One-step Self-etch Adhesive Bonding to Pre-etched Primary and Permanent Enamel

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ABSTRACT

Purpose: The purpose of this study was to evaluate the effect of pre-etching enamel with phosphoric acid on the bond strength of a 1-step self-etch adhesive system in primary and permanent teeth.

Methods: The enamel of 30 primary molars and 30 permanent molars was ground with wet 600-grit silicon carbide paper, and specimens were randomly assigned according to bonding approach (with or without acid-etching). Half of the teeth received preliminary acid etching with phosphoric acid for 15 seconds before applying the self-etch adhesive system Adper Easy Bond. Polyethylene tubes with an internal diameter of 0.76 mm were placed on the bonded area and filled up with Filtek Z250 resin composite. The microshear bond testing was performed after 24 hours of storage in water at 37°C. Failure mode was evaluated using a stereomicroscope (400X). The microshear bond strength means were analyzed by 2-way analysis of variance and Tukey's post hoc test ($P<.05$).

Results: Bond strength to the enamel of primary teeth was significantly lower compared to permanent teeth. Higher microshear bond strength values were obtained when the enamel was pre-etched with phosphoric acid. For all groups, adhesive/mixed failure prevailed.

Conclusion: The preliminary etching of enamel increases the bonding effectiveness for the 1-step self-etch system, regardless of tooth type (primary or permanent).

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Bonding procedures to enamel play an important role in preventive and restorative dentistry, and the bonds obtained using phosphoric acid etching of enamel were the mainstream of adhesive dentistry for several decades. Self-etch adhesive systems are becoming increasingly more popular, and their market share is still growing, particularly because of their ease of use and fast application procedure.[1] Several studies have demonstrated, however, that their bonding effectiveness to enamel is poor compared to etch-and-rinse adhesive systems.[2-10]

Preliminary phosphoric acid etching of enamel for at least 15 seconds is a potential approach to use with self-etch adhesives to improve their performance,[11-15] especially in the presence of mild pH systems and unground enamel. Nevertheless, to the best of our knowledge, no previous study evaluated the role of preliminary etching in bonding to the enamel of primary teeth.

Since primary and permanent teeth present differences in enamel microstructure and composition[16] and these

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characteristics may interfere with etching pattern, studies are required to evaluate the bond strength of self-etch adhesive system to pre-etched enamel in both substrates.

Additionally, new self-etch adhesive systems have been recently developed. Adper Easy Bond (3M ESPE, Seefeld, Germany) is a typical 1-step self-etch adhesive that uses phosphoric acid ester methacrylates as functional monomers. There is limited information about the supplementary effect of an additional preceding etching step with phosphoric acid on bond strength to enamel when using this adhesive system.[15]

Therefore, the purpose of this study was to evaluate the effect of pre-etching enamel with phosphoric acid on the bond strength of a 1-step self-etch adhesive system in primary and permanent teeth. The research hypothesis tested was that preliminary etching significantly increases enamel bond strength, independent of tooth type.

METHODS

TEETH SELECTION AND PREPARATION

Sixty sound human teeth were selected, 30 primary second molars and 30 permanent third molars, after each patient's informed consent was obtained under protocol approved by the Research Ethics Committee of the School of Dentistry, University of São Paulo. The teeth were stored in 0.5% chloramine-T aqueous solution at 4ºC until use. The teeth were sectioned mesiodistally and most of the root structure was removed. The buccal and lingual crown sections were embedded in PVC rings with self-curing acrylic resin (JET Clássico, São Paulo, Brazil). The surfaces were ground under water with 180 grit silicon carbide (SiC) paper to obtain flat enamel surfaces, and further polished with 600 grit SiC paper for 60 seconds to create a standardized smear layer.

Specimens were assigned into 4 groups (n=15) according to tooth type (primary or permanent) and bonding approach (with or without preliminary etching).

BONDING PROCEDURES

Adper Easy Bond was applied, after either an etch-and-rinse approach involving phosphoric acid-etched enamel (35% etching agent; 3M ESPE, St. Paul, Minn.) applied for 15 seconds, followed by rinsing and vigorously drying with oil-water-free air, or a self-etch approach (no etching with phosphoric acid). One coat of the adhesive system with a fully saturated brush tip was applied for 20 seconds, gently air dried for 5 seconds, and light-cured for 10 seconds, according to the manufacturer instructions.

Polyethylene tubes (Micro-bore Tygon S-54-HL Medical Tubing, Saint-Gobain Performance Plastics, Akron, Ohio), with an internal diameter of 0.76 mm and a height of 1.0 mm, were placed on the bonded area and filled up with resin composite (Filtek Z250, 3M ESPE, St. Paul), covered with a matrix strip, gently pressed with a glass slide, and light-cured with a halogen light unit (Jetlite 4000 Plus, J. Morita USA Inc, Irvine, Calif.) with 600 mW/cm.[2]

After storage in distilled water at 37ºC for 24 hours, the polyethylene tubes were removed using a surgical blade, resulting in cylindrical specimens with an area of 0.45 mm². Specimens were examined under a stereomicroscope at 20X magnification; those with interfacial gaps, bubble inclusion, or other defects were excluded and replaced.

MICROSHEAR BOND STRENGTH (μSBS)

The specimens were attached to the universal testing machine (Kratos Industrial Equipment, Cotia, São Paulo, Brazil). A shear load was applied to the base of the resin cylinders with a thin steel wire (0.20 mm diameter) at a crosshead speed of 1.0 mm/minute until failure. Care was taken to keep the resin cylinder in line with the center of the load cell and the wire loop parallel to the load cell movement direction and bonding interface. The load at failure was recorded in Newtons (**N**). Bond strength was calculated in megapascals (**MPa**) by dividing the load at failure by the adhesive surface area (mm2).

FAILURE MODE

All debonded sticks were examined at 400X magnification using a stereomicroscope (HMV II, Shimadzu, Kyoto, Japan) to determine the failure mode: adhesive/mixed (failure at resin/enamel interface or mixed with cohesive fracture of the neighboring substrate) or cohesive (failure exclusively within enamel or resin composite).

STATISTICAL ANALYSIS

The experimental unit in the current study was the tooth. Thus, the means of μSBS values of all specimens from the same tooth were averaged for statistical analysis.

Table 1. Microshear Bond Strength Means (MPa) and Standard Deviations (±SD) for All Experimental Groups*

Bonding approach	Primary Teeth	Permanent Teeth
Without acid-etching	19.9±6.4[a]	23.2±3.5[b]
With acid-etching	26.9±5.0[b]	29.4±4.4[c]

** Different superscript letters indicate statistically significant difference between "main factor" tooth type (rows) and "main factor" bonding approach (columns; P<.05).*

Table 2. Microshear Bond Strength Means (Mpa) and Standard Deviations (±SD) of the Main Factors and P-values

Tooth type	Mean (±SD)	*P*-value
Primary	23.4±8.6	<.03
Permanent	26.3±6.6	
Bonding approach		
Without acid-etching	21.6±6.3	<.001
With acid-etching	28.2±7.7	

Normal distribution of data was assumed after applying the Kolmogorov-Smirnov test. The μSBS means were analyzed with 2-way analysis of variance (**ANOVA**), using a factorial design with the tooth type and bonding approach as variables. Tukey's HSD multiple comparisons statistical test at a 0.05 significance level was used. Failure mode was only qualitatively evaluated.

RESULTS

Microshear bond strength means (MPa) and standard deviations for all experimental groups are displayed in Tables 1 and 2. ANOVA revealed that the main factors, tooth type ($P<.03$) and bonding approach ($P<.001$), were statistically significant. There was an increase in bond strength for both permanent and primary substrates after pre-etching the surface.

The bond strength of enamel to primary teeth was significantly lower compared to permanent teeth. Higher μSBS values were obtained when enamel was pre-etched with phosphoric acid.

Data regarding the failure mode of debonded specimens are summarized in the Figure 1. For all groups, adhesive/mixed failure prevailed. No cohesive failure in enamel was observed.

DISCUSSION

The results showed that pre-etching enamel with phosphoric acid increases the bond strength of a 1-step self-etch adhesive system for primary and permanent teeth (ie, the hypothesis was supported). Additionally, it was found that the bond strength values of primary enamel were lower compared to permanent enamel.

Since enamel bonding is mainly based on micromechanical interlocking of a low-viscosity resin into micro-porosities, the extent and depth of the etching pattern depends on the acidity of the conditioner agent.[17] These characteristics should influence the bonding performance of the adhesive systems. Easy Bond is a 1-step adhesive system with a relatively high pH (2.4); thus, it can be categorized as an "ultra-mild" self-etch adhesive. The shallower pattern of the demineralization associated with self-etching primers may be due to the difficulty in the penetration of the primer into enamel or due to some mineral precipitation on the enamel, which would modify the depth of demineralization.[18,19] These aspects might jeopardize bonding using self-etching systems, resulting in lower bond strength values compared to etch-and-rinse adhesives.[2-10]

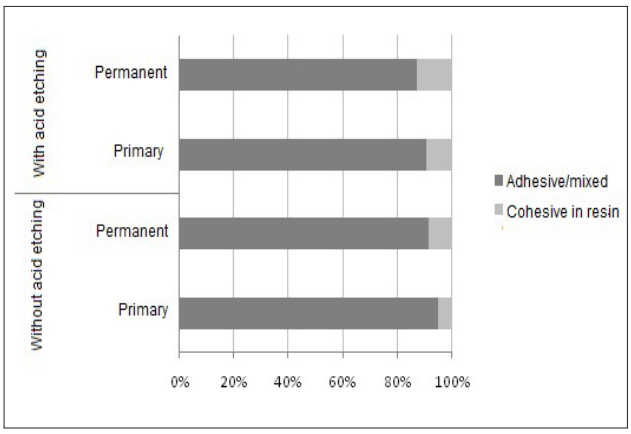

Figure 1. Failure mode distribution (%) for experimental group.

For this reason, the use of preliminary acid-etching has been proposed to deepen the enamel demineralization. Previous studies investigated the bond strength of self-etch systems with or without the use of phosphoric acid etching to permanent enamel, showing that pre-etching enamel enhanced the bond strength of self-etch adhesive systems,[19-21] which was also observed on primary teeth in the current study. Nevertheless, only one previous study has used the adhesive Adper Easy Bond. Taschner et al.[15] analyzed the effect of preliminary etching of enamel before applying Adper Easy Bond and iBond Self-etch adhesives and found that both showed higher bond strength values when permanent enamel was pre-acid-etched.

This approach would be interesting for cases in which the use of self-etch adhesives is suitable, such as in deep dentin carious lesions or when a layer of partially demineralized dentin is maintained over the pulpal floor, particularly in primary teeth.[22] In these situations, bond strength to enamel around carious lesions may be enhanced using preliminary etch without modifying the bond system for both substrates. Fewer enamel margin defects were observed when selective etching of enamel was employed in a clinical study[23] and in a laboratory study[21] where thermo-mechanical loading was used on restorations. Since high bond strength to enamel is critical for a good margin and seal of the restorations, applying the additional etching step should be considered in clinical practice in case of restorations that rely mainly on enamel bonding.

The evaluation of failure mode showed a predominance of adhesive/mixed failures, regardless of the bonding approach or tooth type. It is a characteristic of the μSBS test that few cohesive fractures are verified, as previously found in several studies.[24-26]

Despite the comparison of bonding performance in primary and permanent enamel, the results of this study are very important because this is one of the first known studies regarding μSBS in primary teeth, and questions about the adhesion in this substrate still persist. The pioneer investigation, conducted by Shimada et al.,[27] compared the bond strength of 2 adhesive systems to primary and permanent enamel. Bonding with self-etching primer system or etch-and-rinse adhesive to primary enamel was similar to permanent enamel. By contrast, scanning electron microscopy observations indicated that primary enamel was more reactive to acid conditioning than permanent enamel.

The aprismatic enamel layer of primary teeth is more pronounced than that observed in permanent teeth,[28] and this characteristic can interfere with the acid etching pattern. In the current study, however, this layer was removed when we polished the surface to obtain a flat enamel surface. Clinically, the primary enamel usually has its surface ground down (due to the use of a bur) before the restorative procedure. Therefore, the results obtained are more relatable to clinical conditions.

The lower bond strength values to primary enamel observed in this study can be attributed to a bigger deficiency in resin penetration into etched primary enamel, due to the smear layer formed when the surface was ground. Moreover, differences in chemical composition and micromorphology can be contributing factors.[16] Thus, the results of bond strength testing for permanent teeth cannot be extrapolated to primary teeth. Long-term studies are required to provide conclusive evidence if the preliminary etching with phosphoric acid is able to enhance the bonding of 1-step self-etch systems. This is especially true of primary enamel, since the use of an additional clinical step increases chair time, which is critical for pediatric patients' treatment. Furthermore, Adper Easy Bond has a similar composition to etch-and-rinse system Adper Single Bond 2, except for the presence of phosphoric acid ester methacrylates as functional monomers. Hydrophilic copolymers of the polyalkenoic acid in this composition are believed to form calcium-polyalkenoic acid-base complexes, contributing to immediate stability as well as longevity-bonded interfaces over time.[29] The possible role of chemical bonding for 1-step adhesives should be further investigated.

It is important to remember that this is an in vitro study which cannot be directly extrapolated to an in vivo situation. Only one bonding approach was evaluated (microshear bond test), and the results could be different if other tests were performed (eg, microtensile bond strength test, microleakage, nanoleakage, etc).

CONCLUSION

This study showed that the preliminary etching of enamel increases bonding effectiveness for a 1-step self-etch system, regardless of whether the tooth is primary or permanent.

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