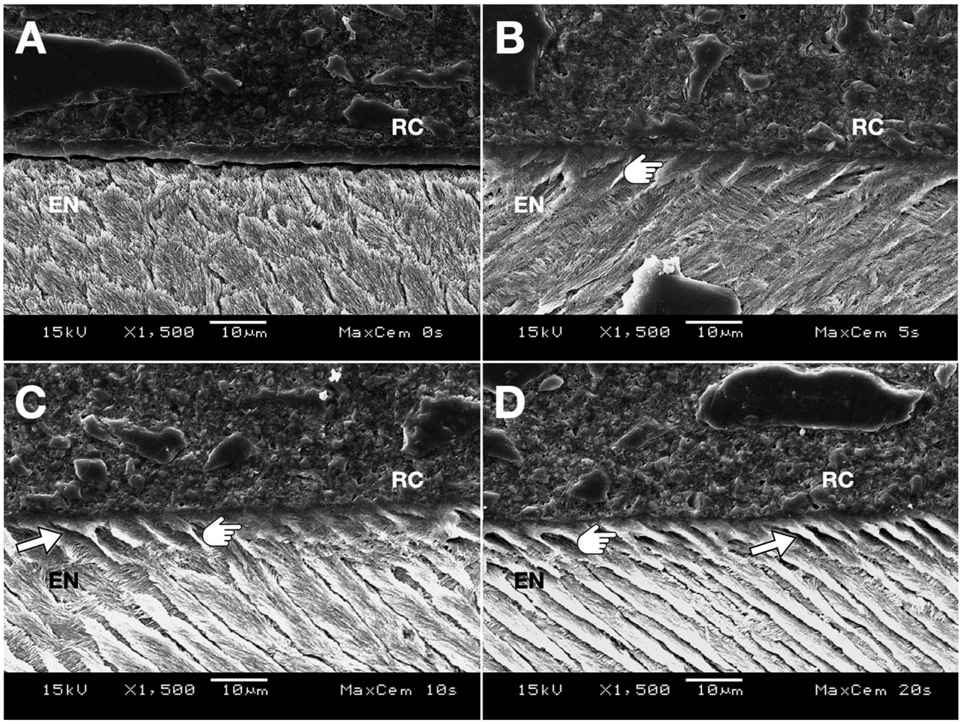

Figure 3. *Representative SEM micrographs of the RC–enamel interface with MaxCem Elite after 24 hours. Control group (A), PA for 5 seconds (B), PA for 10 seconds (C), and PA for 20 seconds (D). Resin tags are indicated with pointers. Empty spaces are indicated by white arrows. Abbreviations: RC, resin cement; EN, enamel. (1500× magnification).*

SEM Analysis of the Enamel–cement Interface

Representative images of the enamel–RC interfaces observed after 24 hours are illustrated in Figures 3 and 4. High magnification micrographs revealed that there was no interaction between the enamel and the RC, when it was treated following the manufacturer's instructions, with no enamel etching (Figure 3A and 4A). Figure 5 shows the etching acid patterns obtained on enamel, with no etching showing a smoother surface than 5- through 20-seconds enamel etching. The presence of gaps and

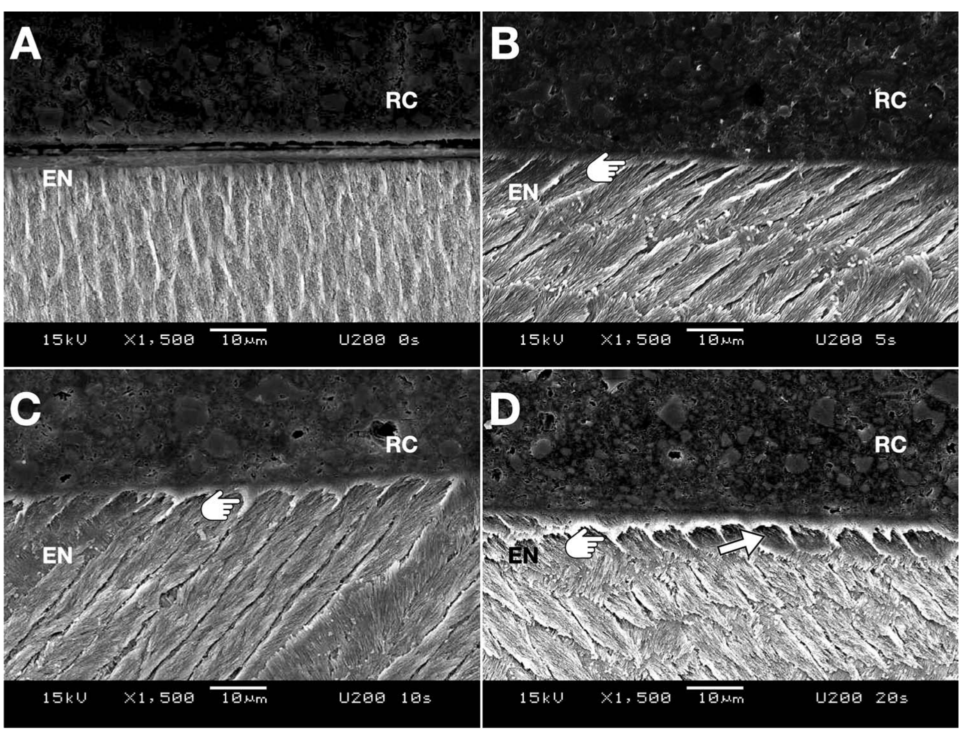

Figure 4. *Representative SEM micrographs of RCs–enamel interface with RelyX U200 after 24 hours. Control group (A), PA for 5 seconds (B), PA for 10 seconds (C), and PA for 20 seconds (D). Resin tags are indicated with pointers. Empty spaces are indicated by white arrows. Abbreviations: RC, resin cement; EN, enamel; PA, phosphoric acid. (1500× magnification).*

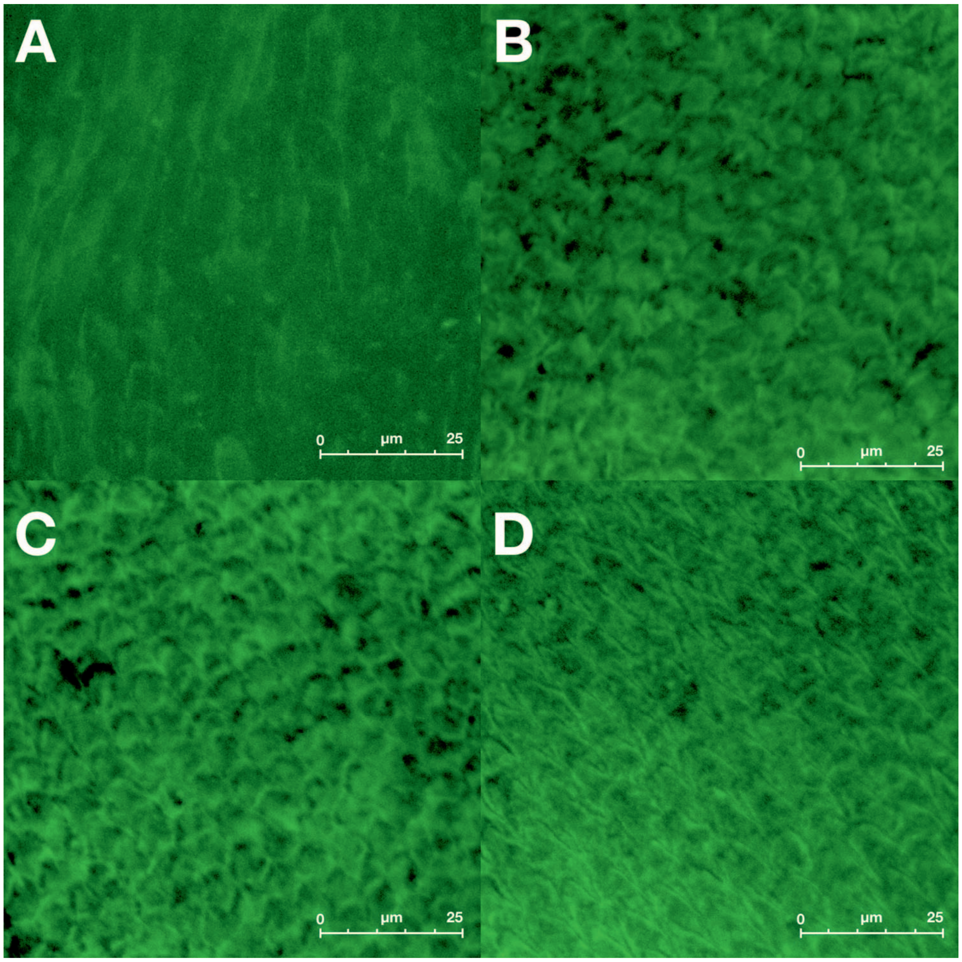

Figure 5. *Representative CLSM images of the enamel surface treated with phosphoric acid (37%) with different etching times (65× magnification). Control group (A), PA for 5 seconds (B), PA for 10 seconds (C), and PA for 20 seconds (D).*

the lack of continuity could have been the result of a very shallow interaction with the enamel (Figure 3A and 4A). When the enamel was etched with phosphoric acid, deeper resin penetration into enamel was observed with the presence of resin tags. The increase in the etching time resulted in deeper hybridization (Figures 3B-D and 4B-D). In the groups etched with phosphoric acid for 20 seconds, some spaces below the resin tags were observed (Figure 3D and 4D). Those spaces could probably be due to the lack of infiltration of the RC into the deeply demineralized enamel.

It is possible to observe differences in the filler size of the self-adhesive RCs. MaxCem Elite showed larger particles when compared with RelyX U200 (Figure 3 and 4).

CLSM Analysis

Representative images of the effects of the etching time with phosphoric acid on the enamel surface are shown in Figure 5. A smooth surface was observed in the untreated enamel (Figure 5A). When the enamel was etched with phosphoric acid, demineralized surfaces were observed (Figures 5B-D). A Type I etching pattern was observed in all the samples. The demineralization was deeper with the increase of the etching time.

Representative images of the enamel–RC interfaces after 24 hours are shown in Figures 6 and 7. No RC tags were detected in the untreated enamel (Figures 6A and 7A). When the enamel was etched with phosphoric acid, deeper resin penetration into enamel was observed, with the presence of resin tags. The increase in the etching time resulted in deeper infiltration with more resin tags (Figures 6B-D and 6B-D). Longer etching times generated greater and deeper tags.

DISCUSSION

We demonstrated the importance of time in enamel acid etching before the application of self-adhesive RC. The results show that enamel acid etching with phosphoric acid increased the bond strength of the self-adhesive RCs to the enamel. Additionally, it was

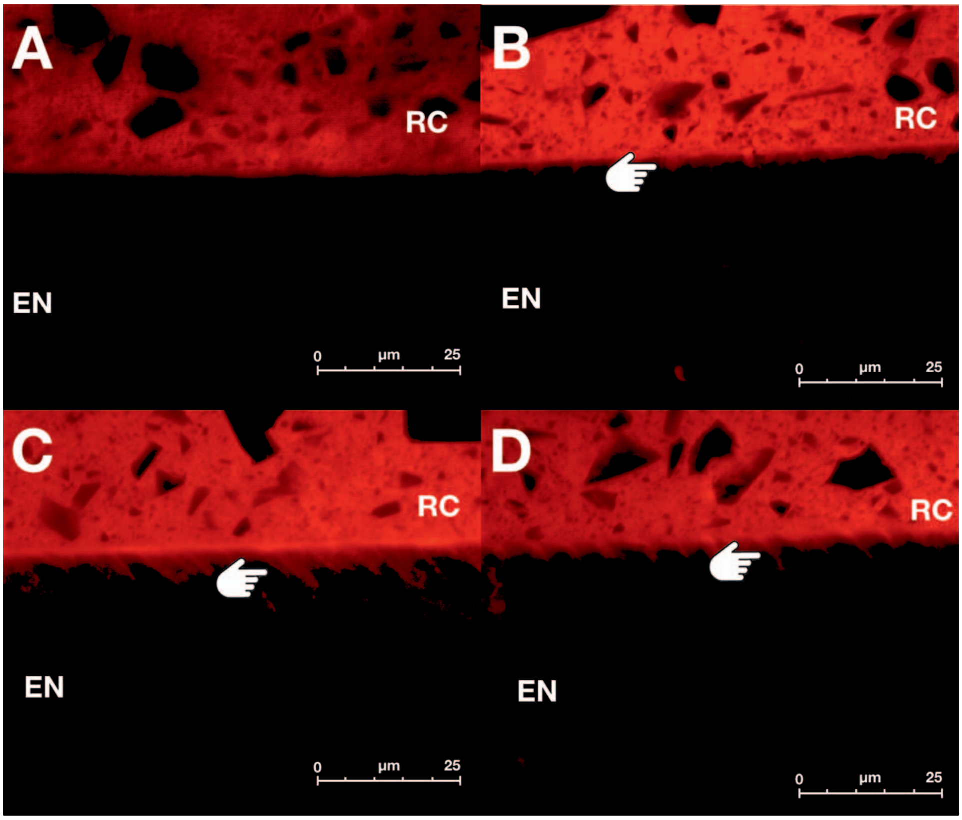

Figure 6. *Representative CLSM images of RCs–enamel interface with MaxCem Elite after 24 hours (65× magnification). Control group (A), PA for 5 seconds (B), PA for 10 seconds (C), and PA for 20 seconds (D). Resin tags are indicated with pointers.*

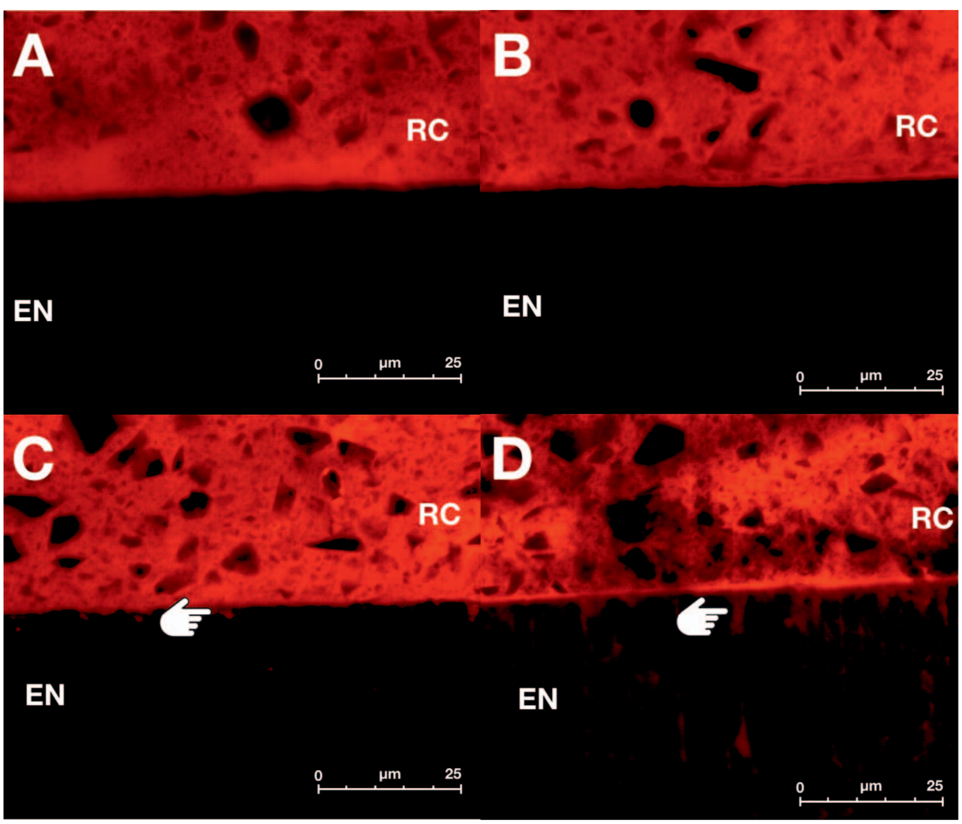

Figure 7. *Representative CLSM images of RCs–enamel interface with RelyX U200 after 24 hours (65× magnification). Control group (A), PA for 5 seconds (B), PA for 10 seconds (C), and PA for 20 seconds (D). Resin tags are indicated with pointers.*

observed that TMC negatively affected the bond strength when the enamel was not etched.

This study used bovine enamel as a substitute of human enamel, because they have similar morphology, mineral composition, and physical properties. Bovine teeth are easy to obtain and present some advantages of having the possibility to use large flat surfaces without variable composition.[18-20]

The first null hypothesis stating there would be no difference in the bond strength to enamel and in its morphology, regardless of the etching time, was rejected. The results indicate that the use of phosphoric acid increases the bond strength values, demonstrating that the etching ability of the self-adhesive RCs is not efficient to condition the enamel surface. Several studies have reported significantly higher bond strength values between enamel and self-adhesive RCs, when the enamel was pre-etched with phosphoric acid.[1,4,5,7,10] When phosphoric acid comes into contact with the enamel, part of the hydroxyapatite crystals on the surface is dissolved. As a result, the enamel increases its wettability, facilitating the infiltration of the resin matrix.[4,21,22] The wettability is essential to enable the materials to spread across the entire surface.[22] Additionally, acid etching removes most of the contaminants from the enamel surface, exposing hydroxyl groups and turning the enamel into a hydrophilic surface,[22,23] similar to the hydrophilicity of the acidic monomers in self-adhesive RCs.[24] However, as phosphoric acid increases the wettability, the interaction among the enamel, the RCs, and the water produced during polymerization is better.[24,25]

Another effect produced by the acid etching is the increase of the surface for adhesion.[22,26] Phosphoric acid attacks the hydroxyapatite crystals, eroding them; as a consequence, a retentive and porous structure on the enamel surface is created, leading to an increase in its bonding area.[17,26,27] Since the enamel is a homogeneous substrate, this treatment creates an ideal surface for bonding, making the adhesion durable and reliable.[28] Thus, the hydrophobic monomers of the RCs may be able to penetrate into the porous structure created on the enamel, improving the micromechanical retention and the bond strength values.[7,10]

The morphology of the enamel changes as a consequence of the etching treatment. Figure 5 shows the effects on the enamel surface; the longer the acid application, the greater the demineralization produced on the enamel. Higher bond strength to the enamel was observed following by the acid etching. However, the increase in the bond strength was not proportional to the increase in the etching time. In this way, shorter etching times were more effective.[17] Similar results were found by Barkmeier and others using self-etch adhesives, where extending the etching time did not improve the performance of the adhesives.[29] Another study showed that the increase in the etching time did significantly increase the surface energy of the enamel.[23] The bond strength to enamel is related to the capacity of the monomers to penetrate into the enamel structure, rather than the length of the resin tags.[10] The longer the acid application, the greater the demineralization produced on the enamel, hindering deeper infiltration by the RC.

The demineralization produced by shorter etching times might be ideal for the viscosity of the RCs tested. It is possible to observe RCs tags throughout the cement–enamel interface for the etched enamel.[7] However, when it was etched for 20 seconds (Figures 3D and 4D), some spaces beneath the tags were observed. The high viscosity of the cement compared to the adhesive could have hampered the penetration of the cement into the microporosities created by the acid.[8] This observation could explain, in part, why shorter etching times resulted in higher bond strength. Another factor that probably favored the results was the pressure applied during the cementation process.[1,5,8] In the present study, the test design did not permit the application of pressure during the placement of the cement. The application of light-pressure might have helped the penetration of the cement into enamel.[1,8]

Regarding the composition of the self-adhesive RCs used in this study, the differences between them might have influenced the results. MaxCem Elite has glycerol phosphate dimethacrylate (GPDM) as an acidic monomer for the enamel etching,[8] and RelyX U200 has multifunctional phosphoric acid methacrylates that react with the hydroxyapatite in an aqueous solution.[30] The filler distribution and particle diameter also varied among the cements. MaxCem Elite is a cement with high filler load (72.3% by weight) with a particle diameter of 3.52 μm, different from RelyX U200 that has lower filler load (62.2% by weight) with particle diameter of 2.47 μm.[31] These differences can explain, in part, why MaxCem Elite showed lower bond strength values. Its larger particle size and lower organic content might make its penetration into the enamel porosities difficult, since these factors influence the viscosity of the self-adhesive RCs. However, viscosity was not measured in this study, and no information in this respect was obtained.

The second null hypothesis stating that the TMC would not affect the bond strength to enamel was also rejected. The TMC negatively affected the SBS of both RCs when applied following the manufacturer's instructions. In those groups, all the specimens had spontaneous failure after TC. The bond strength between the self-adhesive RCs and the enamel was not sufficient to resist the TMC stress. The combination of two factors might explain the results: (i) a poor infiltration of the cement into the enamel structure and (ii) the differences in the thermal conductivity and the coefficient of thermal expansion between the enamel and the self-adhesive RCs.[13,14,32] The influence of the poor penetration of the cement into the enamel has been broadly explained above, and it could be the result of the weak etching capacity of the acidic monomers contained in the self-adhesive RC and their high viscosity.

The ISO/TS 11405 considers that a TMC regimen composed of 500 cycles in water between 5°C and 55°C, with a dwell time of at least 20 seconds is an appropriate artificial aging test.[33] However, many studies demonstrated that 500 cycles were not sufficient to assay long-term bonding durability and did not influence bond strength.[15,34,35] Thus, in the present study, samples were submitted to 5000 thermal cycles.[14]

The differences in the thermal conductivity and the coefficient of thermal expansion between the enamel and the self-adhesive RCs might result in gap formation as a consequence of crack propagation along the bonded interface.[14,15] The TMC promotes the spread of gaps in the interface enamel–self-adhesive RC, resulting in a higher number of pretest failures.[14] The water and oral fluids can infiltrate through these newly formed gaps, leading to adhesive failures.[32]

Based on our results, an enamel etching time of 10 seconds is recommended prior to using self-adhesive RC. Dental practitioners should be careful to avoid extending etching to dentin. Several studies show that the dentin etching prior to cementation with self-adhesive RCs results in reduced bond strength values.[4,5,10]

The presence of other factors that were not evaluated in this study, such as variations in the enamel structure (enamel prisms and orientations), mineral content of the enamel, and buffer capacity; different acid concentrations or alternative acids,[3] water sorption and cement solubility,[36] and different composition of the RCs could also affect the adhesion between the enamel and self-adhesive RCs. Thus, further studies must focus on evaluating the influence of those factors on the bond strength to enamel.

CONCLUSION

Enamel etching improves the bond strength of the two self-adhesive RCs tested. However, there is a limit on the etching time, with 10 seconds suggested as an adequate time before the use of RCs, even after ageing. TMC negatively affected the bond strength when the enamel was not etched.

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Regulatory Statement

This study was performed after approval by the local ethics committee (Protocol: 66522) of Faculty of Stomatology, Universidad Peruana Cayetano Heredia.

Conflict of Interest

The authors of the present study certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in the present article.

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