Tensile Bond Strength To Primary Dentin After Different Etching Times

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ABSTRACT

Purpose: The purpose of this study was to assess in vitro the effect of etching time on tensile bond strength to primary dentin.
Methods: Forty crowns of sound primary molars were embedded in acrylic resin (N=40). Dentin was exposed in the buccal surfaces, and the specimens were randomly assigned into 4 groups (N=10), according to the etching time with 35% phosphoric acid gel: G1=7 seconds; G2=10 seconds; G3=15 seconds; G4=20 seconds. A 3-mm diameter bonding site was demarcated, the adhesive system was applied, and resin composite cones were built up. After 24 hours, tensile bond strength tests were performed. Data were submitted to statistical analysis using one-way analysis of variance and the Tukey test.
Results: Means (±SD) were (MPa): (1) G1=10.04 (±3.30); (2) G2=7.78 (±4,49); (3) G3=8.78 (±2.96); (4) G4=7.60 (±1.91).
Conclusions: Although no statistically significant difference among the times used was found, the 7-second etching time promoted the highest bond strength mean and can be used in primary dentin. Additionally, this lower etching time reduces chair time, which is a significant benefit when treating children. (J Dent Child 2007;74:113-7)

KEYWORDS: PRIMARY DENTIN, ACIDIC CONDITIONERS, TENSILE TEST, COMPOSITE

The advent of acid etching and further introduction of adhesive restorative systems have revolutionized the dental practice, allowing actual dental treatments to be less invasive and more esthetic. Despite major advances in adhesive dentistry, bonding to dentin and completely sealing the exposed dentinal surfaces remains problematic due of the highly hydrated and complex nature of this tissue.[1]

The differences in micromechanical and histological characteristics between primary and permanent dentin might interfere with the adhesion mechanism.[2] Greater tubular density and diameter have been reported for primary teeth,[3] resulting in a reduced area of intertubular dentin available for bonding.[1] Chemically, the dentin of primary teeth seems to be more reactive to acidic conditioners,[4] which could be explained by the reduced degree of mineralization observed for primary hard dental tissues.[2] Different etching times with acidic conditioners result in different depths of demineralization. Excessive etching of dentin may produce weak bonding due to the possibility that the resin monomers may not be able to penetrate into the open dentin tubules and diffuse across the hydrated demineralized collagen network as deeply as the etchant agent. This leaves behind nonimpregnated or poorly infiltrated, unsupported areas at the base of the hybrid layer, which are more prone to microleakage and nanoleakage, collagen hydrolysis, and degradation of the interface over time.[5,6] For this reason, it has been suggested that a reduction in etching time may produce more functional hybrid layers in primary teeth,[4,7] but no conclusion was made regarding the implications of the observed differences relating to bond strengths and subsequent clinical implications.

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Thus, the purpose of this *in vitro* investigation was to assess the effect of etching time on the tensile bond strength to primary dentin.

METHODS

Initially, this study was approved by the Ethics Committee of the School of Dentistry of Ribeirão Preto, University of São Paulo, São Paulo, Brazil. Sound human primary molars exfoliated or extracted within a 6-month period were cleaned with a scaler and water/pumice slurry in dental prophylactic cups and examined under a X20 magnifier to discard those with structural defects. Forty teeth were selected for the study and stored in a 0.9% saline solution with 0.4% sodium azide at 4°C.

Prior to use, the teeth were washed in running water to eliminate residues of storage solution. When necessary, roots were sectioned 2 mm below the cementoenamel junction. Crowns were embedded in chemically activated polyester resin into polyvinyl chloride (PVC) rings (2.1-cm diameter and 1.1-cm height) in such a way that their buccal surfaces were faced up (Figure 1). After resin polymerization, the rings were discarded and the buccal surfaces of teeth were ground with water-cooled no. 180- to no. 400-grit silicon carbide (SiC) papers (Buehler Ltd, Lake Bluff, Ill) on a polishing machine (Politriz DP-9U2, Struers, A/S, Copenhagen, Denmark) to remove the overlying enamel and expose the flat dentin surface (Figure 2). To warrant the complete removal of enamel, the ground surfaces were viewed under a X20 magnifier. Additional wet grinding with no. 600-grit SiC paper was done for 30 seconds to produce a standard smear layer. A bonding site was demarcated by attaching a piece of insulating tape with a 3-mm diameter central hole to each dentin surface (Figure 3). Bonding site delimitation had a double aim to define a fixed test surface area and to warrant that the resin composite cones could be further adhered precisely to treated dentin surface, thus avoiding accidental adhesion to the surrounding enamel.

The specimens were randomly assigned to 4 groups (N=10), according to the etching time: (1) G1=7 seconds; (2) G2=10 seconds; (3) G3=15 seconds; and (4) G4=20 seconds. The surface etching was performed with a 35% phosphoric acid gel (Scotchbond Etchant, 3M/ESPE, St. Paul, Minn) during the time corresponding to each group. Time of acid application was strictly controlled using a chronometer. The surfaces were immediately rinsed thoroughly, and excess water was removed with absorbent paper—leaving a lightly moist dentin surface. Two consecutive layers of a 1-bottle adhesive agent (Single Bond, 3M/ESPE) were applied, slightly thinned with a mild, oil-free air stream and light-cured for 20 seconds with a visible light curing unit with a 450 mW/cm2 output (XL 3000, 3M/ESPE), as measured by a radiometer

FIGURES 1–8. SCHEMATIC ILLUSTRATION OF SPECIMEN PREPARATION AND TENSILE BOND STRENGTH TESTING.

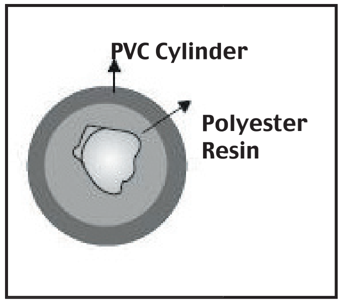

Fig. 1. Hemi-crown embedded in polyester resin.

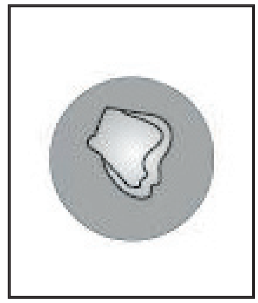

Fig. 2. Dentin surface exposed after grinding.

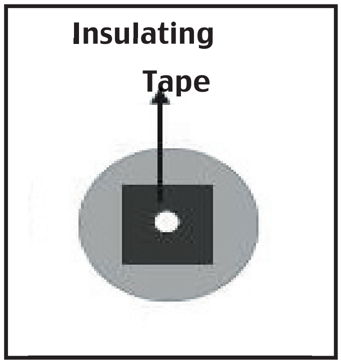

Fig. 3. A 3-mm-diameter dentin bonding site demarcated with insulating tape.

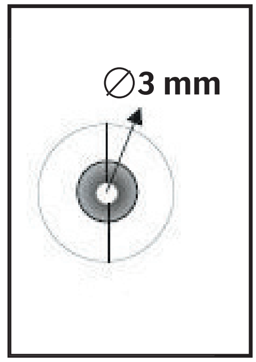

Fig. 4. Split-bisected polytetrafluoroe thylene jig.

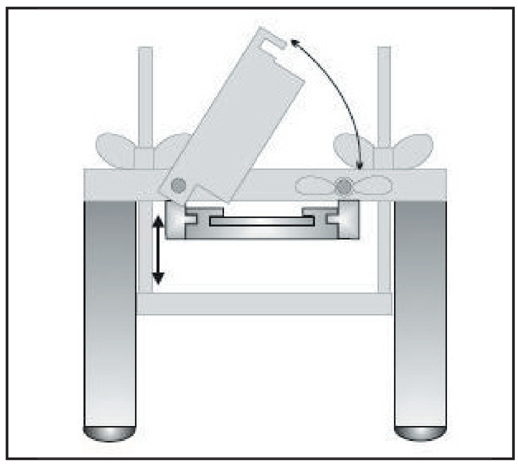

Fig. 5. Metallic clamping device.

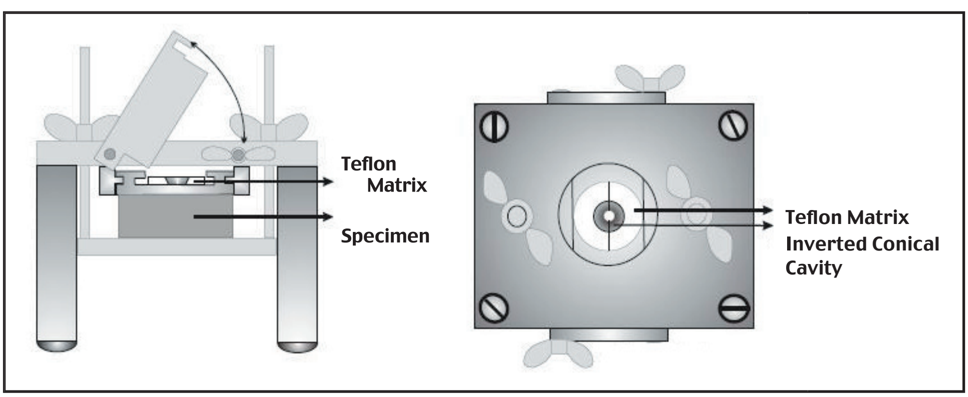

Figure 6. Resin/tooth block and polytetrafluoroethylene jig positioned in the clamping device (lateral and upper views).

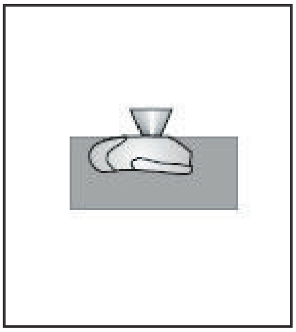

Fig.7. Inverted, truncated resin composite cone adhered to the demarcated dentin site.

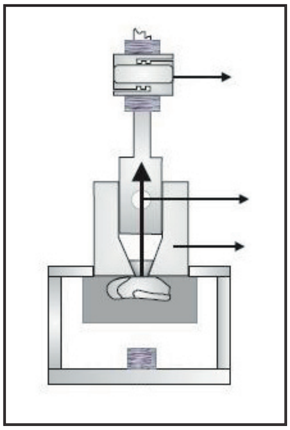

Fig. 8. Apparatus used for tensile bond testing.

(Demetron Research Corp, Danbury, Conn).

The adhesive system was applied onto the delimited dentin surface with disposable brush tips (Microbrush Corporation, Grafton, Wis) to avoid excess and pooling of adhesive along the edges of the insulating tape, which could compromise the distribution of tensions during the test and, hence, the results' validity.

Once the bonding protocol was completed, the specimens were individually fixed in a metallic clamping device (developed at the Houston Biomaterials Research Center, University of Texas Dental Branch, Houston, TX and manufactured at the Precision Workshop of the School of Dentistry of Ribeirão Preto, University of São Paulo, SP, Brazil) that allowed the authors to keep the dentin surface parallel to a flat base (Figure 5). A split-bisected polytetrafluoroethylene jig (Figure 4) was positioned on the tooth/resin block, thus providing an inverted conical cavity with the smaller diameter coincident with the demarcated bonding site (3 mm in diameter; Figure 6). A hybrid light-cured composite resin (Filtek Z250, 3M/ESPE) was inserted into the jig following the incremental technique, with each increment photopolymerized for 40 seconds. As the cavity was completely filled, the specimen was removed from the clamping device and the jig was opened and separated, leaving adhered to the delimited dentin site an inverted, truncated composite resin cone with the same dimensions as that of the jig (4 mm in height and 6 mm diameter tapering to a 3-mm diameter; Figure 7).

After a 24-hour storage time in distilled water at 37°C, the specimens were tested for bond strength using a universal testing machine (Mod. MEM 2000, EMIC Ltda, São José dos Pinhais, PR, Brazil) running at a crosshead speed of 0.5 mm/minute with a 50 kgf load cell (Figure 8). Bond strength values were recorded in kgf/cm and converted into MPa, and the means of each group were calculated. Since a normal and homogeneous distribution was observed, data were submitted to 1-way analysis of variance parametric test using a factorial design with the same etching time as the variable. Individual comparisons were done using Tukey's statistical test at a 0.05 significance level.

Fractured specimens were examined with a X40 stereomicroscope to assess the failure modes (adhesive, cohesive, or mixed). A single examiner who was blinded to the groups to which the specimens belonged did all examinations.

RESULTS

Tensile bond strength means for dentin and standard deviations are displayed in Table 1. No statistically significant difference was observed between the groups.

The analysis of bonding sites after the tensile strength test revealed that mixed failures mostly occurred for the 7-second etching time (100%), while the 10-second and 20-second groups showed 40% and the 15-second group showed 20% of this failure pattern. The adhesive failures occurred predominantly in group 4 (60%), and groups 2 and 3 showed, respectively, 30% and 40% of this type of failure. The 10-second and 15-second etching times showed 30% and 40% of cohesive failures, respectively.

Table 1. Tensile Bond Strength Means (MPa) and Standard Deviations (±SD) of the Different Groups, According to the Correspondent Etching Time

Groups	1 (7 seconds)	2 (10 seconds)	3 (15 seconds)	4 (20 seconds)
Mean±(SD)	10.04±3.30a	7.78±4.49a	8.78±2.96a	7.60±1.91a

** Same letter indicate statistical similarity.*

DISCUSSION

In vitro studies have reported lower bond strength values of distinct adhesive systems to primary dentin compared to permanent dentin.[8-11] Regardless of eventual compositional and morphological peculiarities and differences in bond strengths, the same protocol is being recommended for bonding to primary and permanent teeth. This fact may lead to the establishment of a differentiated resin-dentin interface in the 2 dentitions, with potential deleterious consequences for the dentin bonding system's ultimate performance.

In most currently available adhesive systems, the dentin bonding protocol relies on acid etching of teeth by a strong inorganic acid, which opens and widens dentin tubules' entrances, increases intratubular dentin exposure/permeability, and demineralizes intertubular dentin. The infiltration and further polymerization of a hydrophilic monomer capable of interweaving with the exposed collagen network in the dentin matrix results in a resin-dentin interdiffusion zone or hybrid layer, which is generally accepted as the major factor to achieve optimal dentin bonding.[1,2,13,5] Total etching with 30% to 40% phosphoric acid is one of the steps on the bonding protocol of several contemporary adhesive systems, and an etching time of 15 seconds is deemed ideal for conditioning permanent dentin.[14] Permanent dentin is more resistant to demineralization by phosphoric acid etching than primary dentin.[4,7] Studies with different types of adhesive systems have shown that bond strength to primary dentin is generally lower than that to permanent dentin.[15,10,16] Therefore, it has been suggested that shortening of the acid-etching time would yield the formation of a more functional hybrid layer in primary teeth.[4,7]

Moreover, primary dentin has lower hardness and mineral content than permanent tooth dentin.[2] The peritubular dentin of primary teeth is approximately 2 to 5 times thicker than that of permanent teeth and, hence, the dentine tubules of primary teeth have a smaller diameter.[17] Sumikawa et al (1999)[3] reported that primary teeth have greater tubular numerical density and, hence, lesser intertubular dentin area is available, which may interfere with the establishment of a high-quality adhesion. Since the penetration of acids occurs primarily along the tubule, it could be possible that a large number with a large diameter could result in a deeper penetration of the acidic conditioner and, therefore, a stronger demineralization. Additionally, due to the reduced mineral content of primary dentin compared to permanent dentin, a different effect of acid etching on primary substrate has also been suggested as a possible explanation.

Some authors found a linear relationship between etching time and hybrid layer thickness.[18,19] When acid is applied to dentin, mineral components of this structure are partially removed, exposing the collagen mesh work.[20] A scanning electron microscopy study has demonstrated that hybrid layers formed in primary dentin were nearly 25% to 30% thicker than that of permanent dentin using identical acid etching times.[4] It has been shown, however, that hybrid layer thickness is not directly related to tensile bond strength.[14,18,,21,22]

Despite increasing the thickness of hybrid layers, a prolonged acid etching time tends to reduce resin-dentin bond strength values.[14,21] Excessive etching of dentin might reduce bond strengths by the collapse of collagen fibrils and precipitation of calcium phosphate crystals, thus preventing monomers from fully impregnating the demineralized area.[14] Moreover, adhesive monomers *per se* are not able to completely infiltrate the demineralized dentin.[23,24] The degree of resin infiltration into the demineralized dentin decreases gradually towards the bottom of hybrid layer, resulting in the formation of a demineralized and unprotected dentin zone.[25,26] The prolonging of the phosphoric acid-etching time favors the occurrence of this zone[21] resulting in nanoleakage along the bottom of hybrid layers due to the presence of porosities within the demineralized dentin.[23]

Dentin has high water content and, thus, most modern adhesive systems possess hydrophilic characteristics and require a moist dentin substrate for high-quality bonding. It has been well documented that the bond strength of overdried dentin is lower than that of moist dentin. This is because the collagen fiber mesh collapses if dentin is excessively air-dried and the resin monomers cannot optimally penetrate to form the hybrid zone.[27,28]

The adhesive system used in the present study (Single Bond), contains water as a cosolvent, which does not allow collagen saturation by hydroxyethyl methacrylate (HEMA); molecules due to its lower volatility compared to acetone-based adhesive systems. Therefore, if the substrate is excessively moist, the water present in the adhesive system may lead to water saturation and decrease the bonding efficiency.[29] On the other hand, Single Bond also contains HEMA and polyalkenoic acid, which enhance adhesion to moist dentin. Care was taken to avoid excessive dentin moisture interfering with the results.

Sardella et al (2005)[22] obtained lower microtensile bond strengths when Single Bond was applied after a 15-second etching period in primary dentin, while shortening of the acid etching time (7 seconds) improved bond strength. The authors showed that the reduction of acid etching time of primary dentin was beneficial to bond strength and could be more adequate to produce resin-dentin bonds more resistant to degradation. Another study[18] did not find statistically significant differences when examining shear bond strength in primary dentin, with etching times varying from 15 to 120 seconds. In the present study, there was no statistically significant difference in tensile bond strength means when Single Bond was applied after acid etching times of 7, 10, 15, and 20 seconds. Bolanos-Carmona et al (2006),[19] using a different 1-bottle adhesive system (Excite) in primary dentin, reported that 5 seconds of etching time produced a visible demineralized layer. Under SEM examination, these presented funneling of dentin tubule entrances and some resin tags, but microtensile bond strength was lower in specimens etched for 5 seconds than those etched for 15 or 30 seconds. The differences in the methodology and materials may explain the variations in results when these studies are compared.

The current in vitro study assessed the effect of acid etching time on tensile bond strength to primary dentin. Nevertheless, it is important to highlight that the lack of studies testing the same methodology and materials in this substrate was a hindrance to stating a reliable comparison between the outcomes of the conducted research and the available data.

CONCLUSIONS

The dentin-etching step is fundamental for effective bonding, and times for application should be carefully controlled. In pediatric dentistry, there is a great need to reduce the time for a procedure without compromising the quality of the work. Based this study's findings and within the limitations of an in vitro investigation, it may be concluded that—although no statistically significant difference among the times used was found—the 7-second etching time promoted the highest bond strength mean. Therefore, considering that over-etching dentin may make the adhesive monomer incapable of completely infiltrating the demineralized zone, thus decreasing bond strength, a 7-second etching time can be used in primary dentin. Additionally, this lower etching time reduces chair time, which is a significant benefit when treating children.

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