Research Square

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Effect of Micro-applicator on Universal Adhesives Bond Strength to Enamel

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Abstract

Objective

To compare universal adhesives (UAs) shear bond strength (SBS) to enamel using different brands of disposable micro-applicators.

Methods and materials

Crowns of 216 bovine incisors were used, with their buccal enamel surfaces exposed and polished until 600-grit, and randomly assigned into 12 groups (n = 18). Two UAs with different solvents (ethanol for PeU.Peak Universal Bond Adhesive; or acetone, ethanol, and water for OpU.OptiBond Universal 360) were applied according to manufacturer's instructions. Half of the specimens were applied as etch-and-rinse mode (ER) using 35% phosphoric acid (Ultra-Etch), while the other half were applied as self-etch mode (SE). On control groups, UAs were applied using their own disposable micro-applicator: a brush fibers tip (Ultradent Inspiral Brush Tip) for PeU or a micro-brush with fiber bristles (Kerr Applicators) for OpU. Also, UAs were applied using other 2 disposable micro-applicators: a fiber-free elastomer bristles (ZerofloX) or a general-brand micro-brush with fiber bristles (Aplik). UAs were light-cured for 10 sec using a LED (Valo). Composite cylinders 2.38 mm wide and 2.0 mm high were built (Filtek Z250 XT) and light-cured (20 sec) using an Ultradent Bonding Assembly. Specimens stayed in water (37^{o}C) for 6 months and then were subjected to thermal aging (10.000 cycles of 60 sec, 5 to 55^{o}C) prior to being subjected to SBS test. A hardened steel precise notch with Ø=2.38 mm semi-circular and 0.4 mm thick loaded the composite cylinders until fracture. The failure mode was analyzed under optical microscopy 40× and classified as: adhesive (AD), mixed (MI), cohesive in composite (CC), and cohesive in enamel (CE). To confirm failure mode, five specimens of each group were analyzed under SEM and EDX. Statistical analyses included ANOVA and Tukey HSD *post-hoc* test (α = 0.05). Enamel specimens from buccal and lingual surfaces of human molars (n = 3) were polished (up to 1,200-grit) and prepared for SEM analysis after the application of 2 bonding modes using the UAs.

Results

The mean SBS (± SD) ranged from 13.2 (± 3.8) MPa (SE.OpU.ZerofloX) to 39.7 (± 9.8) MPa (ER.PeU.Aplik). For both UAs, ER showed significantly higher SBS than SE ($p < 0.001$). PeU resulted in significantly higher SBS on enamel than OpU ($p < 0.01$). For OpU, ZerofloX resulted in significantly lower SBS than Kerr Applicators and Aplik ($p < 0.005$). There was no statistical difference between the mean SBS on enamel when PeU was applied with the 3 micro-applicators. MI failures predominated in ER groups. SE groups showed mainly AD failures. For SE, OpU resulted in featureless morphology resembling that of untreated enamel. PeU resulted in shallow intraprismatic etching. For ER, OpU and PeU displayed the classical phosphoric acid etching pattern on the entire surface.

Conclusions

UAs applied using ER mode resulted in higher SBS to enamel compared to SE. Elastomeric fiber-free micro-applicator reduced bond strength to enamel when the UA has the solvent acetone.

Clinical Relevance

Using the micro-applicator provided by the universal adhesive manufacturer is recommended. Elastomeric micro-applicator should not be used with acetone-containing universal adhesive.

Introduction

Adhesive procedures play a key role in the clinical success and longevity of composite resin restorations and support restorative approaches that preserve tooth structure [1–5]. Over the past decades, there has been a trend toward simplifying the adhesive dental clinic procedures by reducing the number of application steps and clinical time [6, 7]. For enamel bonding perspective, phosphoric acid etching step of enamel prior to adhesive system application remains the most widely recommended protocol for achieving effective micromechanical retention to enamel [4, 8–12]. But some adhesive systems combine chemical bonding monomers at low pH that can self-etch enamel surface and result in bond strength to enamel [9, 12–15] and degree of microleakage in enamel composite restoration margins [16–18] similar to etch-and-rinse adhesive systems. And also, clinical retention with good margin sealing composite resin restoration over the years [19–21].

Many universal adhesives (UAs) have been launched by dental manufacturers over the last fifteen years. UAs provide flexibility in its clinical use. They combine adhesion to multiple surfaces while maintaining effective bonding to dental substrates [2, 3, 11, 22]. UAs differ between each other per composition characteristic (functional monomer and its concentration, solvents, acidity). But nowadays, the majority of them contain 10-methacryloyloxydecyl dihydrogen phosphate (MDP) functional monomers. MDP-containing adhesive systems are known to form ionic interactions with hydroxyapatite, which may contribute to bond stability [2, 6, 22, 23]. Considering bonding to enamel, clinicians can use UAs in both self-etch (SE) and etch-and-rinse (ER) modes [2, 3]. And, recent clinical trials have shown similar performance of composite resin restorations with or without enamel phosphoric acid-etching step when using 10MDP-containing adhesive systems [5, 24]. However, some UAs are introduced to the dental market without having been evaluated in independent clinical studies. Because clinical studies are costly, testing bond strength to dental hard tissues under simulated aging conditions is highly important for all UAs in both application modes (SE or ER).

Nevertheless, several factors may influence enamel adhesion, such as adequate substrate cleanliness [25, 26], proper isolation to prevent contamination such as saliva and blood [27–31], and the adhesive application protocol). Regarding the application protocol, the active application of the adhesive system to the dental surface can influence adhesion and restoration longevity [15, 32–35]. Scrubbing the

adhesive system with a micro-applicator promotes deeper penetration of the adhesive into the microporosities of the demineralized dentin and into the micro-retentions created by phosphoric acid etching on enamel [2, 34–37]. However, it has been previously reported Berton el al [38] that micro-applicator microfibers (Microbrush Series Regular, Microbrush International) may remain adhered to the adhesive surface following adhesive application, as demonstrated by scanning electron microscopy (SEM). This may represent an additional risk factor that could reduce bond strength and compromise restoration longevity. The quality of the micro-applicator appears to be an important factor influencing adhesive performance. The current dental market offers a wide range of disposable micro-applicators. Typically, disposable micro-applicators composed of non-linting synthetic fibers are used for this purpose [38]. Many commercially available micro-applicators have bristles made from non-woven material (also known as non-woven fabric) [38]. Recently, a fiber-free disposable micro-applicator (ZerofloX, Medmix, Switzerland) composed of elastomeric bristles was introduced [39]. According to the manufacturer, one of the proposed advantages of this design is the elimination of residual microfiber contaminants during adhesive application [39]. However, to the authors' knowledge, no study has yet evaluated the effect of this elastomeric fiber-free micro-applicator on dentin or enamel bond strength compared with regular fiber-based disposable micro-applicators. Thus, the aim of this study was to evaluate whether the type of disposable micro-applicator influences the bond strength of composite resin to enamel when using two UAs applied in SE and ER modes. Additionally, the enamel etching pattern produced by the UAs was evaluated. The null hypotheses were that composite resin bond strengths to enamel will not be influenced by: (1) UA selected, (2) bonding mode of the UA (SE or ER), nor (3) the disposable micro-applicators used to apply the UA.

Materials and methods

Sample size calculation

Sample size calculation was determined based on previous bond strengths study [40]. According to the literature, the mean composite resin shear bond strengths to enamel after aging using UAs is 18.6 MPa with a standard deviation of ± 5.5 MPa [40]. Considering a significance level of 0.05, statistical power of 80%, and a two-tailed test, the minimum required number of bovine incisors per group was estimated to be 15 (n = 15). Considering a possible sample loss during thermocycling, a sample size of 18 teeth per group was established (n = 18). Sample size calculation was performed using the statistical software G*Power 3.1.

Shear bond strengths to enamel

Two UAs were selected according to its characteristics: functional monomers, solvents, pH, number of steps, and application protocol. The materials utilized, manufacturers, lot numbers, compositions and application steps are shown in Table 1. Figure 1 shows a schematic flow chart for composite resin shear bond strengths to enamel.

Table 1
Materials, compositions, lot numbers, application protocols, and experimental groups

Universal adhesive: composition, pH, lot and application protocols as per manufacturer's instructions	Bonding mode	Group	Microapplicator: composition and lot
OptiBond Universal 360 (Kerr Dental, USA) Lot number: A175999 | pH: 2.0–3.0 **Composition**: MDP, MMA, DMA, MAA, water, ethanol, acetone, initiator, stabilizer, silica filler. **Application**: scrub for 20 sec; gentle air for air-dry for 5 sec; light-cure for 10 sec.	Etch-and-rinse (ER)	ER.OpU.K	**Kerr Applicators (Kerr Dental, USA)** Lot number: A175868 Composition: synthetic fibers.
		ER.OpU.Z	**ZerofloX (Medmix, Switzerland)** Lot number: 0100356114 Composition: fiber-free elastomeric bristles.
		ER.OpU.A	**Aplik (Angelus, Brazil)** Lot number: 75567 Composition: non-woven bristles.
	Self-etch (SE)	SE.OpU.K	**Kerr Applicators (Kerr Dental, USA)**
		SE.OpU.Z	**ZerofloX (Medmix, Switzerland)**
		SE.OpU.A	**Aplik (Angelus, Brazil)**
Peak SE Primer (Ultradent Products Inc., USA) Lot number: BYWBV | pH: 0.9–1.5 **Composition**: ethanol, HEMA, PEGDA, ethyl dimethylaminobenzoate. **Application**: scrub for 20 sec; air-dry for 3 sec; do not rinse.	Etch-and-rinse (ER)	ER.PeU.U	**Ultradent Inspiral Brush Tip (Ultradent Products Inc., USA)** Lot number: BYWBV Composition: brush fibers.
		ER.PeU.Z	**ZerofloX (Medmix, Switzerland)**

Abbreviations: HEMA: 2-Hydroxyethyl methacrylate; PEGDA: Poly(ethylene glycol) diacrylate; BHT: 2,6-di-tert-butyl-p-cresol; MDP: 10-Methacryloyloxydecyl Dihydrogen Phosphate; MMA: methyl methacrylate; DMA: dimethacrylate; MAA: methacrylic acid; BPGMA: Bisphenol A glycidyl methacrylate; UDMA: Urethane dimethacrylate; Bis-EMA: Bisphenol A ethoxylated dimethacrylate; BPA-EDMA: Bisphenol A ethoxylated dimethacrylate.

ER: etch-and-rinse; SE: self-etch; OpU: OptiBond Universal 360; PeU: Peak Universal Bond; K: Kerr Applicators; Z: ZerofloX; A: Aplik; U: Ultradent Inspiral Brush Tip.

Universal adhesive: composition, pH, lot and application protocols as per manufacturer's instructions	Bonding mode	Group	Microapplicator: composition and lot
Peak Universal Bond Adhesive (Ultradent Products Inc., USA) Lot number: BYWBV **Composition**: ethanol, HEMA, glycerol dimethacrylate, methacrylic acid, ethyl 4-dimethylaminobenzoate, organophosphine oxide, chlorhexidine diacetate, BHT, Dymetech Monomer Blend Technology. **Application**: actively apply for 10 sec; aggressively air-thin for 10 sec; light-cure for 10 sec.		ER.PeU.A	**Aplik (Angelus, Brazil)**
	Self-etch (SE)	SE.PeU.U	**Ultradent Black Mini Brush Tip/Ultradent Inspiral Brush Tip (Ultradent Products Inc., USA)**
		SE.PeU.Z	**ZerofloX (Medmix, Switzerland)**
		SE.PeU.A	**Aplik (Angelus, Brazil)**
Ultra-Etch (Ultradent Products Inc., USA). Lot number: BYGWW **Composition**: phosphoric acid solution, polyethylene glycol, dimethicone. **Application**: Apply acid to enamel for 15 sec; Rinse thoroughly, dry, and proceed according to the adhesive manufacturer's instructions.			
Filtek Z250 XT (Solventum, USA). Lot number: 2427600802 **Composition**: 60 wt% fillers, BPGMA, UDMA, Bis-EMA, BPA-EDMA. **Application**: Light-cure for 20 sec.			
Abbreviations: HEMA: 2-Hydroxyethyl methacrylate; PEGDA: Poly(ethylene glycol) diacrylate; BHT: 2,6-di-tert-butyl-p-cresol; MDP: 10-Methacryloyloxydecyl Dihydrogen Phosphate; MMA: methyl methacrylate; DMA: dimethacrylate; MAA: methacrylic acid; BPGMA: Bisphenol A glycidyl methacrylate; UDMA: Urethane dimethacrylate; Bis-EMA: Bisphenol A ethoxylated dimethacrylate; BPA-EDMA: Bisphenol A ethoxylated dimethacrylate.			
ER: etch-and-rinse; SE: self-etch; OpU: OptiBond Universal 360; PeU: Peak Universal Bond; K: Kerr Applicators; Z: ZerofloX; A: Aplik; U: Ultradent Inspiral Brush Tip.			

Two hundred sixteen bovine incisors were selected and stored in an aqueous solution containing 0.5% thymol for a maximum period of 30 days. The teeth were cleaned with Robinson brush and prophylactic paste, and subsequently examined to identify the presence of enamel cracks, fractures, structural anomalies, or caries lesions using a magnifying loupe (ExamVision ApS, Samsø, Denmark) with a magnification of 3.3× under lighting. After root removal, the crowns were embedded in phenolic rings using acrylic resin (Coe Tray Plastic, GC Corp., Tokyo, Japan), ensuring that the buccal surface remained exposed. Silicon carbide (SiC) papers with grit sizes of 200, 400, and 600-grit were used a rotary electric polisher each applied for 30 seconds under continuous water irrigation to obtain a flat buccal enamel surface of approximately 5.0 x 5.0 mm. Once cleaned from griding and polishing residues, a double-sided adhesive vinyl tape (3M Co., St. Paul, USA) with a circular orifice of 2.5 mm in diameter was positioned on the buccal surface to limit the bonding area.

The specimens were randomly assigned into 12 groups (n = 18), according to the UA used (PeU.Peak Universal Bond Adhesive, Ultradent Products Inc. or OpU.OptiBond Universal 360, Kerr Dental), type of micro-applicator (K for Kerr Applicator [Kerr Dental], Z for ZeroflоX [Medmix, Haag, Switzerland], A for Aplik [Angelus, Londrina, Brazil], or U for Ultradent Inspiral Brush Tip [Ultradent Products Inc.]), and bonding mode (SE for self-etch mode or ER for etch-and-rinse mode). Two UAs with different chemical formulations, number of application steps, pH value, and type of solvents (Ethanol for Peak Universal Bond Adhesive [Ultradent Products Inc.] and acetone, ethanol, and water for OptiBond Universal 360 [Kerr Dental]) were applied according to the manufacturers' instructions (Table 1). For half of the groups (ER), the enamel surface was etched with 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc.) for 15 seconds, followed by rinsing with water and air spray for 5 seconds, and gently drying with compressed air applied at approximately 2.0 cm from the enamel surface for an additional 5 seconds. For the other half of the groups (SE), the UAs were applied in self-etching mode. For the PeU groups, the UA primer (Peak SE Primer, Ultradent Products Inc.) was applied with active scrubbing onto the surface for 20 seconds using the proper brush tip (Black Mini Brush Tip, Ultradent Products Inc) followed by air drying for 3 seconds prior to applying the UA (Peak Universal Bond Adhesive, Ultradent Products Inc.) onto the surface for 10 seconds using the UA proper brush tip (Ultradent Inspiral Brush Tip, Ultradent Products Inc.), aggressively air drying, and light-cured for 10 seconds. For the OpU groups, the UA (OptiBond Universal 360, Kerr Dental) was applied with active scrubbing for 20 seconds, air-drying for 5 seconds and light-cured for 10 seconds using a LED light-curing unit (Valo, Ultradent Products Inc.) at an irradiance of approximately 1,200 mW/cm².

In the control groups, the UAs were applied using their respective disposable micro-applicators: brush fibers (U.Ultradent Black Mini Brush Tip and Ultradent Inspiral Brush Tip) for PeU (respectively for Peak SE Primer and Peak Universal Bond Adhesive, Ultradent Products Inc.) or fiber bristles (K.Kerr Applicators, Kerr Dental) for OpU. Additionally, the UAs were applied using two other disposable micro-applicators: one with fiber-free elastomeric bristles (Z.ZeroflоX universal size, Medmix, Switzerland), and another one was a general-brand micro-brush with fiber bristles (A.Aplik regular size, Angelus, Brazil).

Cylinders measuring 2.38 mm in diameter and 2.0 mm in height were built using a micro-hybrid composite resin (Filtek Z250 XT, Solventum, Saint Paul, USA) and bonding mold inserts (Ultradent Bonding Assembly, Ultradent Products Inc.), and light-cured for 20 seconds. The specimens were stored in water for 6 months at 37°C, and subjected to thermal aging (10,000 cycles of 60 seconds, 5 to 55°C) using a thermocycling machine (OMC350TS, Odeme, Luzerna, SC, Brazil). Then, composite resin shear bond strengths to enamel were evaluated using a universal testing machine (Instron 4444, Instron Corp., Norwood, MA, USA), and a linear slide shear contact device (Linear Test Slide, Ultradent Products Inc.). This device was attached to a central plate containing high-precision semicircular notches made of hardened steel, having a diameter of 2.38 mm and a height of 0.4 mm, designed to induce shear stress on the previously prepared composite resin cylinders. Mechanical testing was performed at a constant crosshead speed of 1 mm/min. Shear bond strength (MPa) was calculated by dividing the failure load (Newtons) by the bonded surface area (mm²).

Failure mode

The enamel surfaces were examined to identify the failure mode after testing using optical microscopy at 40× magnification (Leica DM4000 M, Leica Microsystems GmbH, Germany). Failures were classified as CE (cohesive in enamel, failure restricted to enamel), AD (adhesive, failure at the interface between composite resin and enamel), MI (mixed, involving composite resin and the enamel interface), or CC (cohesive in composite resin, failure restricted to the composite resin) [23, 40].

Also, the enamel surfaces were analyzed using a scanning electron microscope (SEM) to confirm the failure mode. For this purpose, five specimens from each experimental group were randomly selected and prepared according to the following procedures (n = 5): the bovine crowns were removed from the phenolic rings/acrylic resin, and subjected to a standardized cleaning and dehydration protocol performed in an ultrasonic bath. For that, they were immersed in ethanol solutions at concentrations of 25%, 50%, and 75% for 15 minutes each, followed by immersion in 95% ethanol for 30 minutes, and finally in absolute ethanol (100%) for 60 minutes.

The specimens were mounted on stubs and gold-sputter coated (SCD 500 EVN, Bal-Tec AG, Liechtenstein). Then, analyzed at magnifications of 75×, 1,000×, 5,000×, and 10,000× under SEM (Tescan Vega, Brno, Czech Republic). In the presence of contaminants resembling micro-applicator fibers or components of the elastomeric micro-applicator, Energy Dispersive X-ray Spectroscopy (EDX) was used to chemically analyze of the surface and characterize the debris were from: micro-applicator, fragments from composite resin, adhesive layer or enamel.

Enamel etching pattern under scanning electron microscopy

Figure 2 shows a schematic flow chart for SEM surface analysis. Specimens of human enamel surfaces were prepared for SEM analysis using procedures similar to previously described. Three extracted human molars free of caries, cracks, or visible structural defects, were obtained from the tooth bank of the Federal University of Santa Catarina (research project number 2723358). The protocol was approved by Universidade Federal de Santa Catarina Research Ethics Committee Involving Human Beings in accordance with the Resolutions of the (Brazilian) National Health Council resolution number 466/2012. The buccal and lingual surfaces of the teeth were subjected to sequential polishing using silicon carbide (SiC) papers with decreasing grit sizes (200, 400, 600, and 1,200-grit) under constant irrigation, using a rotary electric polisher. After polishing, each tooth was sectioned, resulting in 12 specimens (n = 2).

The specimens were then randomly assigned according to the surface treatment applied: (1) enamel polished (control), (2) enamel etched with 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc.) for 15 seconds, (3) enamel treated with PeU (self-etch mode), (4) enamel treated OpU (self-etch mode); (5) enamel etched with 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc.) for 15 seconds followed by PeU; and, (6) enamel etched with 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc.) for 15 seconds followed by application of OpU. Afterward, the specimens were cleaned with 100%

acetone in an ultrasonic bath for 24 hours. The specimens were mounted on stubs and gold-sputter coated and analyzed under SEM (Tescan Vega, Czech Republic) at magnifications of 1,000×, 5,000×, 10,000×, and 20,000×.

Statistical analysis

Bond strength data were initially assessed for normality using the Shapiro–Wilk test and for homoscedasticity using the Bartlett test. After confirming normal distribution and homogeneity of variances, data were analyzed using multivariate ANOVA (Independent variables: universal adhesive, bonding mode, and micro-applicator) and Tukey HSD *post hoc* test ($\alpha = 0.05$).

Results

Shear bond strength to enamel

The mean shear bond strengths [MPa ± (SD)] to enamel are presented in Table 2. Mean composite resin shear bond strengths to enamel ranged from 13.2 (± 3.8) MPa (SE.OpU.Z) to 39.7 (± 9.8) MPa (ER.PeU.A). UAs applied in ER bonding mode showed significantly higher SBS compared to SE mode ($p < 0.001$). PeU resulted in significantly higher enamel SBS values than OpU ($p < 0.01$). For OpU, ZerofloX (Medmix) resulted in significantly lower shear bond strength compared to Kerr Applicator (Kerr Dental) and Aplik (Angelus) ($p < 0.005$). No statistically significant difference was observed among resin composite shear bond strengths to enamel when PeU was applied using the 3 different micro-applicators (Ultradent Inspiral Brush Tip, ZerofloX, or Aplik).

Table 2
Means resin composite shear bond strength to enamel (MPa ± SD) and failure mode

Universal adhesive	Bonding Mode	Group	Mean SBS (MPa ± SD)	Failure Mode (AD/MI/CC/CE)
OptiBond Universal 360 (Kerr Dental)	Each-and-Rinse[a]	ER.OpU.K	38.2 ± 8.9^{A}	8/10/0/0
		ER.OpU.Z	30.4 ± 8.7^{B}	7/11/0/0
		ER.OpU.A	36.5 ± 10.4^{A}	1/17/0/0
	Self-Etch[b]	SE.OpU.K	15.6 ± 5.8^{A}	14/4/0/0
		SE.OpU.Z	13.2 ± 3.8^{B}	16/2/0/0
		SE.OpU.A	18.2 ± 8.2^{A}	13/5/0/0
Peak Universal Bond (Ultradent Products Inc.)	Each-and-Rinse[a]	ER.PeU.U	39.2 ± 9.3^{A}	6/12/0/0
		ER. PeU.Z	38 ± 10.7^{A}	4/14/0/0
		ER.PeU.A	39.7 ± 9.8^{A}	4/14/0/0
	Self-Etch[b]	SE.PeU.U	24.0 ± 12.8^{A}	12/6/0/0
		SE. PeU.Z	26.6 ± 12.6^{A}	10/8/0/0
		SE.PeU.A	21.9 ± 11.8^{A}	8/10/0/0

*Different lowercase letters indicate statistical differences among bonding mode. Different uppercase letters indicate statistical differences among micro-applicator groups within the same bonding mode.

Failure mode

Table 2 shows the failure mode results. Only AD and MI failures were observed (Table 2). MI failures predominated in etch-and-rinse mode groups. Self-etch mode groups showed mainly AD failures.

Scanning electron microscopy analysis allowed adequate characterization of the different failure modes observed in each experimental group (Fig. 3). Figure 3A shows the AD failure mode pattern (75× magnification). Figure 3B shows the MI failure mode pattern. Findings compatible with remnants of micro-applicator fibers (Aplik, Angelus) were observed on the bonding surface as illustrated in Fig. 3C (75× magnification). The same findings were observed in two out of 10 specimens analyzed by SEM in which the Aplik (Angelus) micro-applicator was used (ER.OpU.A). In the ER.OpU.Z group, a degassing phenomenon of volatile components was identified, possibly associated with incomplete polymerization

of the material, evidenced by linear formations (“streaks”) in the SEM micrograph associated with energy dispersive spectroscopy analysis (Fig. 3D).

Enamel etching pattern under scanning electron microscopy

Figure 4 shows the enamel surface SEM analysis. The untreated enamel surface appeared smooth (Fig. 4A). Enamel etched with 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc.) resulted in a classic enamel etching pattern (Fig. 4B). In self-etch mode, PeU resulted in superficial intraprismatic etching (Fig. 4C). OpU applied in self-etch mode resulted in a morphology without defined characteristics (Fig. 4D), similar to that of the untreated enamel in the control group (Fig. 4A). In etch-and-rinse mode, both OpU and PeU showed a classic phosphoric acid etching pattern across the entire surface (Figs. 4E and F).

Discussion

The first null hypothesis was rejected. PeU resulted in higher enamel bond strength than OpU. Previous studies have concluded that composite resin bond strengths to enamel may vary depending on the UA applied [40–43]. The two UAs tested in the present study rely on different strategies to result in stable bond strengths to enamel. Therefore, a high and stable enamel bond strength to enamel was expected with OpU in both bonding modes (self-etch and etch-and-rinse modes) for two main reasons: the presence of 3 solvents and the monomer composition. OpU contains acetone, ethanol and water. On enamel, solvents in adhesive systems may help the evaporation of residual water after rinsing off the phosphoric acid gel etchant [40]. Acetone has a high evaporation pressure compared to ethanol, allowing the solvent to evaporate more easily [44–46]. Also, OpU contains glycerol phosphate dimethacrylate (GPDM) and incorporates the MDP monomer to improve chemical bonding capacity [46, 47]. MDP exhibits high chemical interaction with hydroxyapatite (HPa) in acid-etched enamel, since the acid conditioning step partially exposes HPa, compared to a milder effect in SE mode [50–53]. Thus, the phosphate group of 10-MDP binds to HPa through electrostatic interaction (ionic attraction), ensuring stronger bonds than other adhesive systems without MDP [50–53]. To the best of our knowledge, the scientific literature still lacks a study comparing composite resin bond strengths to enamel using PeU and OpU in both UA bonding modes (self-etch and etch-and-rinse). However, Foly et al [54] compared composite microtensile bond strengths to enamel using eight UAs applied in self-etch mode, including PeU and OpU predecessor (OptiBond Universal, Kerr Dental), which contains GPDM, phosphate dimethacrylate, HEMA and 2 solvents (acetone and ethanol). In that study, PeU resulted in mean composite bond strengths to enamel similar to OptiBond Universal (Kerr Dental) after 6 months of aging (22.2 [± 4.3] and 25.9 [± 4.6] MPa, respectively) [54]. In turn, PeU adhesion mechanism is two-fold: micromechanical entanglement of Peak Universal Bond Adhesive (Ultradent Products Inc.) with the partly decalcified enamel after the Peak SE Primer application (Fig. 4C) and the chemical interaction of functional monomer with the dissolved crystallites of hydroxyapatite [53]. PeU has methacrylic acid

monomer and other acid monomers from the Dymetech Monomer Blend Technology (Ultradent Products Inc.). Ertuğrul et al [53] compared the bond strength of the previous version of PeU (Peak SE Primer + Peak LC Bond, Ultradent Products Inc.) used in SE and ER modes to a single-bottle SE adhesive system (Xeno V, Dentsply Caulk) on fluorotic and non-fluorotic enamel. Peak SE Primer + Peak LC Bond (Ultradent Products Inc.) resulted in higher bond strength to enamel substrates in SE mode compared to Xeno V (Dentsply Caulk) [54]. This finding supports the higher enamel bond strength observed with PeU compared to OpU in the present study. Future clinical evaluations should confirm whether the higher enamel bond strength observed after specimen aging in the present study will result in a high percentage of clinical retention over the years in composite resin restorations with enamel margins.

In the present study, the second null hypothesis was rejected, the mean composite resin bond strengths to enamel for groups in which UAs were applied in ER mode was higher compared to SE mode. This finding is consistent to previous studies demonstrating that enamel acid-etching improves composite bond strengths to enamel when UAs are used [23, 55–60]. In a study conducted by Suzuki et al [58] the means composite shear bond strength to enamel (± SD) were 46.4 (± 5.4) MPa for ER mode and 27.7 (± 3.8) MPa for SE mode using the first UA launched in 2011 (Scotchbond Universal Adhesive, Solventum). Scanning electron microscopy images of the enamel surface (Fig. 4) help explain this finding. In SE mode, OpU resulted in a morphology without defined characteristics and no evidence of dissolution (Fig. 4D), similar to the image of the untreated enamel (Fig. 4A). PeU resulted in superficial intraprismatic etching (Fig. 4C). Neither of the UAs applied in this mode (SE) produced an etching pattern similar to that of the enamel etched with 35% phosphoric acid (Fig. 4B). This finding is consistent with the results of the study by Imai et al [61] who evaluated enamel surfaces treated with UAs under SEM. These authors also observed a more favorable etching pattern for micromechanical retention in ER mode compared to SE [61]. However, the higher, albeit superficial, demineralization produced by PeU may also explain the higher bond strength values observed for PeU compared to OpU when both UAs were applied in SE mode. PeU SE Primer has a pH between 0.9 and 1.5 [62] and can be classified as intermediately strong SE whereas OpU has a pH between 2.0 and 3.0 [63] can be classified as mild SE [6]. Their pH values directly influenced the enamel etching pattern (Figs. 4C and 4D). Perdigão et al [13] compared the etching pattern of ground and ungrounded enamel resulted by the application of six self-etch adhesive systems to that produced by 35% phosphoric acid gel (Scotchbond Etchant Gel, 3M ESPE, USA). One self-etch adhesive system (Adper Prompt L-Pop, 3M ESPE), with a pH between 0.9 and 1.0, showed a well-defined interprismatic etching pattern on the enamel substrates [13]. The other five self-etch adhesive systems, with pH ranging from 1.8 to 2.5, were not able to produce a similar enamel etching pattern compared to phosphoric acid [13]. The observations of the present study are consistent with these previous findings [13], as the enamel demineralization depends on the pH of the adhesive systems. Similarly, Rifane et al [64] evaluated intact and abraded enamel subjected to different surface treatments using Clearfil Universal Bond Quick (Kuraray Noritake, Japan). When this UA was applied to intact enamel in SE mode, without prior phosphoric acid etching or abrasion, it produced minimal enamel etching pattern, with only shallow depressions and a surface roughness similar to that of untreated intact enamel [64]. In the present study, both PeU and OpU applied using ER mode resulted in a classic

phosphoric acid etching pattern across the entire surface (Figs. 4E and 4F) that was similar to enamel acid-etched without UA application (Fig. 4B). Additionally, a higher number of specimens in SE mode groups exhibited adhesive failures compared to ER mode groups, which showed a higher number of mixed failures (Table 2). These failure modes were confirmed by SEM analysis (Figs. 3A and 3B). The higher frequency of adhesive failures in SE groups may be explained by the greater difficulty of UAs in interlocking with enamel when a classic enamel etching pattern is not present, relying primarily on chemical bonding promoted by monomers. This is particularly relevant considering that the present study employed two aging methods, 6 months of water storage plus 10,000 thermocycles.

The third null hypothesis was partially rejected. Regarding different types of disposable micro-applicators, an influence on composite resin bond strengths to enamel was observed only when the ZerofloX (Medmix) micro-applicator was used in combination with OpU in both SE and ER modes. A possible explanation for this effect may be attributed to the interaction between the elastomeric tip of the micro-applicator and the solvents present in this UA (acetone, ethanol, and water). Among the two UAs tested, acetone is present only in OpU. This interaction might be particularly amplified because the UA manufacturer's application protocol includes an active application by scrubbing it onto the enamel surface for 20 seconds. An attempt was made to perform chemical analysis under SEM using Energy Dispersive X-ray Spectroscopy (EDX) in the groups that used the elastomeric micro-applicator (groups ER.OpU.Z and SE.OpU.Z). The analysis was hindered by a degassing phenomenon, observable as streaks or artifacts in the micrograph (Fig. 3D). This effect was observed only in these two groups and indicates the evaporation of volatile components (outgassing), possibly residual moisture or other solvents, even after subjecting the specimens to a rigorous dehydration protocol using sequential concentrations of absolute ethanol for the SEM protocol. It is hypothesized that the interaction between elastomeric components present in the ZerofloX (Medmix) micro-applicator and OpU may have negatively affected the enamel bonding. Acetone has the potential to induce epoxidation of synthetic rubber [65]. Thus, the presence of residual elastomeric chemical components from this micro-applicator on the surface may have reduced composite resin bond strengths to enamel. Such interference could result in incomplete monomer conversion, leaving unreacted compounds or retained solvents within the polymer matrix. Consequently, these volatile components would be released under the high vacuum conditions of the SEM chamber, leading to specimen degassing during EDX analysis. Since there is little published literature to date on the influence of elastomeric applicators on dental adhesion. Future investigations should evaluate the possible interaction between this elastomeric micro-applicator (ZerofloX, Medmix) and other adhesive systems containing acetone, as well as the potential reduction in bond strength to enamel. On the other hand, the manufacturer of the elastomeric micro-applicator highlights several advantages, as described in the ZerofloX Product Technical Manual (Medmix), such as the absence of nylon and glue, which prevents fiber release and helps to maintain a clean surface. Additionally, the manufacturer emphasizes its efficiency and cost-effectiveness, as its bristles do not absorb excess material, allowing a single applicator to be used for multiple teeth, as well as its flexibility and adaptability to different cavity sizes [39]. Indeed, some enamel specimens in the Aplik (Angelus) groups showed remnants on the enamel surface resembling bristle fragments. These remnants were not

observed on any of the 10 specimens analyzed by SEM in the groups using Kerr Applicators (Kerr Dental). Similarly, remnants were not observed on enamel surfaces in groups where UAs were applied using the elastomeric micro-applicator (ZerofloX), or the smooth and long brush-like bristles micro-applicator (Ultradent Inspiral Brush Tip, Ultradent Products Inc.). It is important to note that these small remnants of fragments were observed on isolated areas in only two specimens (Fig. 3C) In addition, their presence did not negatively influence composite resin bond strengths to enamel. Berton et al [38] evaluated the presence of micro-applicator bristles (Microbrush Tube Series Regular 2.0 mm, Blue, Microbrush International, USA) at the adhesive interface of teeth subjected to Class I and II cavity preparations after application of Clearfil SE Bond (Kuraray Noritake, Japan). Residual bristles were observed on all analyzed surfaces [38]. Studies evaluating micro-applicator quality are scarce. But recently, one study evaluated the absorption capacity and wettability of a UA (Scotchbond Universal, Solventum, USA) for several micro-applicator models [66]. The authors also quantified fiber loss after three active applications of 30 seconds of the UA on the internal surface of a plastic crown, aiming to identify a potential contamination factor at the adhesive interface [66]. Five micro-applicators were compared: Cavibrush (FGM), Micro-brush KG (KG Sorensen), Aplik (Angelus), ZerofloX (Medmix), and Points (SDI) [66]. ZerofloX (Medmix) samples showed the best performance, with good absorption and wettability and no visible structural loss [66]. In contrast, the other fiber-based micro-applicators showed good initial absorption and wettability but visually lost several fibers [66]. In other study conducted by Bourgi et al [67], four brands of fiber-based micro-applicators (Angelus, FGM, KG Sorensen, and SDI) were evaluated regarding the quality of their active tips. SDI micro-applicator showed the least deformation, followed by Angelus, KG Sorensen, and FGM micro-applicators [67]. The latter showed lower bristle density and visible spaces between fibers [67]. It is plausible that some micro-applicator bristles may detach, especially during active adhesive system application on the dental substrate. In the present study, possible bristle remnants were identified only for the Aplik (Angelus) micro-applicator (Fig. 3C). Although these remnants were not sufficient to negatively affect composite resin bond strengths to enamel, they may help explain the higher bond strength observed for PeU compared to OpU. Even though no remnants from Kerr Applicator (Kerr Dental) were observed on specimen surfaces, it should be noted that both Kerr Applicator (Kerr Dental) and Aplik (Angelus) micro-applicators are manufactured using similar technology. More recently, Miguel-Calvo et al [68] evaluated residual fiber contamination after active application of a UA using two different fiber-based micro-applicators: Kerr Applicators (Kerr Dental) and Proclinic Applicators (Proclinic Products, Spain). Residual fibers were detected with both applicators, although Kerr Applicators (Kerr Dental) released significantly fewer and shorter fibers than Proclinic Applicators (Proclinic Prod.) [68]. These findings reinforce that fiber detachment may vary according to the micro-applicator design and manufacturer. The smooth and long brush-like bristles of the Ultradent Inspiral Brush Tip (Ultradent Product Inc.) micro-applicator were not observed in the specimens, which may indicate higher resistance to rupture and fragmentation under active scrub-like UA application. With all this in mind, we can conclude that using the micro-applicators provided by the manufacturer of the universal adhesive is recommended.

Conclusion

Both UAs applied in etch-and-rinse mode resulted in higher enamel bond strengths compared to self-etch mode. The fiber-free elastomeric micro-applicator reduced composite resin bond strengths to enamel only for the acetone-containing universal adhesive tested. Remnants from micro-applicator made with fiber bristlers seem to be occasional and do not seem to compromise composite resin bond strengths to enamel.

Declarations

Author Contributions Conceptualization, C.M., G.M.O. and G.C.L.; writing—original draft preparation, C.M., G.M.O. and G.C.L.; specimen preparation and measurement, C.M.; supervision, G.C.L.; resources, C.M., G.M.O. and G.C.L. All authors have read and agreed to the published version of the manuscript.

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Enlightenment Figures 1 and 2 were used using AI.

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Figures

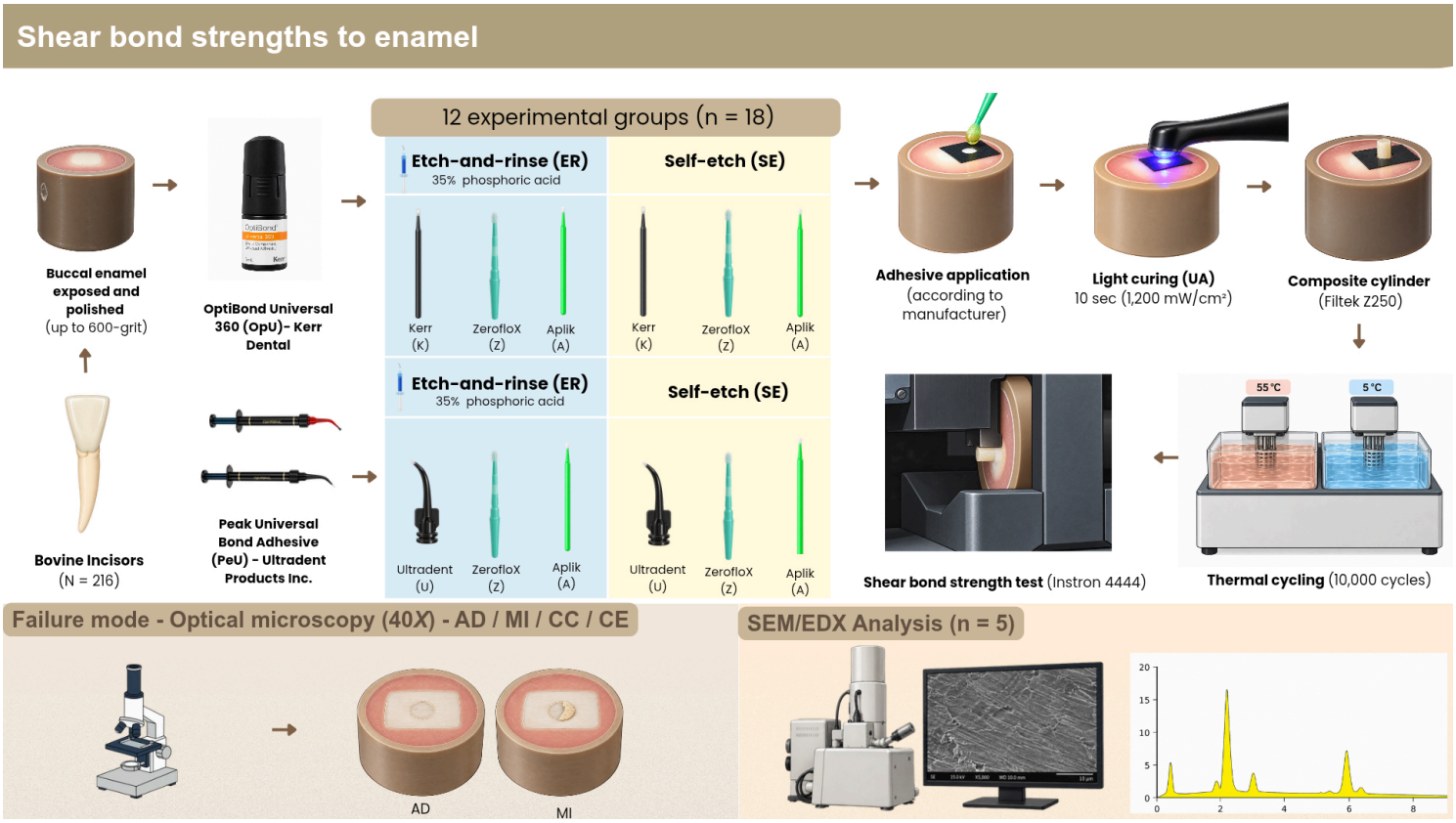


Figure 1

Schematic flow chart for composite resin shear bond strengths to enamel.

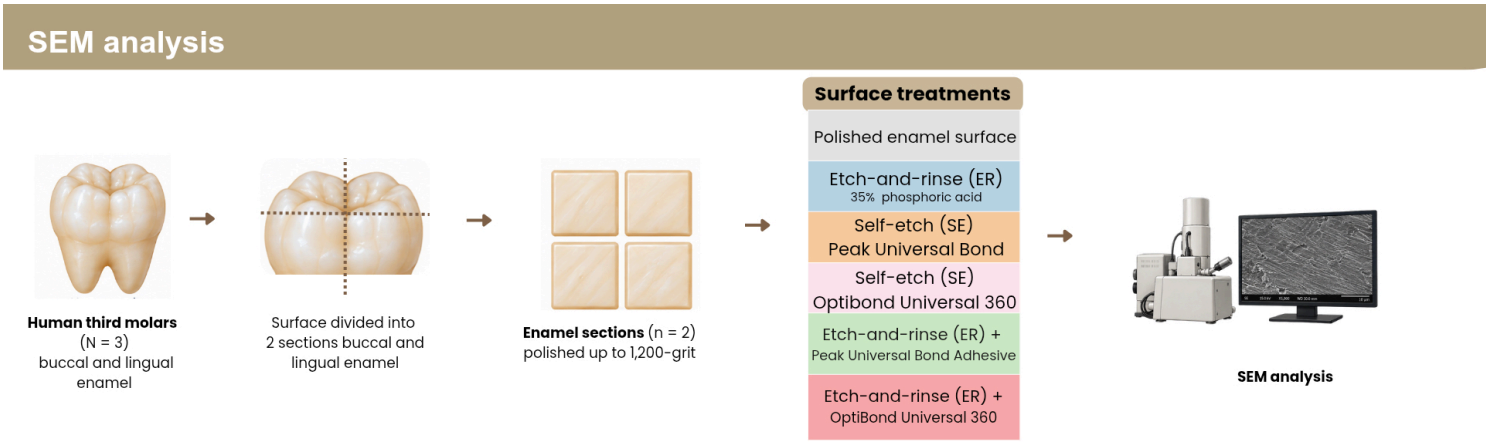


Figure 2

Schematic flow chart for SEM surface analysis and EDX.

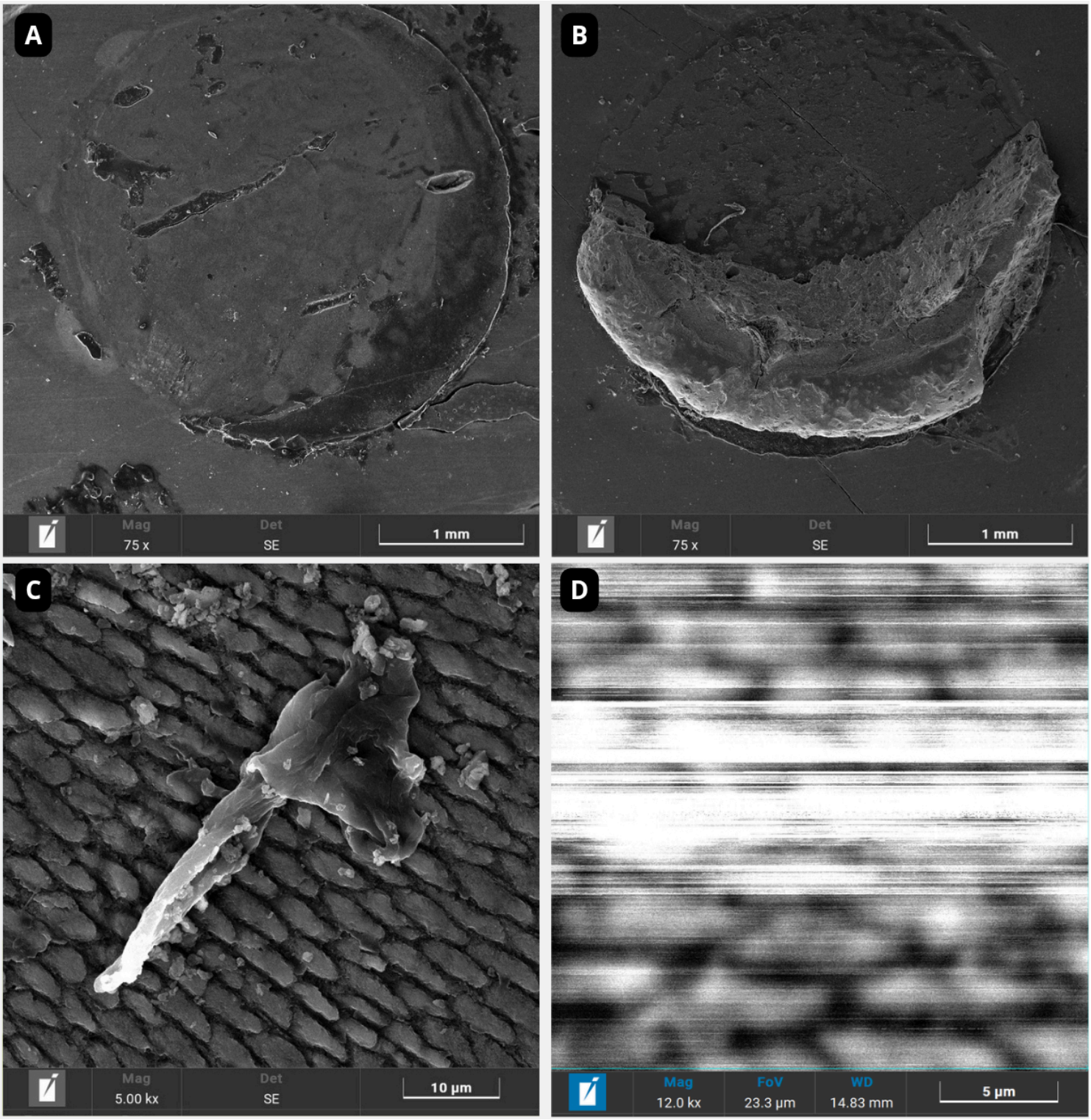


Figure 3

A) shows the adhesive failure mode of SE.PeU.U group, characterized by a predominantly smooth surface, with relatively homogeneous coverage and limited exposure of the enamel prismatic structure, indicating reduced infiltration of the adhesive system (75× magnification). B) for the ER.PeU.A group, shows a mixed failure pattern, with the simultaneous presence of areas covered by composite resin and regions of substrate exposure, as well as irregular edges and a fractured appearance, suggesting a combination of adhesive and cohesive failure (75× magnification). C) SEM micrographs of the enamel

surface from the ER.OpU.A group showing the presence of possible remnants of the Aplik (Angelus) micro-applicator over the surface. An enamel etching pattern is observed, associated with topographical irregularities and residual composite resin with heterogeneous morphology, suggesting micromechanical interaction between the universal adhesive and enamel (10,000× magnification). E) Outgassing of volatile components in the ER.OpU.Z enamel surface specimen, possibly resulting from incomplete polymerization, observed as streaks in the SEM/EDX micrograph.

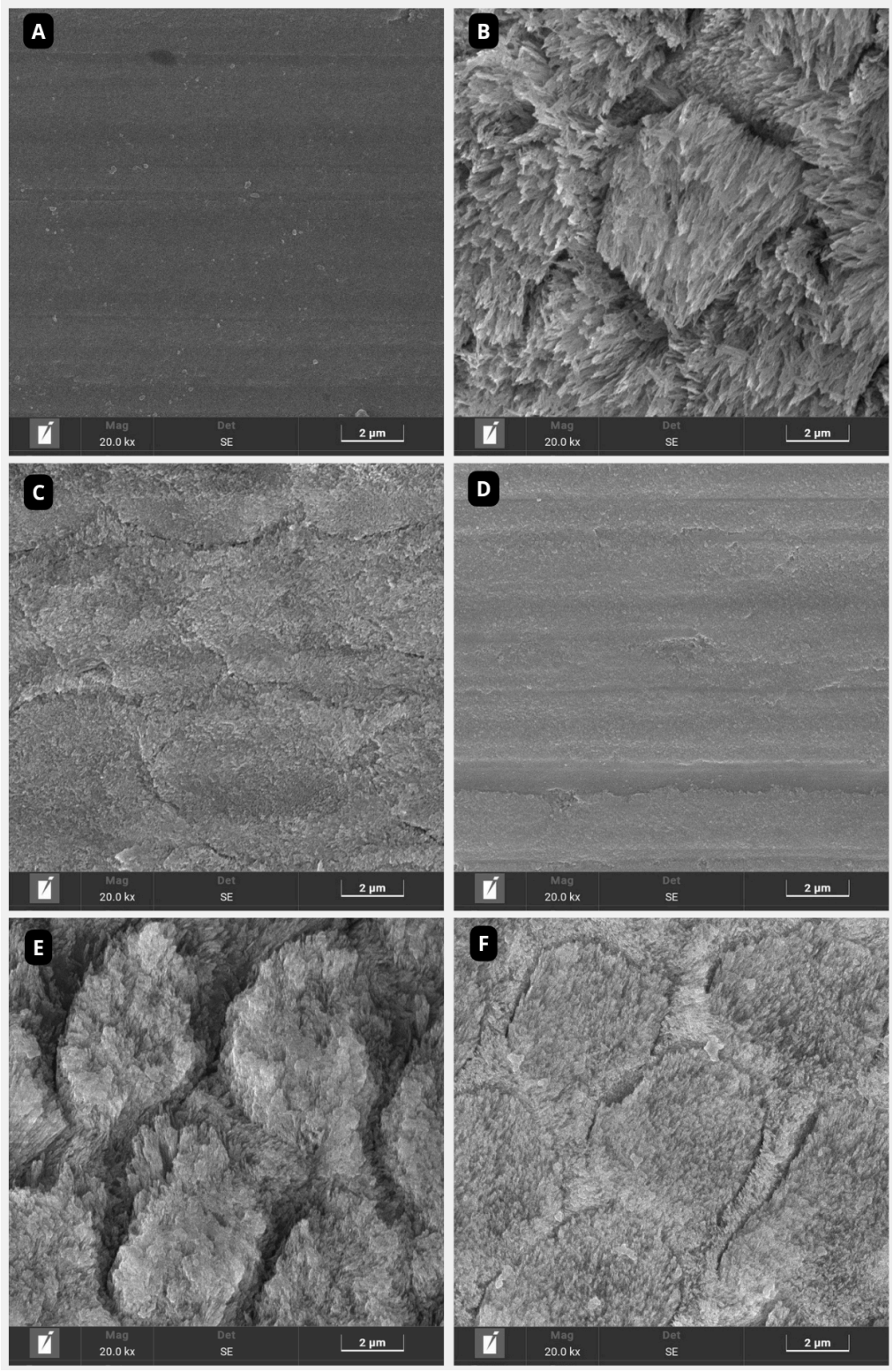


Figure 4

SEM micrographs of enamel surfaces after different treatment protocols, images obtained at 20,000× magnification. A) Polished enamel surface (control), morphology without defined features; B) Enamel surface treated with 35% phosphoric acid gel (Ultra-Etch, Ultradent) for 15 seconds, exhibiting an irregular and microporous surface with a marked increase in surface roughness. Selective removal of the mineral enamel matrix is observed, revealing the prismatic enamel pattern with a honeycomb-like appearance; C) Enamel surface treated with active application of PeU showing a superficial intraprismatic etching pattern with slight demineralization and a mild increase in roughness, without clear evidence of complete prism exposure; D) Enamel surface treated with application of the OpU showing a relatively homogeneous and smooth surface with linear grooves and minimal morphological alteration, indicating low or absent enamel etching capacity; E) Enamel surface treated with 35% phosphoric acid gel (Ultra-Etch, Ultradent Products Inc.) for 15 seconds followed by active application of PeU (Ultradent Products Inc.) showing a more irregular and microporous surface with more pronounced areas of demineralization suggesting higher effective enamel etching; F) Enamel surface treated with 35% phosphoric acid gel (Ultra-Etch, Ultradent) for 15 seconds followed by application of OpU showing a well-defined etching pattern with pronounced exposure of enamel prisms, resulting in a porous and heterogeneous appearance similar to honeycomb structures.