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Research

Enamel bond strength comparison of self-limiting and traditional etchant systems

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ABSTRACT

Objective: The objective of this experiment was to compare the shear bond strengths of a self-limiting etchant to a standard etchant system at 15-, 30-, 60-, 90-, and 120-second enamel etching time intervals.
Methods: The facial surfaces of 150 bovine maxillary incisors were mounted and etched with one of two different etchants (Dentsply 34% Phosphoric Acid or Opal Orthodontics Opal Etch 35%, a self-limiting phosphoric acid) at varying timed intervals of 15, 30, 60, 90, and 120 seconds. The teeth were then primed, cured, and bonded with resin rods. Shear bond strength and fracture mode were recorded.
Results: The results of the two-way ANOVA found that Opal Etch had significantly greater bond strengths to enamel compared with the control etchant ($P < 0.001$). Ninety seconds of etching time resulted in the greatest bond strength for both groups, but was not significantly different from 30 seconds; 120 seconds had the lowest bond strength to enamel in both groups, but was not significantly different from 15 seconds. A one-way ANOVA was performed per etchant. A Bonferroni correction was applied because multiple comparisons were completed ($\alpha = 0.025$). For the Opal Etch, a significant difference was found based on time ($P = 0.007$).
Conclusion: The shear bond strength of Opal Etch consistently tested higher than the Dentsply etchant, but was not found to be self-limiting.

1. Introduction

The chemical etching of tooth enamel with phosphoric acid was discovered by Buonocore in 1955 [1]. In 1973, Retief [2] reintroduced the idea of etching and bonding teeth with improved composites that significantly reduced shrinkage and microleakage. This new method of bonding provided strength suitable for bonding orthodontic brackets to teeth [3].

Other etchant types have been reported in the literature to include EDTA, citric acid, and nitric acid. These may be beneficial if clinically useful orthodontic bracket bond strengths can be maintained while decreasing the depth of enamel dissolution [4]. Despite the discovery of different etchant materials, phosphoric acid remains the gold standard.

Buonocore's [1] initial 85% phosphoric acid was not the ideal concentration of acid. Retief [2] discovered that teeth etched with 50% phosphoric acid achieved bond strengths that exceeded 600 psi and that this would be sufficient to retain orthodontic brackets. Further research found that phosphoric acid in concentrations of 30% to 40% produced retentive enamel surfaces and increased bond strengths [5,6]. Studies show that concentrations lower than 30% can have similar adhesion values [7–9]. Currently, manufacturers produce acid etchants with phosphoric acid concentrations between 30% and 40%. At these concentrations, enamel surfaces are well etched and enamel surface loss during etching is between 10 and 30 μm [6,10].

The initial recommended acid etching time was 60 seconds [11]. Further research demonstrated etching times of 15 to 20 seconds was equally effective [6,12–15]. Etch time should be varied according to the clinical situation. If a tooth is suspected of having

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high fluoride content, it is advised to etch the tooth for a total of 25 to 30 seconds [16].

Currently, ideal etch time varies from 15 to 60 seconds [17–19]. Overetching can occur at etch times that exceed 1 to 2 minutes such that bond strength, specifically shear bond strength, is compromised. Wang and Lu [20] demonstrated there were no statistically significant differences in bond strength among 15-, 30-, 60-, or 90-second etching times. However, the 120-second etching group showed significantly less bond strength. Overetching occurs when both tooth structure and bond strength become compromised. Wang and Lu [20] showed that between 60 and 90 seconds, resin-enamel fractures occur and that at 120 seconds, bond strengths significantly decrease. Ideal etch depth is gauged to be between 5 and 50 μm, as determined by scanning electron microscope [6,11]. The bonding process involves discrete etching of enamel to provide selective dissolution of prism cores with resultant microporosity into which resin can flow and be polymerized to form a mechanical bond to the enamel. Retief et al. [21] noticed that this procedure practically eliminated microleakage at the tooth/restoration interface.

Recently, Opal Orthodontics, a division of Ultradent Corporation (Ultradent, South Jordan, UT) promoted Opal Etch (35% phosphoric acid) as a self-limiting etchant. The manufacturer states that Opal Etch has unique self-limiting properties that prevent overetching [22]. There is no research to support this claim. Overetching can present itself as a linear phenomenon (depth), but clinically overetching would present as early bond failure due to a compromised adherend (the tooth surface). The objective of this experiment was to compare the shear bond strengths of Opal Etch to a standard etchant system at 15, 30, 60, 90, and 120 seconds.

2. Methods and materials

Bovine maxillary incisors were used for this study (Animal Technologies, Inc., Tyler, TX). Teeth with defects were excluded. Teeth were stored in 0.5% chloramine-T solution and kept at 37°C in a laboratory oven (Model 20GC; Quincy Lab, Chicago, IL). A total of 150 teeth were selected. Retention cuts were made with a high-speed handpiece (Midwest Quiet Air, York, PA) and a tapered diamond bur (Brasseler Super Coarse RCBK #5877K.31.016, Savannah, GA). Specimens were stored in the laboratory oven in a 0.5% chloramine-T at 37°C.

Each tooth was placed on soft boxing plate wax (Red Boxing Wax Strips; KINCO, Avon, OH) so that the tooth was raised above the height of a small piece of PVC piping. The piping was placed around the tooth and Yellow Dental Stone (Golden Microstone; Whipmix, Louisville, KY) was poured into the PVC piping. Once set, the boxing wax was removed and mounted specimens were placed back into chloramine-T solution and stored in the oven at 37°C.

The bovine teeth were tested within a 6-month period after extraction. The two etchant groups, each containing 75 teeth, were subdivided into five subgroups of 15 specimens. Each subgroup was etched with phosphoric acid using either 35% Opal Etch or 34% Tooth Conditioner Gel for the subgroup's designated time (15, 30, 60, 90, and 120 seconds). The facial surface of the crown was flattened using a drill press (TBM 115 Type 38128; Proxxon, Luxembourg) and a coarse wheel (diamond coarse wheel #6909DC.31.040; Brasseler). Specimens were sanded flat using 600-grit sandpaper. Each tooth was rinsed using a heavy stream of tap water for 10 seconds and dabbed dry using a paper towel.

Curing light output was measured (L.E.D. Radiometer, Demetron; Kerr Corporation, Orange, CA) to verify a minimum energy of 1200 mW/cm^2. A thin layer of Transbond XT Primer Light Cure Adhesive Primer (3M Unitek, Monrovia, CA) was applied to the tooth and brushed vigorously on the facial surface of the tooth for 3 seconds. A stream of moisture-free air was used to produce a thin, uniform layer of primer on the tooth surface. A dental curing light (Valo LED Curing Lite; Ultradent, South Jordan, UT) was used to polymerize the primer for 3 seconds. The mounted tooth was then placed in a jig (Bonding Clamp; Ultradent, South Jordan, UT) and secured beneath a bonding mold insert (Ultradent, South Jordan, UT). The bonded area was limited to the 2.4-mm circle diameter determined by the mold. Composite resin (Z250 Filtek; 3M ESPE, St. Paul, MN) was applied incrementally using a modified microbrush to adapt the composite within the plastic mold to a height between 3 and 4 mm. Each layer was polymerized for 20 seconds. All samples were stored in the solution and oven for 24 hours, then samples were placed in a jig (Test Base Clamp; Ultradent, South Jordan, UT) to hold the flat surface of the sample vertical. The jig was placed in a universal testing machine (Instron 5943R9153; Instron, Norwood, ME). A straight blade was mounted onto the 1 kN load cell and placed against the flat surface of the test sample. The blade engaged the resin rod at a crosshead speed of 1 mm/min until bond failure occurred. Shear bond strength was calculated for each specimen in megapascals by calculating the peak load of failure in Newtons divided by the specimen surface area (4.41 mm^2). The mean and standard deviation were determined per group and subgroup. Specimens were examined under a ×20 stereomicroscope (Nikon stereomicroscope model #553730, Tokyo, Japan) to determine failure mode as follows: (1) adhesive fracture at the adhesive interface, (2) cohesive fracture in the enamel or composite, or (3) mixed (combination of adhesive and cohesive) in enamel and composite.

A power analysis was used to anticipate the likelihood that 15 specimens per group would yield a significant effect, providing guidelines to accept or reject the null hypothesis. A one-way ANOVA and Tukey's post hoc test were used to determine if there was a significant difference based on etching time of the two etchants at $\alpha = 0.05$. A Bonferroni correction was applied because multiple comparisons were made between time groups and etchant ($\alpha = 0.025$). A sample size of 15 teeth per group provided 80% power to detect an effect size of 0.23 (approximately 0.46 SD difference) among means for the main factor of etch material, and a small effect size of 0.29 (or approximately 0.58 SD difference) among means for the main factor of time and for the interaction term, when testing with a two-factor ANOVA at $\alpha = 0.05$ (NCSS PASS 2002, NCSS, LLC, Kaysville, UT).

3. Results

Results of the bond strength tests appear in Table 1. The results of the two-way ANOVA revealed that there was no significant difference in bond strength between Opal Etch compared with the Dentsply etchant ($P = 0.28$) with no significant interaction ($P = 0.32$). Ninety seconds of etching time resulted in the greatest bond strength in both groups, and was significantly different from the other four time groups; 120 seconds had the lowest bond strength to enamel in both groups. A one-way ANOVA was performed for each etchant. A Bonferroni correction was applied because multiple comparisons were completed ($\alpha = 0.025$). For the both the Opal and Dentsply etchants a significant difference was found based on time. Both etchants showed a decrease in shear bond strength at 120 seconds, which was significantly less than 90 seconds ($P < 0.001$).

Additional nonparametric data were collected based on the fracture mode of the resin rods (Table 2). A comparison between all groups was performed to evaluate the mode of fracture. There was no difference in the frequency of mode of fracture among the five groups ($P > 0.05$); however, within each group and type of etch there was a significant difference in the mode of fracture. Of the 10 possible groups that were statistically analyzed (etch time and type of etch), only the 90-second etch with the traditional etchant was not significant. For all other groups, the fracture mode was predominantly adhesive. This type of fracture mode displayed a clean