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Influence of Surface Treatments on the Bond Strength of a Self-Adhesive Bulk-Fill Composite Resin to Cut and Uncut Enamel: An In-Vitro Study

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ABSTRACT

Objectives: The objective of this study was to investigate how various surface treatments impact the microshear bond strength (μSBS) of a self-adhesive bulk-fill composite resin to cut and uncut enamel.

Materials and Methods: Eighty sound human third molars were collected in this in vitro study. Forty were prepared to create cut enamel surfaces, and 40 were left uncut. Each group was randomly divided into four subgroups (n=10) according to the type of surface treatment: no treatment (control), 18% ethylenediaminetetraacetic acid (EDTA), All-Bond Universal adhesive in etch-and-rinse (E&R) mode, and All-Bond Universal adhesive in self-etch (SE) mode. Surefil One self-adhesive bulk-fill composite resin was bonded to enamel, and μSBS was measured after 24 hours. Data were analyzed by two-way ANOVA and Tukey's HSD post-hoc test (alpha=0.05).

Results: Two-way ANOVA showed significant main effects of enamel preparation ($P<0.001$) and surface pretreatment ($P<0.001$), as well as their significant interaction effect ($P=0.005$) on μSBS. Cut enamel exhibited a higher mean μSBS (7.12±2.66MPa) than uncut enamel (5.88±2.70MPa; $P=0.019$). Universal adhesive application in E&R mode yielded the highest μSBS, followed by the SE mode; whereas, EDTA and no-treatment groups showed the lowest μSBS values.

Conclusion: Cut enamel samples exhibited a greater μSBS in comparison to uncut samples. The highest μSBS values were observed when both cut and uncut enamel surfaces received pretreatment with a universal adhesive applied in E&R mode.

Keywords: Composite Resins; Dental Enamel; Shear Strength; Smear Layer

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INTRODUCTION

Self-adhesive composite resins were developed to simplify application, save time, and reduce technical sensitivity. Employing these materials in clinical scenarios where isolation is challenging can greatly benefit dentists, helping them to provide long-lasting, high-quality restorations. Development of Surefil One (Dentsply Sirona, Konstanz, Germany), a self-adhesive bulk-fill composite resin, was a recent advancement in restorative dentistry [1]. This material enables efficient, unlimited bulk filling by combining a self-cure initiator system, which activates the curing process without the need for light, with camphorquinone within its pre-measured capsule formulation.

Additionally, its light-curing capability provides a faster and more convenient method for completing the restorative process [1].
In vitro studies have verified that hybrid self-adhesive composite resins exhibit mechanical strength comparable to that of well-established conventional composites used in the clinical settings [2,3]. Furthermore, they have demonstrated comparable or superior wear resistance to more recent self-adhesive restorative materials [2,3]. In a practice-based clinical trial, a self-adhesive resin-based bulk-fill restorative material showed satisfactory short-term outcomes, with service durations up to 12 months [4].
Universal (multi-mode or multi-purpose) adhesive bonding systems represent the most recent generation of adhesives [5]. These bonding agents, although provided in one single bottle, have versatile applications in both etch-and-rinse (E&R) and self-etch (SE) modes [5].
The smear layer, formed during tooth preparation, can influence the bond strength by acting as a physical barrier that may prevent the adhesive from effectively penetrating the dentin and enamel, thus reducing the overall adhesive properties of self-adhesive materials. While the thickness of the smear layer may not affect adhesion with self-etch adhesives, studies indicate lower adhesion with resin-based self-adhesive cements and thicker smear layers [6,7]. Limited research exists on self-adhesive materials, and uncut enamel—despite its clinical relevance—poses bonding challenges due to its aprismatic, highly mineralized layer, which hinders etching and bonding [8].
Sparse data are available on the effect of surface treatments on the bond strength of self-adhesive composite resins. Some studies have explored related aspects, such as ethylenediaminetetraacetic acid (EDTA) or phosphoric acid pretreatment on ground enamel, bond strength of Surefil One to dentin, universal-adhesive preconditioning of self-adhesive restorative materials, and application of SE adhesives on bur-cut and uncut enamel [8-10]. However, direct comparison of microshear bond strength (μSBS) of self-adhesive bulk-fill composites to cut versus intact enamel has not been previously performed. Therefore, the present study aimed to evaluate how diverse surface pretreatments affect the μSBS of a self-adhesive bulk-fill composite resin to cut and uncut enamel surfaces. The null hypothesis was that surface pretreatment would have no significant effect on the bond strength of composite resin.

MATERIALS AND METHODS

This study was approved by the Research and Ethics Committee of Shiraz University of Medical Sciences (Protocol #: IR.SUMS.DENTAL.REC.1401.098). A total of 80 sound third molars, free from caries, fractures, or enamel defects, extracted as part of orthodontic or periodontal treatments, were included. Patients were informed about the study objectives and the use of their extracted teeth, and provided their written informed consent. All procedures were performed by one single trained operator. The extracted teeth were carefully cleaned with a periodontal curette, and stored in 0.5% chloramine T solution at 4°C. The teeth were used within one month of collection.
Table 1 provides a summary of the materials used in this study, including product names, manufacturers, and compositions. All materials were selected based on their relevance to the bonding process and surface treatment of enamel in the context of the study.
Tooth crowns were detached from the roots. Forty teeth were prepared to test the μSBS to bur-cut enamel; while, the remaining 40 were prepared to assess the μSBS to uncut enamel. To prepare the bur-cut specimens, flat enamel surfaces at a depth of 0.5 mm were generated at the mid-buccal region of the tooth crowns using a medium-grit (107 μm) diamond bur (842; Komet, Lemgo, Germany) in a high-speed handpiece under sufficient water cooling. A stereomicroscope (Carl Zeiss, Oberkochen, Germany) was utilized to confirm absence of dentin on enamel surfaces. For the uncut specimens, only the buccal enamel underwent thorough prophylactic cleaning using a rotary dental prophylaxis brush under water irrigation. Next, all specimens were embedded in acrylic resin blocks, with their buccal surface facing upward and parallel to the base of the blocks.

Table 1. Summary of materials used in this study, including product names, manufacturers, and compositions

Material	Product name	Manufacturer	Composition
EDTA	Ultradent™ EDTA 18% Solution	Ultradent Products, Inc.	Ethylenediaminetetraacetic acid, water, nonhazardous additives
Phosphoric Acid Etchant	Total Etch	Ivoclar Vivadent AG, Liechtenstein	37 wt.% phosphoric acid in water, thickening agent, color pigments
Universal Adhesive	All-Bond Universal	Bisco Inc., Schaumburg, IL, USA	Bis-GMA, 10-MDP, HEMA, ethanol, initiators, water
Self-Adhesive Composite Resin	Surefil One	Dentsply Sirona, Konstanz, Germany	Powder: silanated aluminum-phosphor-strontiumsodium-fluoro-silicate glass, dispersed silicon dioxide, ytterbium fluoride, pigments Liquid: acrylic acid, polycarboxylic acid, bifunctional acrylate, self- cure initiator, camphorquinone, stabilizer

The uncut and cut enamel samples were randomly divided into four subgroups (n=10) based on the surface treatment type. The initial subgroup (control) received no treatment before the composite resin bonding. The enamel surfaces in the second subgroup were exposed to 18% EDTA for 60 seconds. The third subgroup underwent etching with phosphoric acid for 15 seconds, followed by thorough rinsing and gentle air-drying. Next, a universal adhesive (All-Bond Universal adhesive) was applied in E&R mode according to the manufacturer's guidelines. In the fourth subgroup, the universal adhesive was applied in SE mode as per the manufacturer's instructions. The adhesive was then cured using a light-curing unit (VIP Junior; Bisco, Schaumburg, IL, USA) with an intensity of 600mW/cm^2.

The bonding area of composite resin to the prepared enamel surfaces was defined using adhesive tape. A translucent polyvinyl chloride microtube (with an internal diameter of 0.7mm and a height of 0.5mm) was placed on the enamel surface, and the self-adhesive bulk-fill composite resin capsule (Surefil OneTM) was activated, mixed for 10 seconds with a capsule mixer as per the manufacturer's instructions, and applied into the microtubes and light-cured.

After bonding, the specimens were stored in distilled water at 37°C for 24 hours. The μSBS testing was then performed using a universal testing machine (Instron Z020; Zwick Roell, Germany) equipped with a 500-N load cell. The bonded composite specimens were loaded using the wire loop technique; accordingly, a thin orthodontic stainless-steel wire (with 0.2mm diameter) was carefully positioned around the cylindrical composite resin at the adhesive interface (Fig. 1). Shear force was applied by pulling the wire in a vertical direction at a crosshead speed of 1mm/minute until bond failure occurred. The μSBS values (in MPa) were calculated by dividing the recorded load at failure in Newtons (N) by the bonding surface area in square-millimeters (mm^2), which was standardized at 0.385mm^2 (based on the 0.7mm internal diameter of the microtube).

After debonding, the enamel-composite interface was examined under a stereomicroscope (Carl Zeiss Inc., Oberkochen, Germany) at ×40 magnification to assess the mode of failure. Each specimen was visually analyzed by two independent examiners to ensure reliability of classifications. In cases of disagreement, a consensus was reached through discussion.

The failure modes were categorized into three types:

Adhesive failure: Fracture occurring at the interface between the composite resin and enamel, indicating inadequate adhesion.

Cohesive failure: Fracture within either the enamel or the composite resin, suggesting that the bond strength exceeded the internal strength of the material.

Mixed failure: A combination of adhesive and cohesive failures, where remnants of composite were observed on the enamel surface alongside areas of clean detachment [11].

The frequency of each failure mode was recorded for all experimental groups.

Fig 1. A micro-cylinder of composite resin (Surefil One) firmly held in place at its base using a stainless-steel ligature wire for measurement of μSBS.

Statistical analysis:

Data normality was confirmed using the Shapiro-Wilk test. Two-way ANOVA analyzed the effects of enamel preparation and surface pretreatment on the μSBS, followed by the Tukey's HSD test for post-hoc comparisons. Failure mode distributions were analyzed using the Chi-square test. All statistical analyses were performed using SPSS version 17.0 (SPSS Inc., Chicago, IL, USA), with a significance level set at $P<0.05$.

RESULTS

Figure 2 shows the mean μSBS values (MPa) and the corresponding standard deviations for the study groups. Two-way ANOVA revealed statistically significant main effects of both enamel preparation (cut or uncut) ($P<0.001$) and surface pretreatment type ($P<0.001$) on μSBS values (Table 2). Moreover, the interaction effect of enamel preparation and surface pretreatment on μSBS was statistically significant ($P=0.005$). Post-hoc analysis showed that cut enamel exhibited a significantly higher μSBS (7.12±2.66MPa) than uncut enamel (5.88±2.70MPa) ($P=0.019$). Among pretreatment groups, universal adhesive applied in E&R mode produced the highest μSBS value compared to all other groups ($P=0.034$), followed by the SE mode, which also showed a significantly higher μSBS than the control and EDTA groups ($P=0.000$). In contrast, the control (no pretreatment) and EDTA-treated groups exhibited the lowest μSBS, both significantly lower than the other pretreatment groups ($P=0.001$ and $P=0.016$, respectively). No statistically significant difference was detected between the control and EDTA-treated groups ($P=0.137$).

Failure modes determined after the μSBS test were classified as adhesive, cohesive, or mixed, with representative examples shown in Figure 3. The distribution of these failure types across all experimental groups is illustrated in Figure 4. Mixed failure was the most commonly observed mode in all subgroups. To determine whether the distribution of failure modes varied significantly among the groups, the Chi-square test of independence was conducted.

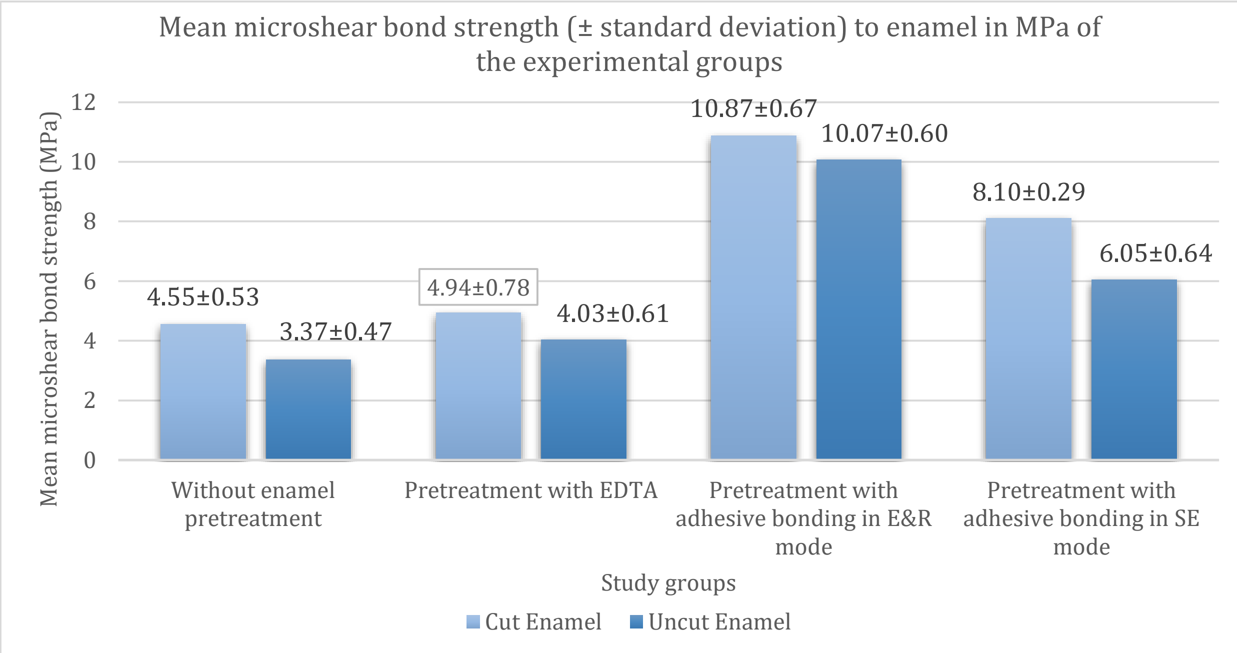

Fig 2. Mean µSBS (MPa) values and standard deviations for each experimental group. (Abbreviations: SE, self-etch; E&R, etch-and-rinse)

Table 2. Results of two-way ANOVA

Source	Type III Sum of Squares	df	Mean square	F	P value
Enamel preparation (cut or uncut)	30.443	1	30.443	86.577	<0.001*
Pretreatment type	532.253	3	177.418	504.561	<0.001*
Enamel preparation × Pretreatment type	4.879	3	1.626	4.625	0.005*
Error	25.317	72	0.352	-	-
Total	3976.403	80	-	-	-

Df: degree of freedom; F: F statistic; Enamel preparation: Cut = enamel surface prepared with a diamond bur; Uncut = intact enamel surface; Pretreatment type: EDTA = 18% ethylene diamine tetra acetic acid; E&R = etch-and-rinse mode; SE = self-etch mode; Control = no surface treatment; Significant at P<0.05

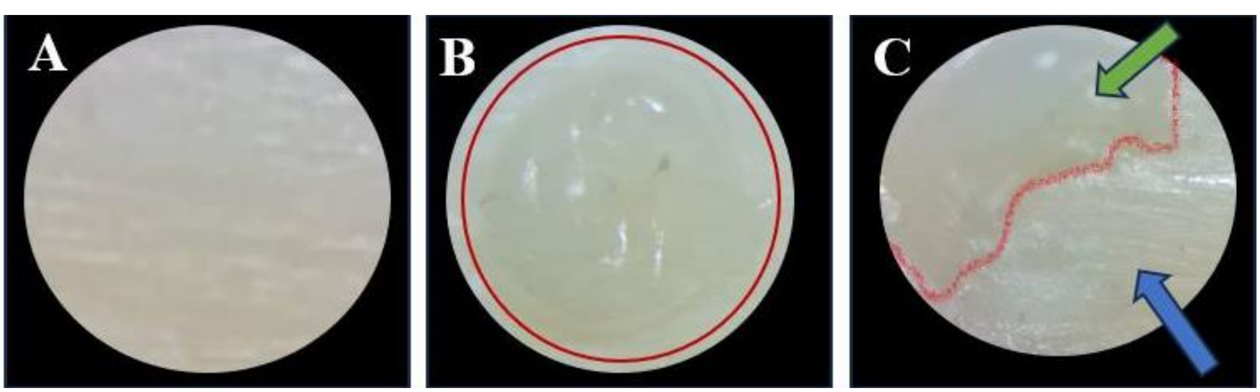

Fig 3. Typical failure patterns determined after the µSBS test using ×40 stereomicroscopy: (A) Adhesive failure at the interface; (B) Cohesive failure confined to the composite resin (Surefil One), with the fracture zone highlighted in red; (C) Mixed failure mode, comprising cohesive failure within the composite resin (Surefil One) (green arrows and red outline) and adhesive separation at the bonding interface (blue arrows).

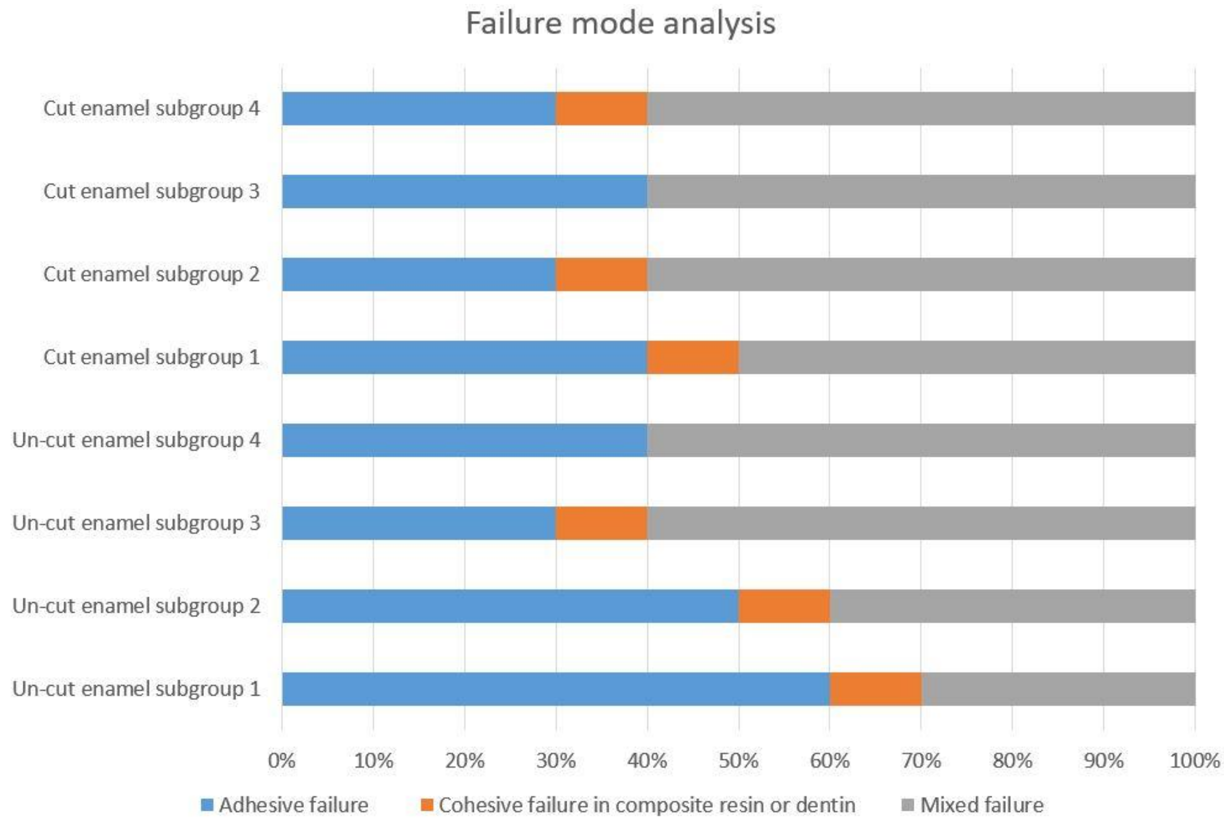

Fig 4. Bar graph showing the frequency of different failure modes recorded across all study groups after the μSBS test

The results showed no statistically significant difference (P=0.971), suggesting that the predominance of mixed failure was consistent across different enamel conditions and subgroups.

DISCUSSION

The present results rejected the null hypothesis, confirming that both enamel surface preparation and type of surface pretreatment significantly affected the μSBS of the self-adhesive bulk-fill composite to enamel. The results of the present study indicated that the μSBS of Surefil One to enamel significantly improved when the universal adhesive was applied beforehand. Specifically, the highest μSBS values were observed when the universal adhesive was used in E&R mode, suggesting that additional surface conditioning is essential for optimizing enamel bonding with Surefil One. Additionally, cut enamel samples showed a significantly higher μSBS than uncut samples, highlighting the beneficial effect of mechanical surface preparation.

There are some previous studies that examined the bond strength of Surefil One to tooth structure under different pretreatment conditions. In a study by Alghamdi et al, [12] the micro-tensile bond strength (μTBS) of Surefil One was evaluated under various dentin conditions, including no treatment, phosphoric acid etching, and universal adhesive application. Interestingly, the highest μTBS was observed in the no-treatment group, with no statistically significant differences among the pretreated groups. This finding contrasts with the current results, where universal adhesive pretreatment in E&R mode significantly increased the μSBS to both cut and uncut enamel. The discrepancy likely arises from differences in substrate (dentin vs. enamel), test methods (μTBS vs. μSBS), and enamel's aprismatic layer, which hinders bonding without etching. Elraggal et al. [9] examined the bond strength of self-adhesive and conventional composite resins to dentin, with and without universal adhesive pretreatment, using SBS and TBS tests. They found that universal adhesive improved the bond strength of self-adhesive composite resin, but the conventional composite resin still showed superior bond strength and marginal adaptation. In contrast, the present study focused on enamel, and only evaluated Surefil One self-adhesive bulk-fill composite resin without comparing it to conventional composite resins, since it was beyond the scope of the present study. The highest bond strength values were observed when enamel, cut or uncut, was pretreated with the universal adhesive in E&R mode. Differences between the results of the two studies may be due to differences in type of substrate (dentin vs. enamel), testing method (shear/tensile vs. microshear), and the specific focus on Surefil One and surface pretreatment rather than comparison of composite resins. Additionally, Aldowsari et al. [13] reported a mean SBS of 17MPa for bonding of Surefil One to silver diamine fluoride-treated dentin without any bonding agent, suggesting its strong inherent adhesion to dentin. In contrast, the present study evaluated the μSBS of Surefil One to enamel and found lower values overall, with the highest value (10.87 MPa for cut and 10.07 MPa for uncut enamel) achieved only when the universal adhesive was applied in E&R mode. These differences likely stem from the distinct bonding characteristics of dentin versus enamel, and the use of different testing methods (shear vs. microshear). Notably, Surefil One appears to bond more effectively to dentin, where the organic content and tubular structure facilitate adhesion, even without pretreatment. In contrast, enamel's mineralized, non-porous nature limits bonding, unless the surface is conditioned. This underscores the importance of substrate-specific protocols when using self-adhesive materials like Surefil One.

Surefil One contains MOPOS, a modified polyacid system that combines the self-adhesive characteristics of glass-ionomer cements with the cross-linking ability of composite resins. This enables chemical adhesion through ionic bonds between its carboxylic groups and calcium in hydroxyapatite, as well as micromechanical retention via smear-layer infiltration and mild surface demineralization [1,14]. Despite Surefil One's intrinsic bonding ability, application of a universal adhesive—especially in E&R mode—significantly enhanced its bond strength to enamel, consistent with studies showing that the E&R mode outperformed the SE mode [10,15]. This improvement stems from phosphoric acid etching, which removes the aprismatic enamel layer, increasing surface roughness and microporosities for deeper resin infiltration [10]. The universal adhesive then forms a strong hybrid layer with enamel and composite, enhancing micromechanical interlocking and chemical bonding. This dual action surpasses Surefil One's limited self-adhesive ability, yielding stronger, more durable enamel adhesion [10]. In contrast, ultra-mild SE adhesives have limited demineralization and weaker enamel bonding [16].

This study used All-Bond Universal, a universal adhesive containing 10-MDP, which partially demineralizes tooth surfaces and forms stable MDP-Ca salt nano-layers with hydroxyapatite, enabling micro-retention and strong ionic bonding [17]. Bond strength, especially in SE adhesive application mode, depends on surface preparation and smear layer properties [18]. This study tested both cut and uncut enamel. Cut enamel was prepared with a medium-grit diamond bur, removing 0.5mm of labial enamel and creating a smear layer over the hypermineralized surface, while uncut enamel was cleaned to preserve intact enamel without a smear layer [8]. Surefil One showed a higher bond strength to cut enamel in the current study, consistent with reports that SE adhesives bond less effectively to intact enamel, likely due to increased surface area and removal of the fluoride-rich aprismatic enamel layer [10]. Although rougher surfaces improve wetting, the enamel smear layer—high in hydroxyapatite and low in organic content—has a strong buffering effect that limits SE adhesive acidity and effectiveness [6]. Mechanical preparation reduces this buffering effect, improving bonding despite the thick smear layer, which ultra-mild adhesives (pH of 3.2) cannot fully demineralize [10,15,16]. Phosphoric acid etching removes the smear layer, creates microporosities, and enables deeper resin infiltration for stronger bonds and a robust hybrid layer [8,10,15]. SE adhesives are generally less effective on enamel than E&R systems, reflected by the highest μSBS values obtained with All-Bond Universal in E&R mode regardless of enamel type [10]. Application of universal adhesive enhances Surefil One's bond strength by improving wetting, promoting chemical bonding, and infiltrating enamel microporosities, while its separate application reduces polymerization shrinkage stress compared to self-adhesive materials [19,20]. Thus, diamond bur roughening, acid etching, and universal adhesive pretreatment are recommended before restoration with Surefil One for optimal enamel bonding [8].

A clinical study reported increased marginal discoloration with a self-adhesive bulk-fill material when selective enamel etching was omitted, highlighting the limitations of simplified procedures [21]. A recent clinical trial also found more marginal staining when universal adhesives were used in SE mode compared to E&R mode for Class II restorations with bulk-fill composites [22]. These findings, along with the present results, support using a universal adhesive in E&R mode as a pretreatment for enamel when applying Surefil One. Additionally, a previous research on dentin bonding showed that thicker smear layers reduce Surefil One's ability to penetrate and bond effectively [7]. Thus, to enhance bonding with self-adhesive bulk-fill composites, it is advised to use fine diamond or carbide burs for final cavity preparation to minimize the smear layer thickness.

Researchers have noted that one-step SE adhesives bond more effectively to ground enamel surfaces due to increased surface roughness, which enhances mechanical anchorage [23,24]. However, conflicting findings exist regarding the effects of ground versus uncut enamel on bond strength. For example, Yazici et al. [24] demonstrated that ground enamel does not always significantly improve the bond strength with one-step SE adhesives. Their performance is more dependent on the adhesive composition and etching aggressiveness, suggesting that adhesive properties may play a more crucial role than surface preparation in certain cases [24]. Furthermore, a more recent study by Jäggi et al, [25] also concluded that bonding effectiveness is not solely dependent on the aggressiveness of the etching process, but also on the specific resin monomers used in the adhesive. This study reported that adhesives containing HEMA and phosphate-based monomers showed stable bond strength even after thermocyclic aging, highlighting the importance of both the etching mode and adhesive chemistry in achieving optimal bonding [25]. Moreover, bond strength values can vary based on the testing method used, and discrepancies in the results compared to prior studies may stem from the μSBS test employed in this study, while previous studies used μTBS testing or shear testing [23-25]. Additionally, variations in adhesive and composite resin composition, as well as differences in mechanical properties, may also contribute to the observed discrepancies.

In this study, EDTA was used to remove the smear layer but did not significantly improve the bond strength of Surefil One. This aligns with previous findings showing no positive effect of EDTA on the bond strength of SE adhesives [8]. Ibrahim et al, [8] also reported that EDTA pretreatment failed to enhance enamel bond strength, likely due to its neutral pH (6.4–7.4), which is insufficient for effective demineralization. Consistent with these results, the current findings suggest that EDTA is not suitable for enamel conditioning prior to SE adhesive application.

This study used μSBS to assess the impact of different surface pretreatments and smear layer thickness on the efficacy of Surefil One self-adhesive composite resin for bonding to cut and uncut enamel surfaces. The μSBS test is a reliable method for evaluation of bond strength, offering efficiency by producing multiple specimens from one single tooth, and minimizing stress distribution issues compared to macroshear tests [26,27]. Unlike macroshear testing, where failures often occur within the tooth substrate, μSBS testing better reflects the performance of adhesive interface [26].

The μSBS values in this study (4–10MPa) were lower than those typically reported for All-Bond Universal used with composite resins [28]. This is the first study assessing All-Bond Universal's bond strength with Surefil One, a self-adhesive bulk-fill composite resin with a modified polyacid system [14]. Surefil One's unique self-adhesive properties may affect adhesive–composite interaction, potentially resulting in lower bond strength values compared to conventional composites bonded with All-Bond Universal. Variations in enamel preparation, specimen handling, and curing protocols may also contribute to these differences.

This study underscored the clinical advantage of enamel pretreatment with a universal adhesive, particularly in E&R mode, which significantly improved the Surefil One's μSBS. For larger or more complex cavities, phosphoric acid etching is recommended to enhance bonding durability and marginal integrity. Future research should explore the Surefil One's bonding performance with different adhesives to clarify factors affecting its bond strength.

The limitations of this study must be noted as well. As an in vitro experiment, its findings may not fully translate to clinical settings, with short-term enamel bond strength tested only after 24 hours. Long-term studies are needed to assess the effects of aging, mechanical stresses, and thermal cycles on bonding performance. Future research should also evaluate oral environmental factors, including functional forces and parafunctional habits. Additionally, the results pertain specifically to the tested universal adhesive system, and may not apply broadly to other adhesives.

CONCLUSION

Cut enamel samples' μSBS was higher than that of uncut samples. The most optimal μSBS values were achieved when cut and uncut enamel surfaces were pretreated with a universal adhesive in E&R mode. Therefore, it is recommended to use an acid etchant to remove the smear layer and apply a universal adhesive when utilizing Surefil One for enamel surface restoration, even when dealing with cut enamel surfaces.

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CONFLICT OF INTEREST STATEMENT

None declared.

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