ORIGINAL RESEARCH

Does Etching of the Enamel with the Rubbing Technique Promote the Bond Strength of a Universal Adhesive System?

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Abstract

Aim: The aim of this *in vitro* research was to study the effect of etching by phosphoric acid with rubbing technique on the shear bond strength (SBS) of adhesive universal to enamel.

Materials and methods: Sixty extracted teeth were obtained. Three application methods (self-etch, etch-and-rinse, and etch-and-rinse with rubbing technique) were performed to bond the enamel surfaces by a universal adhesive. After 24 hours of immersion in water at 37°C, the specimens were prepared for the SBS test. Scanning electron microscopy was performed to observe the adhesive–enamel interfaces. Optical numeric microscope was used to observe the failure style. Statistical analyses were done with one-way analysis of variance test.

Results: Statistically significant higher bond strength values were observed for etch-and-rinse mode with rubbing technique (25.98 ± 5.70) MPa then for the etch-and-rinse without rubbing (22.07 ± 5.27) MPa and self-etch modes (9.96 ± 2.98) MPa.

Conclusion: Enamel etched by 37% phosphoric acid with rubbing technique for 20 seconds showed an increase in the SBS of the universal adhesive to enamel surfaces. The tags of the adhesive can be presented more efficiently by rubbing the acid before the bonding process, consequently, an optimal interface for the bonding.

Clinical significance: According to the results of this *in vitro* study, the selective enamel etching mode with rubbing technique is advisable when using the universal adhesive, as it significantly increased the bond strength of this adhesive to enamel surfaces. The clinician should etch the enamel using phosphoric acid with rubbing technique for 20 seconds to promote the bond strength of the universal adhesive system.

Keywords: Etching, Phosphoric acid, Rubbing technique, Universal adhesive.

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Introduction

Enamel structure is an essential portion of the tooth substance and is the hardest structure of the body.[1,2] In modern restorative dentistry, bonding to enamel has become a routine and predictable process.[3]

The etch-and-rinse adhesive techniques on enamel require two steps; the first step is an etching of the surface by an acid and the second step is to apply the bond agent and use a cure light to polymerize the resin, in situ, directly inside the treated surface.[4]

The current adhesive systems and methods are developed to provide an optimal adhesion with less complication in application protocol.[5]

On the contrary, the use of acidic monomers in self-etch adhesives eliminates the separated etching step.[6]

This method has proven to be easier to use and less sensitive.[7]

Numerous studies have shown that the etch-and-rinse mode have a crucial effect on the durability of universal adhesives on enamel; this fact was also observed on the previous generation of one-step adhesives.[8]

Several studies have shown that the acid-etching step is particularly critical on the formation of the mechanical bond between the resin and the enamel surface.[5,8,9] The most common acid used in dental restoration is phosphoric acid.[10]

One of the most relevant ways to characterize the commercial dental bonding product is to measure the bond strength.[11] Bond strengths are typically measured in shear or tensile, and most adhesion strength tests are performed on enamel or dentine grounded surfaces.[12]

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Many factors can have an impact on the adhesion strength: chemical composition of the adhesive, light-curing equipment, acid concentration, and differences in the experimental protocol.[13]

Ayar and Erdemir[14] compared the shear bond strength (SBS) of a universal adhesive used in etch-and-rinse mode or self-etch mode to the SBS of Er,Cr:YSGG laser-irradiated enamel bonded with the

same universal adhesive. They noted that acid etching may increase bonding strength values of universal adhesive to teeth surfaces.

Sai et al.[15] recommended using the etch-and-rinse mode in order to improve the bond strength of universal adhesive to the enamel surfaces, regardless of the application time.

The present study discusses the etching procedure, if should be done only by squirting the acid on an enamel surface and rinse it or should be accompanied with rubbing action.

This study aimed to analyze the SBS of two different etch-and-rinse protocols and a self-etch mode for universal adhesive to enamel surface. The initial assumption was that the three application techniques, including two etch-and-rinse modes and one self-etch technique, would not alter the bond strength of universal adhesive systems to enamel surfaces.

Materials and Methods

Specimen Preparation

In total, 30 recently extracted teeth, caries-free, human mandibular third molar, were extracted for orthodontic and ectopic eruption reasons, with patient informed consent [Ethics committee of Strasbourg University Hospital (protocol no. 2018-89)]. The intact enamel with no crack induced by extraction was the criteria for teeth selection. The teeth were stored at 4°C in 70% ethanol for 2 weeks. We divided the selected teeth into three groups (10 teeth each). After removing the root using a wire saw (Walter EBNER, Le Locle, Switzerland), a buccolingual sectioning of the teeth was used to prepare the enamel bonding surfaces. The samples were immersed in epoxy resin. The mesial and distal enamel were prepared with a P320-grit silicon carbide paper (Escil, Chassieu, France) to obtain plane surface equivalent to that obtained with dental diamond burs drill.[16] The first group (GI) consisted of 20 enamel surfaces bonded in self-etch mode using a universal adhesive (YBOND Universal, Yller Biomateriais, Pelotas\RS, Brazil). The second group (GII) consisted of 20 sections bonded in etch-and-rinse technique (etching by 37% phosphoric acid "ITENA Clinical, Paris, France" for 20 s then rinsing it with water for 30 s). The third group (GIII) consisted of twenty sections that were bonded in etch-and-rinse method with the rubbing technique (etching for 20 s with the rubbing technique using a micro-brush and then rinsed with water). The adhesive was used following the manufacturer's protocol (20 s brushing, 5 s air-drying and 20 s light curing). The adhesive was light cured for 20 seconds (Luxite Lampe LED, ITENA Clinical, Paris, France).

Shear Bond Strength

Seventeen specimens of each group were prepared for the SBS analysis. Resin composite build-ups were performed in silicone mold of 3 mm of diameter on the enamel plate surfaces using a resin composite Reflectys (ITENA Clinical, Paris, France) in four layers of 1.5 mm each. Each layer was photo-polymerized for 40 seconds following the manufacturer's instructions. The samples were immersed for 24 hours in distilled water at 37°C. For the SBS tests, specimens were attached to a testing machine (Instron 3345, "ISO/TS 11405 standard"). A constant crosshead speed of 0.5 mm/minute was used to submit the samples to a shear loading until fracture. By dividing the load at failure on the bonded surface area, we calculated the SBS (MPa).

Optical Numeric Microscope Observations

After the shear bond tests, the enamel surfaces were investigated under a digital microscope (KEYENCE; Osaka, Japan) and then analyzed using the VHX-5000 software to calculate the percentage of each area at 50× magnification to define the type of fracture. Three types of failure modes were categorized:[17]

Type I: Adhesive failure: <20% of the adhesive stayed on the enamel;

Type II: Cohesive failure: >80% of the adhesive stayed on the enamel; and

Type III: Mixed failure: certain area presented type II while other areas presented type I.

SEM Preparations and Observations for the Resin–Enamel Interface

After the bond process, the other three samples of each group were dehydrated in a graded series of ethanol solutions. The samples were sectioned sagittally with a diamond wire saw (Well Walter Ebner, Manheim, Germany). Then, the samples were polished using 1200, 2400, and 4000 P-grade abrasive paper. After that, the sectioned specimens were prepared with 20% citric acid for 5 minutes to eliminate the enamel in the sectioned surfaces and observe the internal surface of the adhesive layer in order to observe the resin tags. The specimens were sputter coated with gold-palladium alloys (20/80). Finally, the SEM was used for the observation of all coated samples.

Statistical Analysis

Samples that exhibited mixed or cohesive failures (type II and III) were kept out of the statistical analysis.[18,19] Data analyses were performed with Sigma Plot (release 11.2, Systat Software, San Jose, CA, USA). The one-way analysis of variance test was used to compare the SBS data (MPa) of the three techniques with statistical significance set at $\alpha = 0.05$.

Results

Shear Bond Strength Test

Table 1 shows the mean values of the SBS and standard deviations. The one-way analysis of variance showed a statistically significant difference between the three tested groups (p value < 0.05). Etch-and-rinse mode with rubbing technique (GIII) exhibited statistically significant greater SBS values than the other groups, GI (p value $<$ 0.001) and GII (p value $= 0.033$). Therefore, the initial assumption was rejected.

Optical numeric microscope observations were applied after the SBS tests in order to characterize the failure mode obtained after SBS tests. There was a predominance of adhesive failure (Fig. 1A) in all groups. However, mixed failure (Fig. 1B) and cohesive failure (Fig. 1C) increased in etch-and-rinse mode, regardless of the rubbing technique (Table 1).

SEM Observation of the Enamel–Bond Interface

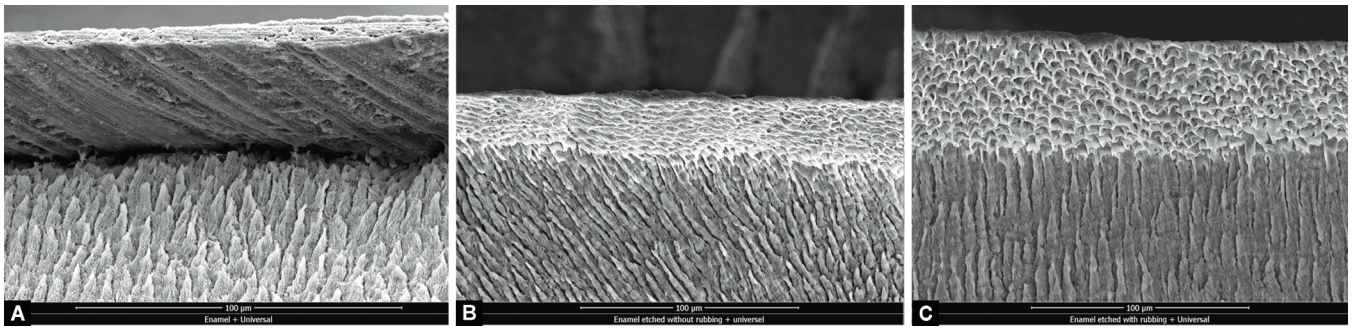
The enamel surface that was bonded in self-etch mode (GI) showed a smooth internal surface of the adhesive layer (Fig. 2A). The bonded

Table 1: Descriptive statistics of the shear bond strength of an enamel bond in adhesive fracture specimens as mean ± standard deviation

Experimental group	*n (total)*	*n (cohesive and mixed failures)*	*n (ad*)*	*Mean ± SD (MPa)*
Group I	17	0	17	9.96 ± 2.98
Group II	17	3	14	22.07 ± 5.27
Group III	17	3	14	25.98 ± 5.70

*Adhesive failure

Figs 1A to C: Representative photos of numeric optical microscope of: (A) Adhesive failure (×50 magnification); (B) Mixed interfacial failure (×50 magnification); (C) Cohesive failure in composite layers (×50 magnification)



Figs 2A to C: (A) scanning electron microscope (SEM) micrographs of the enamel-adhesive interface in self-etch mode reveal a smoothie internal surface of the universal adhesive layer with no tags penetration (×1,500 magnification); (B) SEM micrograph of the enamel-adhesive interface in etch-and-rinse mode showing a roughened surface due to the etching step of the enamel surface (×1,500 magnification); (C) SEM image of the enamel adhesive interface in etch-and-rinse mode with rubbing technique showing a larger and greater tags penetration depth on the internal surface of the universal adhesive layer (×1,500 magnification)

enamel in etch-and-rinse mode (GII) provided a roughened surface with more reliefs on the internal surface of the adhesive layer (Fig. 2B) than the group I. The enamel that was bonded in etch-and-rinse technique with rubbing (GIII) showed a greater and larger resin-tag depth (Fig. 2C) compared to the enamel etched without rubbing (Fig. 2B).

Discussion

The two etch-and-rinse techniques (with or without the rubbing technique) in this *in vitro* study improved the bonding efficacy of the universal adhesive to the enamel. Surface treatment significantly influences the bond strength of the universal adhesive system to enamel surfaces; accordingly, the initial assumption that there would be no difference between the three treatments must be rejected.

The resin-tags infiltration of universal adhesive was compared using different enamel bonding methods (self-etch, etch-and-rinse, and etch-and-rinse with rubbing technique).

In an effort to enhance the consistency of adhesive-resin systems, some studies recommend different clinical procedures, such as applying several adhesive layers, increased the time of light curing, and use of warm air for solvent evaporation.[20]

However, in our research, the rubbing technique is proposed during the etching procedure to increase the bonding quality.

It would seem that it is necessary to prepare the enamel surface by 37% phosphoric acid with rubbing technique for 20 seconds before the bonding processes of a universal adhesive. The clinician should use 37% phosphoric acid to clean a dental surface and prepare it for the bonding process.[21]

Jacobsen et al.[22] noted that using phosphoric acid can lead to an early formation of monocalcium phosphate monohydrate (MCPM); due to this fact, in the present study, the samples were rinsed for 30 seconds in order to eliminate precipitates.[23]

When the enamel surface is prepared by etch-and-rinse method with rubbing technique, the microporosity increased which led to increase the resin-tags depth of the adhesive layer into the etched enamel surface. Those interface bond-enamel of the specimens (GIII) revealed a larger resin-tags depth in situ inside the enamel surface when compared to the specimens etched without the rubbing technique (GII). Pouyanfar et al.[24] noted that the resin bond to enamel is durable and based on penetration of resin monomers into porosities of the enamel surface induced by acid etching and during formation of resin tags. According to the results of our present study, SEM images demonstrate that etching with rubbing technique for 20 seconds before the bonding processes would leave a great roughened enamel surface, which arise the presence of tags and provide the micromechanical interlocking of the adhesive material. Another study[9] said that the tensile bond

strengths of tests specimens prepared on enamel surfaces to which the acid was applied by a rubbing action for 60 seconds were not significantly different from those in which the acid was applied without rubbing technique. Kharouf et al.[25] who discussed the effect of etching the dentin with rubbing technique noted that the rubbing technique decreases the microtensile bond strength of the universal adhesive to dentine.

SEM images showed that bonding the universal adhesive without selective-etching revealed an enamel surface with no reliefs and no microporosity induced a smooth internal surface of the adhesive internal layer (GI). The micrograph (Fig. 2A) revealed a space between adhesive layer and enamel surface due to the SEM high pressure which induced a peeling off of the adhesive layer from the dental surface. The gap was observed only on the specimens that were bonded in self-etch mode (GI). These observations could be due to the fact that the bond strength in this group was less important when compared to the specimens prepared with etch-and-rinse mode.

In order to complete our evaluation and observation that were obtained by the SEM images SBS tests were performed for the three bonding methods. The SBS test is considered as a reference method.[26]

In self-etch mode (GI), the universal adhesive showed a significantly lower SBS values than the other two groups (GII and GIII) as described in Table 1. Enamel surface without reliefs lead to a nonpropagation of adhesive materials inside the etched enamel microstructure which decreases the micromechanical interlocking of the adhesive resin. Similar conclusions[5,8] were previously noted, where the selective enamel etching before the use of a universal adhesive is a pertinent strategy to obtain a better bonding. The effectiveness and long-term durability of bonding the universal adhesives to enamel are very important criteria. Therefore, etch-and-rinse mode has been proposed to realize durable enamel bonds in the case of using universal adhesives.[27]

Significantly higher mean values of SBS in etch-and-rinse with the rubbing technique mode were observed compared to the mean values of the enamel etched without the rubbing technique (one-way analysis of variance test, p value = 0.033). Some universal adhesives include acidic monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). One of the advantages of MDP is its capacity to create a hydrolytically stable ionic bond with the hydroxyapatite.[7]

Etching with rubbing technique creates grooves in the enamel surface, thus resulting in a larger connection surface between adhesive layer and hydroxyapatite.

The higher bond strength for the etch-and-rinse method with rubbing technique could be related to the augmentation of the micro-retentive tooth surface and the increase in microporosity obtained when the enamel was etched by phosphoric acid with rubbing technique, thus forming a more hydrolytically stable ionic bond with the calcium when compared to the tooth that was prepared by self-etch mode or by etch-and-rinse conventional mode.

In all debonded samples, the most common failure was adhesive failure type. From the finding of this research, the higher bond strength was correlated with greater cohesive or mixed fractures.

However, this *in vitro* research needs to study the degradation and the stability of the adhesive–enamel interface in the long-term. The main limitation of this present study was that long-term follow-up was not investigated; the samples were tested immediately after 24 hours. Emamieh et al.[28] highlighted that the bond strength of an adhesive resin might be affected by the "in water" storage period. The active application of a substance by rubbing has already been described for the application of adhesive resin to dentin and enamel.[29,30] The main interest is to have a better contact surface between the adhesive and the substrate and to allow the evaporation of solvents.[31] However, the consequence of the active application of phosphoric acid on the bond strength of universal adhesive to enamel has never been tested.

Other limitations of this study were that we have not evaluated the amount of reduction in the thickness of the enamel accompanying the rubbing technique and we have not measured the rubbing force applicate during the rubbing.

Conclusion

Etching by 37% phosphoric acid with rubbing technique for 20 seconds before the bonding process using a universal adhesive to enamel surfaces is recommended. This applied method creates numerous and larger reliefs on the enamel surface and shows a better tag depth which improves bond strength.

Clinical Significance

According to the results of this *in vitro* study, the selective enamel etching mode with rubbing technique is advisable when using the universal adhesive, as it significantly promoted the bond strength of this adhesive to enamel surfaces.

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