# EFFECTS OF ETCHING MODE ON BOND STRENGTH OF UNIVERSAL ADHESIVES

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**ABSTRACT**

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**Introduction:** The aim of this study was to analyze the bond strength to dentin produced by new universal adhesive systems used in self-etch and etch-and rinse application modes.
**Materials and Methods:** Sixty human teeth were divided into 6 groups according to the different universal adhesive systems: Scotchbond Universal (SBU - 3M ESPE), Clearfil Universal (CFU - Kuraray), Futurabond U (FBU - VOCO) Xeno Select (XS - Dentsply De Trey), Prime&Bond Elect (PBE – Dentsply Caulk) and All Bond Universal (ABU, Bisco). Then, the teeth were subdivided into 2 subgroups, according to the application mode: etch-and-rinse or self-etch. Composite crowns were built after application of the adhesive systems and the restored teeth were sectioned in both "X" and "Y" directions into sticks with a cross-sectional bonded area of approximately 1 $mm^2$. The microtensile test was carried on a universal testing machine operated at a crosshead speed of 1 mm/min. Bond strength values were statistically evaluated using two-way ANOVA and the Tukey post-hoc test.
**Results:** SBU, XS and ABU presented significantly higher bond strength values when applied on the etch-and-rinse mode ($p < 0.05$). CFU, FBU and PBE presented no significant difference in bond strength values between etch-and-rinse and self-etching groups ($p > 0.05$).
**Conclusion:** The adhesive performance of Universal Adhesives was similar or higher when they were used in the etch-and-rinse mode in comparison with the self-etching mode.
**Keywords:** Acid etching; Dental; Bond strength; Dental bonding; Universal adhesives.



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## 1. Introduction

Bonding to enamel and dentin is mainly accomplished by micromechanical interlocking between synthetic, naturally degradable polymers, and enamel or dentin collagen fibrils [1]. Effective, long-lasting bonding to dentin has been a challenge to dental clinicians, because in order to promote adhesion to dentin, the mineral phase needs to be totally or partially removed, and substituted by an adhesive solution, that will permeate this collagen-rich layer, and polymerize in situ, forming what has been called the hybrid layer [1-3].
Different approaches, with different numbers of steps and degrees of sensitivity have been used to bond resin-based materials to enamel and dentin [4-6]. Efforts have been directed to reduce the number of steps and technique sensitivity. One-bottle self-priming etch-and-rinse systems, as well as single-step self-etching adhesives are simplified versions of their multiple-step precursors, and have been recently combined and marketed as Universal adhesives [7,8]. These multimodal adhesives may be used in etch-and-rinse mode, self-etch mode or selective-etch mode, depending on the clinician's preference [9,10].
Although recent studies reported that universal adhesives applied using either the etch-and-rinse or the self-etch mode produce excellent immediate bond strength to bonding substrates [11], limited information is available on the newest universal adhesives recently introduced by different manufacturers. Thus, the aim of this study was to evaluate the bond strength to dentin produced by six universal adhesives applied either on the etch-and-rinse or self-etching mode. The tested null hypothesis was that there is no difference in bond strength produced by universal adhesives applied on the etch-and-rinse or self-etching mode.

**Table 1.** Universal adhesives short name, manufacturer, pH, composition, and application instructions.

| Adhesive, (Batch Number) | pH | Composition | Self-Etch | Etch-and-Rinse |
|---|---|---|---|---|
| Scotchbond Universal (SBU) 3M Espe, St Paul, MN, USA (554836) | 2.7 | 2-HEMA, 10-MDP, dimethacrylate resins, VitrebondTM copolymer, silane, filler, ethanol, water, iniciators | 1. Apply the adhesive to the prepared tooth and rub in for 20 s<br>2. Gently air-dry the adhesive for 5 s for the solvent to evaporate<br>3. Light cure for 10 s | 1. Apply etchant for 15 s<br>2. Rinse for 10 s<br>3. Air dry 2 s<br>4. Apply adhesive as for the self-etch mode |
| Clearfil Universal Bond (CFU) Kuraray, Tokyo, Japan (C40001) | 2.3 | HEMA, MDP, Bis-GMA, ethanol, camphorquinone, hydrophilic aliphatic dimethacrylate, silane coupling agent, colloidal silica, water, and accelerators | 1. Apply bond and rub for 20 s or 40 s<br>2. Dry by blowing mild air for 5 s<br>3. Light cure for 10 s | 1. Apply etchant for 10 s<br>2. Rinse thoroughly<br>3. Dry<br>4. Apply adhesive as for the self-etch mode |
| Futurabond U (FBU) VOCO, Cuxhaven, Germany (1333352) | 2.3 | 2-HEMA, Bis-GMA, HEMA, acidic adhesive monomer, urethane dimethacrylate, catalyst, silica nanoparticles, ethanol | 1. Activate single dose adhesive package<br>2. Apply adhesive to the cavity surface using the Voco Single Tim brush and rub adhesive in for 20 s<br>3. Dry adhesive with dry, oil-free air for at least 5 s<br>4. Light cure for 10 s | 1. Apply etchant for 15 s<br>2. Rinse for 10 s<br>3. Air dry 2 s<br>4. Apply adhesive as for the self-etch mode |
| Xeno Select (XS) Dentsply De Trey, Konstanz, Germany (1402000636) | 1.3 | Bifunctional acryl resin with amide functions, Acryloylamino alkylsulfonic acid, "inverse" functionalized phosphoric acid ester, Camphorquinone, Coinitiator, Butylated benzenediol, Water, tert-Butanol | 1. Apply the adhesive to the prepared tooth and rub in for 20s<br>2. Gently air-dry the adhesive for 5 s for the solvent to evaporate<br>3. Light cure for 10 s | 1. Apply etchant for 15 s<br>2. Rinse for 10 s<br>3. Air dry 2 s<br>4. Apply adhesive as for the self-etch mode |
| Prime&Bond Elect (PBE) Dentsply Caulk, Milford, DE, USA (141008) | 2.5 | Mono-, di- and trimethacrylate resins, PENTA, diketone; organic phosphine oxide, cetylamine hydrofluoride, acetone, water, self-cure activator | 1. Apply generous amounts of adhesives to thoroughly wet all tooth surfaces<br>2. Agitate applied adhesive for 20 s. Re-apply to coat preparation for the entire 20 s period<br>3. Remove excess solvent by gentle dryingwith clean, dry air for at least 5 s<br>4. Light cure for 10 s | 1. Condition enamel for at least 15 seconds and dentin for 15 seconds or less.<br>2. Rinse for 15 s<br>3. Dry<br>4. Apply adhesive as for the self-etch mode |
| All Bond Universal (1300006652) | 3.2 | 2-HEMA, 10-MDP, Bis-GMA, ethanol, water, initiators | 1. Apply two separate coats of adhesive with agitation for 10-15 s per coat<br>2. Evaporate solvent by thorough air-drying for least 10 s.<br>No visible movement of adhesive<br>3. Surface should have a uniform glossy appearance. If not, repeat steps 1 and 2<br>4. Light cure for 10 s | 2. Rinse thoroughly<br>3. Remove excess water by blotting surface with an absorbent pellet or high volume evacuation for 1-2 s, leaving the preparation visibly moist<br>4. Apply adhesive as for the self-etch mode |

**Abbreviations:** 2-HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A glycidyl methacrylate; PENTA, dipentaerythritol penta acrylate monophosphate.

## 2. Materials and Methods

Sixty freshly extracted human third molars were used. The teeth were obtained following an approved protocol by the review board of the University of Guarulhos (# 641.271). After disinfection and removal of soft tissues, the flat coronal dentin surfaces were exposed using 600-grit SiC paper under running water to create a standardized smear layer.

The teeth were randomly assigned to six experimental groups, which were restored using six commercially available universal adhesive systems: Clearfil Universal (Kuraray), Scotchbond Universal (3M Espe), Futurabond U (Voco), Prime&Bond Elect (Dentsply Caulk), All Bond Universal (Bisco) and Xeno Select (Dentsply De Trey). The composition, batch number and application instructions are listed in Table 1. The sixty teeth were randomly assigned to 6 test groups, according to the universal adhesives used, and then subdivided into 2 subgroups according to the application mode: etch-and-rinse or self-etching ($n = 5$). For the etch-and-rinse groups, 35% phosphoric acid was applied for 15 s, thoroughly rinsed with water, and excess water was removed with cotton pellets. Care was taken not to dehydrate dentin surfaces prior to adhesive application. For the self-etching groups, the dentin surface was dried with an air stream prior to the adhesive application.

After application of the adhesive resins according to the manufacturers' instructions, composite crowns of 5 mm in height were built up incrementally with composite resin (TPH3, Shade A3, Dentsply Caulk, Milford, DE, USA). A LED light-curing unit (Radii Plus - SDI, Victoria, Australia) with a power output of 1,500 $mW/cm^2$ was used to polymerize all specimens. Each increment (not exceeding 2 mm thickness) was light cured for 20 seconds. The restored teeth were stored in distilled water at 37°C for 24 hours.

Afterwards, the restored teeth were serially sectioned perpendicularly to the adhesive-tooth interface into slabs, and the slabs into beams with a cross-sectional bonded area of approximately 1 $mm^2$ using a diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA). Beams were fixed to the grips of a universal testing machine (EZ Test; Shimadzu Corp, Kyoto, Japan) using a cyanoacrylate adhesive (Loctite Super Bonder Gel; Henkel, Düsseldorf, Germany) and tested in tension at a crosshead speed of 1 mm/min until fracture occurred. The maximum tensile load was divided by specimen cross-sectional area, measured with a digital caliper (Mitutoyo Co., Tokyo, Japan), to express results in units of stress (MPa). Five beams were selected from each restored tooth, and the average value for each tooth was used in the calculations. Bond strength values were statistically evaluated using a two-way ANOVA and the Tukey post-hoc test at a preset significance level of 0.05. Statistical analyses were performed using a personal computer program (SAS V9, SAS Institute, Cary, NC).

Failure modes were determined by visual examination of fractured specimens in a stereomicroscope at a magnification of 50X (PanTec, Panambra Ind. e Tecnica SA, Sao Paulo, Brazil). Failure was classified according to one of four types: cohesive failure in dentin, adhesive failure at the adhesive-dentine interface, cohesive failure in resin composite or mixed failure.

## 3. Results

The mean bond strength values and standard deviation for the different groups are are shown in Table 2. The two-way ANOVA revealed that there were statistically significant differences for the factor "universal adhesive" ($p < 0.00021$) and for the factor "etching mode" ($p = 0.00001$). In addition, it identified a significant interaction between the two factors ($p = 0.00157$). The Tukey post-hoc test showed significant differences among adhesive systems for the different etching modes ($p < 0.05$).

Scotchbond Universal, Xeno Select and All Bond Universal presented significantly higher bond strength values when applied on the etch-and-rinse mode ($p < 0.05$). Clearfil Universal, Futurabond U and Prime&Bond Elect presented no significant difference in bond strength values between the etch-and-rinse and self-etchnig groups ($p > 0.05$).

When the etch-and-rinse mode was used, Scotchbond Universal and Xeno Select presented the highest μTBS values, with no significant difference between them ($p > 0.05$). However, Xeno Select was not significantly different from the other groups ($p > 0.05$). For the self-ecthing mode groups, the highest μTBS values were presented by Futurabond U and Scotchbond Universal, with no significant difference between them ($p > 0.05$). However, Scotchbond Universal was not significantly different from the other Universal Adhesives when used in self-etching mode ($p > 0.05$).

Fig. 1 shows the distribution of fracture patterns for the different groups. The failure mode analysis revealed that the majority of failures were adhesive at the adhesive-dentin interface for most groups, except for Scotchbond Universal and All Bond Universal applied on the etch-and-rinse mode, which presented a high number of cohesive failures in resin composite.

## 4. Discussion

Recently, a new type of single-step self-etching adhesive has been introduced. This type of self-etching adhesive is categorized as "universal" or "multi-mode" as it can be used either with the etch-and-rinse or the self-etching approaches [12-15]. Therefore, universal adhesives are used with phosphoric acid pre-etching in the etch-and-rinse or selective-etch approaches, which enhances bond strength to enamel. In addition, it also provides a simplified self-etching approach for dentin substrate [16]. However, this type of adhesive has only recently been introduced to the market, and there is little information as to whether the different etching

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**Table 2.** Mean bond strength values in MPa (SD) produced by the universal adhesives applied in self-etching and etch-and-rinse modes.

| Universal Adhesives | Etch-and-Rinse | | Self-etching | |
|---|---|---|---|---|
| Scotchbond Universal | 96.8 (14.9) | Aa | 47.5 (17.6) | ABb |
| Clearfil Universal | 52.2 (11.1) | Ba | 36.6 (13.0) | Ba |
| Futurabond U | 63.7 (14.4) | Ba | 67.5 (5.3) | Aa |
| Xeno Select | 76.1 (31.5) | ABa | 40.4 (10.7) | Bb |
| Prime&Bond Elect | 56.0 (8.4) | Ba | 40.7 (7.2) | Ba |
| All Bond Universal | 65.0 (7.1) | Ba | 27.6 (4.2) | Bb |

Means followed by different letters (lower case – row, upper case – column) differ among them by Tukey test at 95% confidence level.

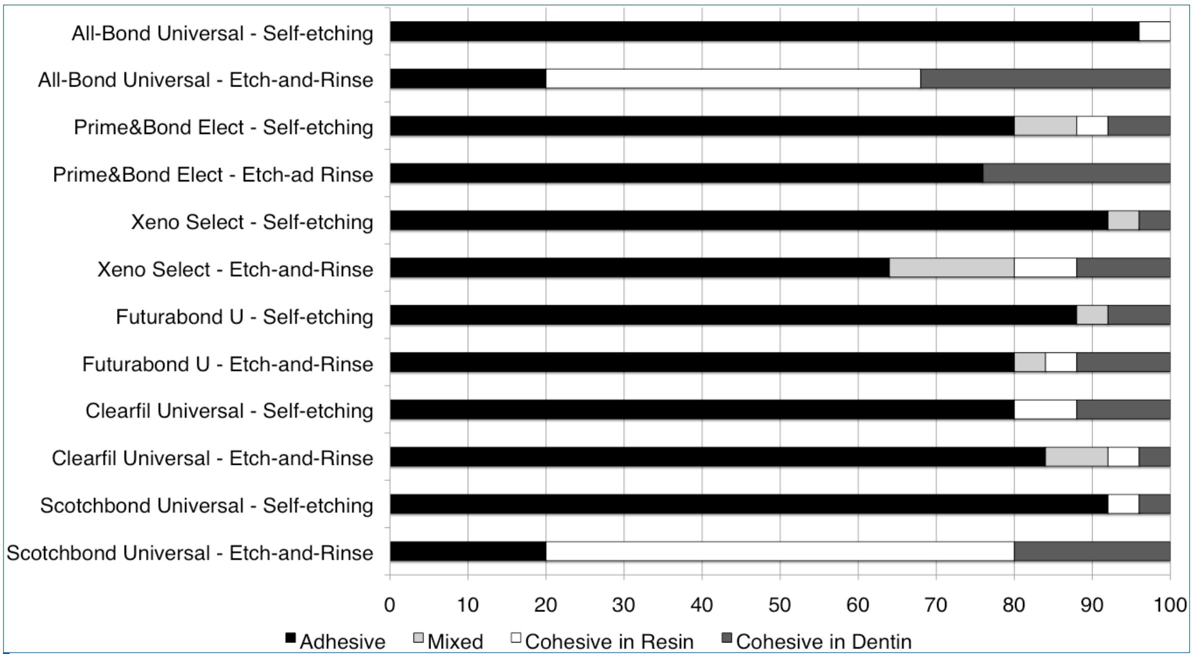


**Figure 1.** Distribution of failure modes for the different groups.

modes achieve equivalent bonding performance to dentin. Our null hypothesis was rejected, because for three of the tested universal adhesives, bond strength was significantly higher when the etch-and-rinse approach was used.

The resin composition as well as the presence and type of fillers might play important roles in bonding effectiveness [17]. Each self-etch adhesive contains its specific functional monomer that, to a large extent, determines its actual adhesive performance [18]. The specific molecular formula of the functional monomer and the dissolution rate of its calcium salt are thought to influence bonding performance. The potential to chemically interact with interfacial hydroxyapatite might be helpful in the adhesion process. This interaction occurs with mild self-etching adhesives that partially demineralize the dentin surface. It has been shown that MDP (10-methacryloxydecyl dihydrogen phosphate) is effective in bonding to hydroxyapatite, and seems to be very stable. Three of the tested adhesives (Clearfil Universal, Scotchbond Universal and All Bond Universal) present MDP as functional monomer. While the components in these materials are similar, there may be differences in the quantities and proportions of water, solvent, MDP, and dimethacrylate resins among the adhesives. There is a possibility that such differences may influence viscosity and wettability of each bonding agent, affecting the ability of resin monomers to penetrate into decalcified dentin [16]. When used in the self-etching mode, these three MDP-containing universal adhesives presented bond strength values that were not significantly different from each other. However, when used on the etch-and-rinse mode, Scotchbond Universal presented significantly higher bond strengths.

More than a decade ago, when single step self-etching adhesives were first introduced to the market, they were not recommended for use in the etch-and-rinse mode, because lower bonding performance to dentin was observed when phosphoric acid was used prior to adhesive application [4,19,20]. In the present investigation the immediate adhesive performance of the recently introduced universal adhesives was always significantly higher or not significantly different when used in the etch-and-rinse mode. Demonstrating that adjustments in the chemical formulation of single-step self-etching adhesives was made so they can also be used in the etch-and-rinse mode. It seems that the problem of the bonding mode incompatibility has been solved by manufacturers through blending less acidic resin monomers in the appropriately reduced concentrations with other resin monomers [21].

Three of the tested adhesives (Scotchbond Universal, Xeno Select and All Bond Universal) presented significantly lower bond strength values when used in the self-etching mode, in comparison with the etch-and-rinse groups. This reduction probably occurs due to the higher pH of these adhesives, classified as ultra-mild systems, in comparison with the other products. The interaction depth with dentin depends on the pH of the adhesives [21]. Depending on the pH, self-etch adhesives may be classified into ultra-mild (pH > 2.5, 0.2–0.5 µm interaction depth), mild (pH ≈ 2; 0.5–1 µm interaction depth), intermediate (pH, 1–2; 1–2 µm interaction depth), and strong (pH < 1, > 5 µm interaction depth, similar to etching with phosphoric acid) [22]. More aggressive self-etching systems present higher contents of acidic monomers and water, resulting in increased hydrophilicity, which will result in increased water sorption, and consequently, decreased hydrolytic stability [4]. In addition, continued etching along the base of hybrid layers after polymerization of those adhesives can occur [23]. Among the products tested, All Bond Universal presents the highest pH, 3.2. Even though not significantly different, it also presented the lowest bond strength values when used in the self-etching mode. On a study by Chen et al. [21], TEM observations revealed that All Bond Universal presented the shallowest interaction with dentin when used in the self-etching mode, approximately 0.2 µm. On the other hand, when used in the self-etching mode, Futurabond U presented the highest bond strength values. However, in recent reports on the long-term performance of universal adhesives, Zhang et al. [10] and Chen et al. [21] reported remarkable decrease in bond strengths and nanoleakage with signs of water-treeing on resin dentin interfaces produced with Futurabond U [21,10]. In fact, all universal adhesives tested in the above-mentioned study of Zhang et al. [10], with the exception or Prime&Bond Elect and Scotchbond Universal (applied in self-etching mode), presented significant reduction in bond strengths after 12 months of storage. Previous investigations are in accordance with the present study, which demonstrated similar or higher performance, clinically or in vitro, when universal adhesives are applied in the etch-and-rinse mode [12,13,24]. Even though the hybrid layer thickness is approximately 10 times thicker when used in the etch-and-rinse mode (≈ 5 µm) in comparison with the self-etching approach (≈ 0.5 µm), thicker hybrid layers formed in dentin substrates have been shown not to necessarily produce higher bond strengths [25].

## 5. Conclusion

According to the results of the present investigation, the immediate bonding performance of Universal Adhesives was similar or higher when they were used in the etch-and-rinse mode in comparison with the self-etching mode.

## Author Contributions

AR: participated in the study design, statistical analy. PA and RK: participated in the microtensile bond strength test, and manuscript writing. sis and manuscript writing.

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# Questions

**1. Universal adhesives can be applied in:**

❑a. Self-etching mode;
❑b. Etch-and-rinse mode;
❑c. Selective etching mode;
❑d. All the answers are correct.

**2. What are the advantages of using the self-etching mode when applied on deep dentin?**

❑a. Lower post-operative sensitivity, due to maintenance of smear plugs and shallower demineralization in comparison with 35% phosphoric acid etching;
❑b. Higher bond strengths;
❑c. Self-etching adhesives don't need to be light-cured;
❑d. Better esthetics.

**3. Depending on the pH, self-etch adhesives may be classified into ultra-mild (pH > 2.5, 0.2–0.5 µm interaction depth), mild (pH ≈ 2; 0.5–1 µm interaction depth), intermediate (pH, 1–2; 1–2 µm interaction depth), and strong (pH < 1, > 5 µm interaction depth, similar to etching with phosphoric acid). About the adhesives/ pH, it is correct to say that:**

❑a. Lower pH results in more aggressive demineralization;
❑b. Lower pH results in higher hydrophilicity;
❑c. For ultra-mild and mild self-etching adhesives, enamel selective etching with phosphoric acid is recommended;
❑d. All the answers are correct.

**4. The potential to chemically interact with interfacial hydroxyapatite might be helpful in the adhesion process. This interaction occurs with mild self-etching adhesives that partially demineralize the dentin surface. It has been shown that MDP (10-methacryloxydecyl dihydrogen phosphate) is effective in bonding to hydroxyapatite, and seems to be very stable. It is correct to say that:**

❑a. All functional monomers are the same;
❑b. The specific molecular formula of the functional monomer and the dissolution rate of its calcium salt are thought to influence bonding performance;
❑c. MDP is the only functional monomer available in the market;
❑d. All Universal Adhesives present MDP in their composition.