

## Original Article

# *In vitro* Comparative Effects of Universal, Self-Etch, and Etch-and-Rinse Adhesives on Microshear Bond Strength of Primary Enamel

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## KEY WORDS

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## ABSTRACT

**Background:** Selecting a suitable adhesive to achieve optimal micromechanical retention in the shortest possible time is a challenge in composite restoration of primary teeth.

**Purpose:** This study compared the microshear bond strength ( $\mu$ SBS) of universal, self-etch (SE), and etch-and-rinse (E&R) adhesives to primary enamel.

**Materials and Method:** In this *in vitro* experimental study, the buccal- and lingual-surface enamel of 56 extracted primary molars was wet-polished, and randomly divided into 4 groups (n=14) of (A) Clearfil SE Bond SE adhesive, (B) Scotchbond Multipurpose E&R adhesive, (C) G-Premio Bond universal adhesive with SE mode, and (D) G-Premio Bond Universal adhesive with E&R mode. Composite build-up was then performed, and the specimens underwent thermocycling. The  $\mu$ SBS was measured in a universal testing machine, and the mode of failure was determined stereomicroscopically. One specimen from each group underwent scanning electron microscopic (SEM) assessment. Data were analyzed by one-way ANOVA and Tukey HSD test using SPSS ( $\alpha=0.05$ ).

**Results:** The  $\mu$ SBS of Scotchbond Multipurpose was significantly higher than that of Clearfil SE Bond and G-Premio Bond ( $p$  Value  $<0.001$ ). The  $\mu$ SBS of G-Premio Bond universal adhesive with E&R technique was significantly higher than that with SE technique ( $p=0.001$ ). However, the difference between Clearfil SE Bond and G-Premio Bond in E&R ( $p=0.379$ ) and SE ( $p=0.064$ ) modes was not significant.

**Conclusion:** It appears that Scotchbond Multipurpose three-step E&R adhesive still has the best performance in bonding to primary enamel. G-Premio universal adhesive can yield a  $\mu$ SBS comparable to that of Clearfil SE Bond two-step SE adhesive to enamel.

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## Introduction

Selecting a suitable adhesive that achieves optimal micromechanical bonding to tooth structure in the shortest possible time remains a significant challenge in the composite restoration of primary teeth [1]. Dental adhesive systems fundamentally consist of three components including etchant, primer, and bonding agent [2-4]. Depending on the separate application or combined use of these components, modern bonding agents can be cate-

gorized into three main groups: three-step or two-step etch-and-rinse (E&R) systems, two-step self-etch (SE) systems, and one-step SE systems [2, 5].

Despite their documented effectiveness, E&R adhesive systems are associated with several drawbacks, most notably increased technique sensitivity and a greater number of procedural steps. These limitations may result in a higher risk of clinical errors, prolonged chair time (which is particularly challenging in pediatric

dentistry), marginal discoloration secondary to microleakage, and ultimately, restoration failure [6]. Consequently, pedodontists have shown considerable interest in simplifying adhesive protocols. However, owing to the marked structural differences between enamel and dentin, the simplification of clinical steps in SE adhesive systems may compromise bond strength and increase microleakage [6].

In recent years, significant advancements have been made to improve the performance of adhesive systems by enhancing their chemical and micromechanical interactions with the tooth structure [7-8]. Prior to the introduction of universal adhesives, the concept of chemical bonding was already established through earlier SE systems containing functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). For instance, Clearfil SE Bond (Kuraray Medical Inc., Japan), widely regarded as a gold standard in its category, is a sixth-generation, two-step SE adhesive with a mild self-etching primer ( $\text{pH} \approx 2.0$ ) that establishes a stable chemical bond with hydroxyapatite [9].

Following the patent expiration of the 10-MDP molecule originally held by Kuraray, a new family of adhesive systems with enhanced chemical bonding potential was launched on the market [10]. These adhesives, called multi-mode or universal adhesives, represent the latest generation available and offer exceptional versatility, as they can be used with either E&R or SE protocols [3, 8, 10-12]. Among these, G-Premio Bond (GC Corp., Tokyo, Japan) is a one-bottle universal adhesive applicable in both SE and E&R modes ( $\text{pH} \approx 1.5$ ), offering both procedural versatility and low film thickness [13-14]. To properly evaluate these newer systems, they must be compared against established benchmarks. Adper Scotch Bond Multipurpose (3M Dental Products, St. Paul, MN, USA), a three-step E&R adhesive system known for its reliable dentinal tubule sealing and reduced microleakage, serves as the ideal control, as it is widely considered the gold standard for E&R systems due to its proven clinical success [15].

Current evidence indicates that universal adhesives yield higher bond strength to etched enamel compared to non-etched enamel [16-20]. However, long-term stability remains debated; a previous study showed that bond strength decreased in etched enamel but remained unchanged in non-etched enamel after one year [17].

Conversely, another study has demonstrated comparable shear bond strength (SBS) between universal adhesive systems and traditional two-step or three-step E&R systems [1].

While bond strength to permanent enamel has been extensively investigated [4], crucial differences exist in the chemical composition, physical structure, and micromorphology of permanent and primary teeth that directly affect the efficacy of adhesive systems [21-22]. Primary enamel is thinner, less mineralized, and has lower microhardness. These features increase susceptibility to acid demineralization and may alter both etching patterns and micromechanical retention [21-23]. Despite advancements in adhesive technology and the wealth of evidence regarding permanent teeth, comprehensive studies comparing different generations of adhesive systems in primary teeth remain conspicuously limited.

Considering the scarcity of literature regarding the bond strength of universal adhesives to primary enamel, this study aimed to compare the micro-shear bond strength ( $\mu\text{SBS}$ ) of universal, SE, and E&R adhesive systems applied to primary enamel. The null hypotheses were as follows: (1) there is no significant difference in the  $\mu\text{SBS}$  to primary enamel among the tested adhesive systems; and (2) the application mode of the G-Premio Bond universal adhesive (SE versus E&R) does not affect its  $\mu\text{SBS}$  to primary enamel.

## Materials and Method

This *in vitro* study was conducted on 56 extracted maxillary and mandibular primary molars (ethical approval code: IR.IAU.DENTAL.REC.1399.093). The primary teeth had been extracted for orthodontic reasons or had been naturally exfoliated. Written informed consent was obtained from the participant's legal guardians prior to the use of the samples. Specimens were collected using the convenience sampling method. Teeth with cracks, fractures, caries, hypoplasia, restorations, or structural defects and those extracted more than 6 months earlier were excluded [24-25].

The sample size was calculated to be 12 in each group according to a study by Isolan *et al.* [1] and using one-way ANOVA Power Analysis assuming  $\alpha=0.05$ ,  $\beta=0.2$ , mean standard deviation of 6.2, and effect size of 0.5.

Soft tissue remnants and debris were removed using a scaler (Hu Friedy, USA). The teeth were examined

under a stereomicroscope (Olympus, Tokyo, Japan) at 20× magnification, and intact specimens were selected. caries, crack, wear, or decalcification were selected. They were disinfected in 0.5% chloramine T solution for a week, and stored in distilled water at 4°C until use.

For specimen preparation, buccal and lingual enamel surfaces were wet-polished with 600-grit silicon carbide paper to create a standardized smear layer [1]. The surfaces were re-examined under a stereomicroscope to confirm absence of cracks and defects [26]. Specimens were randomly assigned to four groups (n=14), ensuring equal distribution of buccal and lingual surfaces. Adhesives were applied according to the manufacturers' instructions in all groups. The characteristics of the adhesives are presented in Table 1.

In the Group A, Clearfil SE Bond, a two-step SE adhesive (Kuraray Medical Inc., Japan) was employed. The Primer was first applied on the entire surface for 20 seconds and gently air dried for 5 seconds. The adhesive was subsequently applied, air-thinned with gentle air spray, and light cured for 10 seconds [18].

In the Group B, Adper Scotchbond Multipurpose, a three-step E&R adhesive (3M Dental Products, St. Paul, MN, USA) was used. Enamel was etched with 35% phosphoric acid for 15s, rinsed with air/water spray for 30s, and gently air-dried to obtain a chalky-white enamel appearance. One layer of primer was applied, followed by gentle air-drying for 5 s and application of the adhesive, and it was finally light cured for 10s.

In the Group C, a single-bottle universal adhesive, G-Premio Bond (GC Corp., Tokyo, Japan), was applied in SE mode. The bottle was shaken thoroughly before use. The adhesive was applied to enamel, left undisturbed for 10 s, gently air-dried for 5 s, and light-cured for 10 s.

In the Group D, the same single-bottle universal adhesive (G-Premio Bond; GC Corp., Tokyo, Japan) was applied in E&R mode. Enamel was etched with 35%

phosphoric acid for 15 s, rinsed for 30 s, blot-dried, and the adhesive was applied and light-cured for 10 s [18].

Composite buildup (3M ESPE, St Paul, MN, USA) was performed immediately after the application of bonding agents on the tooth surface by using Tygon tubes with 2 mm height and 0.9 mm internal diameter [27]. Light-curing was performed vertically for 40 seconds using a LED curing unit (Woodpecker, China) with a light intensity of 600 mW/cm<sup>2</sup>. The light intensity (mW) of the curing unit was checked by a radiometer (Dentamerica, LITEX, Taiwan) before each time of use.

All the specimens were stored in distilled water at 37°C for 24 h and subsequently subjected to thermocycling in water baths between 5°C and 55°C with a dwell time of 30 seconds and a transfer time of 10 seconds for 5000 cycles to stimulate oral conditions. For  $\mu$ SBS testing, each specimen was mounted in acrylic resin, and loaded in a universal testing machine (Zwick Roell, Germany) at a crosshead speed of 1 mm/minute until fracture. The fracture load (N) was recorded and converted to  $\mu$ SBS values (MPa) by dividing the load by the bonded cross-sectional area (mm<sup>2</sup>) [28]. To ensure the precision and accuracy of the measurements, the universal testing machine was calibrated according to the manufacturer's guidelines prior to testing. Then the output of the light-curing unit was verified with a radiometer before each use, and all specimens were tested by a single calibrated operator in accordance with ISO 29022:2013. These measures minimized instrument and operator related variability and enhanced the reliability and reproducibility of the recorded  $\mu$ SBS values.

After  $\mu$ SBS testing, the debonded surfaces of all specimens were examined under a stereomicroscope at 40× magnification to determine the failure mode. Failure modes were classified as adhesive, cohesive, or mixed. Adhesive failure was defined as failure occurring primarily at the enamel–adhesive or adhesive–com-

**Table 1:** Characteristics of the adhesives used in this study

| Adhesive type                        | Composition                                                                                                              | Manufacturer                       |
|--------------------------------------|--------------------------------------------------------------------------------------------------------------------------|------------------------------------|
| Clearfil SE Bond SE adhesive         | Primer: 10- MDP, dimethacrylate, HEMA, photo-initiator, water<br>Bond: HEMA,10-MDP, dimethacrylate, microfiller, Bis-GMA | Kuraray, Kurashiki, Okayama, Japan |
| Scotchbond Multipurpose E&R adhesive | Primer: Polyalkenoic acid, copolymer HEMA, water<br>Bond: Bis-GMA, HEMA, tertiary amines, photo-initiator                | 3M ESPE, St. Paul, MN, USA         |
| G-Premio Bond universal adhesive     | MDP, MDTP, 4-MET, photo-initiator, water, acetone, dimethacrylate                                                        | GC, Kasugai, Japan                 |

**Abbreviations:** self-etch (SE); etch-and-rinse (E&R); 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP); 2-hydroxyethyl methacrylate (HEMA); bisphenol A-glycidyl methacrylate (Bis-GMA); methacryloyloxydecyl dihydrogen thiophosphate (MDTP); 4-methacryloyloxyethyl trimellitate (4-MET)

**Table 2:** Measures of central dispersion of the  $\mu$ SBS (MPa) of primary enamel in the four groups

| Group                                        | Mean $\pm$ std. deviation | Minimum | Maximum | p Value* |
|----------------------------------------------|---------------------------|---------|---------|----------|
| Clearfil SE Bond SE adhesive                 | 25.74 $\pm$ 4.52          | 20.53   | 34.09   | <0.001   |
| Scotchbond Multipurpose E&R adhesive         | 36.71 $\pm$ 7.27          | 22.81   | 48.03   |          |
| G-Premio Bond universal adhesive in SE mode  | 19.51 $\pm$ 5.23          | 12.61   | 29.20   |          |
| G-Premio Bond universal adhesive in E&R mode | 29.70 $\pm$ 8.16          | 19.20   | 43.18   |          |

\*One-way ANOVA

**Abbreviations:** Microshear bond strength ( $\mu$ SBS); Megapascal (MPa); Self-etch (SE); Etch-and-rinse (E&R)

posite interface, with no or minimal remnants of composite resin remaining on the enamel surface. Cohesive failure was defined as fracture occurring within either the composite resin or the enamel substrate, while the adhesive interface remained partially or completely intact; cohesive failures were further recorded as cohesive failure in composite or cohesive failure in tooth structure. Mixed failure was defined as a combination of adhesive and cohesive failure, characterized by interfacial debonding together with remnants of composite resin and/or fractured enamel on the bonded surface [25, 29-30].

For scanning electron microscopy (SEM) analysis, one specimen with adhesive failure from each group was randomly selected. Specimens were sectioned perpendicular to the bonded interface and polished with 400, 600, 1000, and 2000 grit silicon carbide papers under water cooling. Surfaces were etched with 37% phosphoric acid (10 s), rinsed (30 s), treated with 5% sodium hypochlorite (2 min), dehydrated in ascending ethanol concentrations (70–99%), sputter-coated with gold, and examined under SEM (S4160; Hitachi, Japan) at 20 kV and 500 $\times$  magnification [31].

Statistical analysis was performed using one-way ANOVA followed by Tukey HSD post-hoc tests in SPSS version 26 ( $\alpha=0.05$ ).

## Results

The highest  $\mu$ SBS of primary molars was recorded in the Scotchbond Multipurpose group, followed by the G-Premio Bond in E&R mode, Clearfil SE Bond SE adhesive, and finally G-Premio Bond universal adhesive in

SE mode. One-way ANOVA revealed a significant difference in this regard among the four groups ( $p < 0.001$ , Table 2).

Pairwise comparisons by the Tukey HSD test showed significantly higher  $\mu$ SBS of Scotchbond Multipurpose than Clearfil SE Bond ( $p < 0.001$ ), G-Premio in SE mode ( $p < 0.001$ ), and G-Premio in E&R mode ( $p = 0.030$ ). The  $\mu$ SBS of G-Premio Bond in E&R mode was significantly higher than that in SE mode ( $p = 0.001$ ). However, the difference in  $\mu$ SBS of Clearfil SE Bond with G-Premio Bond in E&R ( $p = 0.379$ ) and SE ( $p = 0.064$ ) modes was not significant.

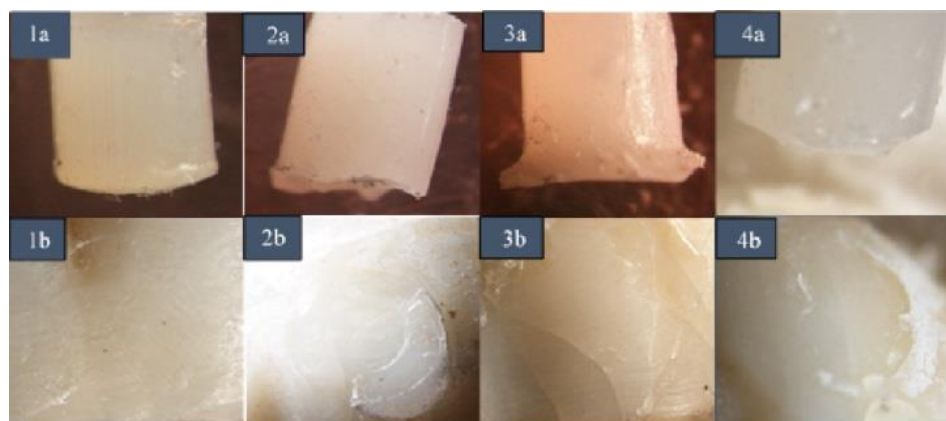
Mixed failure followed by adhesive failure were dominant in the Scotchbond Multipurpose group. In the Clearfil SE Bond and G-Premio Bond groups, adhesive failure had the highest frequency followed by mixed failure. The frequency of cohesive failure in the G-Premio Bond in SE mode group ( $n=1$ ) was lower than that in other groups (Table 3).

Photographs of different failure modes are shown in Figure 1, and SEM micrographs are presented in Figure 2. The debonded surfaces were first evaluated under a stereomicroscope, and then underwent SEM assessment for further evaluation at higher magnification. SEM micrographs confirmed the mode of failure determined stereomicroscopically. SEM micrographs revealed mixed and adhesive failures at the enamel-adhesive interface at a higher magnification with greater clarity. Micrographs of the debonded surfaces in the universal adhesive group revealed higher porosities and grooves compared with the Scotchbond Multipurpose E&R adhesive and Clearfil SE Bond SE adhesive groups.

**Table 3:** Frequency percentage of different failure modes in the four study groups

| Groups                                       | Failure mode | Adhesive  | Cohesive (in composite) | Cohesive (in tooth) | Mixed     |
|----------------------------------------------|--------------|-----------|-------------------------|---------------------|-----------|
| Clearfil SE Bond SE adhesive                 |              | 7(50%)    | 2(14.28%)               | 1(7.14%)            | 4(28.57%) |
| Scotchbond Multipurpose E&R adhesive         |              | 5(35.71%) | 2(14.28%)               | 1(7.14%)            | 6(42.85%) |
| G-Premio Bond universal adhesive in SE mode  |              | 8(57.14%) | 1(7.14%)                | 0(0%)               | 5(35.71%) |
| G-Premio Bond universal adhesive in E&R mode |              | 6(42.85%) | 3(21.42%)               | 0(0%)               | 5(35.71%) |

**Abbreviations:** self-etch (SE); etch-and-rinse (E&R)



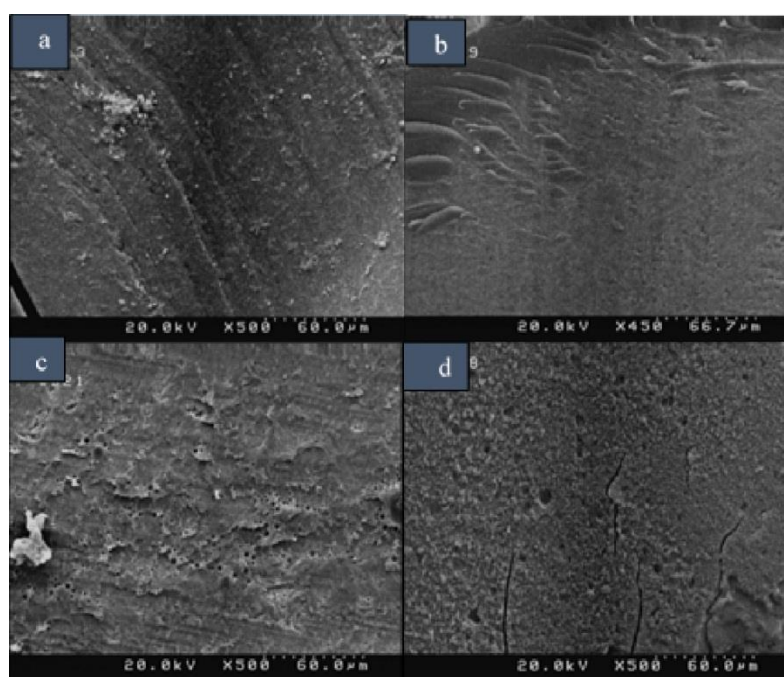
**Figure 1:** **a:** Debonded surface showing adhesive failure; **b:** Cross-section of the same specimen at 40× magnification; (2a) Debonded surface showing cohesive failure in composite resin; (2b) cross-section of the same specimen at 40× magnification; (3a) Debonded surface showing cohesive failure in the enamel; (3b) cross-section of the same specimen at 40× magnification; (4a) Debonded surface showing mixed failure; (4b) cross-section of the same specimen at 40× magnification

## Discussion

The present study compared the effects of G-Premio Bond universal adhesive, Clearfil SE Bond SE adhesive, and Scotchbond Multipurpose E&R adhesive on  $\mu$ SBS of primary molars. The effect of etching on  $\mu$ SBS of G-Premio Bond universal adhesive was also evaluated. The results revealed the highest  $\mu$ SBS in the Scotchbond Multipurpose E&R adhesive group followed by the G-Premio Bond universal adhesive in E&R mode, and Clearfil SE Bond in SE mode; while, the lowest

$\mu$ SBS was recorded in the G-Premio Bond universal adhesive in SE mode. Pairwise comparisons of the groups indicated that the  $\mu$ SBS of the three-step E&R adhesive was significantly higher than that of the SE adhesive and universal adhesive. Based on these findings, both null hypotheses were rejected, as significant differences were found among the adhesive systems, and the application mode (E&R versus SE) significantly influenced the  $\mu$ SBS of the universal adhesive.

Scotchbond Multipurpose belongs to the gold-stand-



**Figure 2:** SEM micrographs illustrating adhesive failure patterns on primary enamel at 500× magnification; **a:** Clearfil SE Bond (SE adhesive) showing a relatively smooth enamel surface; **b:** Scotchbond Multipurpose (E&R adhesive) with more pronounced surface irregularities; **c:** G-Premio Bond universal adhesive applied in self-etch mode showing limited surface roughness; and **d:** G-Premio Bond universal adhesive applied in E&R mode exhibiting increased surface irregularities (**Abbreviations:** Scanning electron microscopy (SEM); Self-etch (SE); Etch-and-rinse (E & R))

ard generation of dental adhesives, which includes three steps of acid etching, followed by priming, and application of a resin bonding agent. The optimal result of Scotchbond Multipurpose may be related to the presence of polyalkenoic acid vitrebond copolymer; vitrebond interacts with the calcium ions in the composition of hydroxyapatite, and increases the interaction of adhesive and enamel. The carboxyl groups of polyalkenoic acid participate in release of protons into aqueous solutions, and acid-base reactions. The carboxyl groups can replace phosphate ions in the substrate, and form ionic bonds to calcium [32].

G-Premio Bond is a universal adhesive with a pH of approximately 1.5 while Clearfil SE Bond is a SE adhesive with a pH of approximately 2. Both of these adhesives have moderate etch-ability [33]. SE adhesives cannot etch the enamel as deep as does the phosphoric acid and therefore, they may yield a lower bond strength. Phosphoric acid etching results in formation of porosities of variable sizes on the enamel surface as the result of dissolution of hydroxyapatite crystals. This process increases the surface area, and allows the resin monomers to penetrate into the enamel, and form prismatic resin tags after polymerization, which increase micromechanical retention [34]. Moreover, acid etching alters the enamel surface and increases its surface energy, polarizing the enamel surface, and enhancing chemical reactions between the enamel and adhesive [18]. Unlike the present study, Jacker-Guhr *et al.* [26] found no significant difference in  $\mu$ SBS of a three-step E&R adhesive and a universal adhesive applied in E&R mode. This difference may be attributed to using different types of universal and E&R adhesives in the two studies.

In the current study, pairwise comparisons of the adhesives revealed equal  $\mu$ SBS of G-Premio universal adhesive and Clearfil SE Bond SE adhesive. Both of these adhesives contain 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) as their acidic monomer, which contains phosphate groups that can form chemical ionic bonds to calcium present in the composition of hydroxyapatite and form a stable calcium-10-MDP stable salt; this may explain the comparable performance of these two adhesives. In addition to the 10-MDP monomer, G-Premio contains another functional monomer i.e., 4-methacryloxyethyl trimellitate. It has been

reported that 4-methacryloxyethyl trimellitate can form strong chemical bonds to calcium-containing substrates [35]. On the other hand, G-Premio adhesive has some drawbacks, compared with Clearfil SE Bond; for instance, one-step SE systems like G-Premio have a hydrophilic nature, which can lead to their water sorption, hydrolysis, and destruction of the adhesive-bonding interface; whereas, in the application of Clearfil SE Bond, which is a two-step SE adhesive, a hydrophobic bonding resin is applied after the application of a hydrophilic primer [24, 36]. Also, similar to other universal adhesives, G-Premio contains water [36]. Although some water is necessarily required for differentiation and ionization of acidic functional monomers, incomplete water evaporation after air drying can result in incomplete polymerization of adhesive, increase hydrolysis after polymerization, and decrease the bond strength. In the current study, the  $\mu$ SBS of G-Premio in SE mode was slightly lower than that of Clearfil SE Bond; however, the  $\mu$ SBS of G-Premio in E&R mode was slightly higher than that of Clearfil SE Bond due to the positive effect of etching on  $\mu$ SBS. In fact, the  $\mu$ SBS of Clearfil SE Bond was somewhere between the  $\mu$ SBS of G-Premio in SE and E&R modes, with no significant difference with any of them.

Jäggi *et al.* [4] evaluated the SBS of five single-bottled universal adhesives in comparison with two two-bottle adhesive systems, specifically identifying Opti-Bond FL (a heavily filled three-step E&R adhesive known for its robust mechanical properties) and G2-Bond Universal (a two-step universal adhesive featuring a distinct hydrophobic bonding layer), to permanent enamel before and after thermocycling for 10,000 thermal cycles. Although they observed no significant difference in shear bond strength between the three-step E&R adhesive and some of the universal adhesives, they explicitly reported that G-Premio Bond exhibited considerably lower bond strength to enamel when used with the etching approach. This specific observation regarding the inferior performance of G-Premio Bond is highly consistent with the findings of the present study. It is noteworthy that this alignment exists despite distinct methodological differences between the two investigations, including the substrate type (permanent versus primary enamel), testing method (macro SBS versus  $\mu$ SBS), and thermocycling duration (10,000 versus 5000

cycles).

The present results revealed that enamel etching prior to the application of G-Premio Bond universal adhesive significantly increased the  $\mu$ SBS, which was in agreement with the findings of previous studies [17-18, 20, 32]. However, some others reported that enamel etching had no significant effect on the bond strength of universal adhesives and did not increase it [1, 37-39]. Demirbuğa *et al.* [40] compared the bond strength of five universal adhesives to enamel in SE and E&R modes. They showed the variable effects of etching on different universal adhesives, such that enamel etching significantly increased the bond strength of Prime and Bond Universal, Single Bond Universal, and All Bond Universal; while, this increase was not significant for Clearfil Universal Bond and Gluma Bond Universal. Therefore, it appears that the effect of etching on bond strength to enamel may vary depending on the type of universal adhesive and its constituents.

Vermelho *et al.* [17] compared the bond strength of a universal adhesive, a SE adhesive, and an E&R adhesive to permanent enamel and dentin, and found significantly higher bond strength of the universal and SE adhesives to etched enamel, which was in line with the present findings. Also, Suzuki *et al.* [20] similar to Demirbuğa *et al.* [40] reported significantly higher SBS of universal adhesives to permanent enamel in E&R mode, compared with SE mode, which was in accordance with the current findings. Although the majority of previous studies that reported an increase in bond strength of universal adhesives when applied in E&R mode have been conducted on permanent teeth, Antoniazzi *et al.* [41] assessed the  $\mu$ SBS of two adhesive systems to primary molar enamel and showed that the bond strength of Scotchbond Universal to etched enamel was higher than that to non-etched enamel. Also, the bond strength of this adhesive in SE mode was similar to the bond strength of Clearfil SE Bond two-step SE adhesive, which was in agreement with the present results.

In the current study, failure mode analysis revealed predominant mixed failures followed by adhesive failures in the Scotchbond Multipurpose E&R group, suggesting stronger interfacial bonds leading to failures involving both the adhesive and substrate. This robust interlocking is fundamentally attributed to the deep penetration of resin tags facilitated by prior phosphoric acid

etching, which shifts the stress concentration away from the pure interface. Whereas adhesive failures followed by mixed failures were more common in the Clearfil SE Bond and G-Premio Bond groups, with cohesive failures being least frequent overall, suggesting weaker micromechanical retention in SE approaches. Because SE primers struggle to deeply demineralize the aprismatic layer of primary enamel, the resulting shallow interface remains the weakest link, failing cleanly before cohesive limits are reached. These findings align with those of Memarpour *et al.* [31] who observed cohesive and mixed failures as dominant in universal adhesives applied in E&R and SE modes to primary dentin, correlating with higher bond strengths and SEM evidence of better resin tag formation in E&R groups. Moreover, El Wakeel *et al.* [42] reported similar patterns in microshear tests of resin-modified glass ionomers to enamel, with mixed failures prevalent in stronger bonds and adhesive failures in weaker ones. However, Antoniazzi *et al.* [41] found predominantly adhesive and mixed failures across all groups, with no significant differences in failure mode distribution between E&R and SE modes when testing Scotchbond Universal and Clearfil SE Bond to primary enamel. This difference may be attributed to differences in the bonded area and stress distribution. In the present study, the microshear test used small cylindrical interfaces that allow more substrate involvement in higher-strength bonds, whereas the microtensile method in their study concentrated stress at the interface, resulting in predominantly adhesive failures regardless of adhesive strategy.

Kim *et al.* [43] evaluated MDP-containing adhesive systems on primary enamel and dentin after 5000 thermocycles and reported that mixed failure was the most frequent failure mode, whereas cohesive failures were uncommon. This is partly consistent with the present findings, in which mixed failure was most frequent in the Scotchbond Multipurpose E&R group, which also showed the highest  $\mu$ SBS. However, adhesive failure predominated in the Clearfil SE Bond and G-Premio Bond groups, especially G-Premio Bond in SE mode. This pattern further confirms that when enamel is treated with SE or simplified universal adhesive strategies, the enamel-adhesive interface lacks sufficient micromechanical depth and may remain the weakest region. In contrast, the higher proportion of mixed failures in

the Scotchbond Multipurpose group may reflect the definitive micromechanical interlocking produced by phosphoric-acid etching. Assunção *et al.* [30] reported no significant difference in failure-mode distribution among adhesive systems tested on sound and eroded primary enamel/dentin, indicating that failure mode may not always parallel bond-strength values. Therefore, while failure modes provide valuable insights into interfacial behavior, discrepancies among studies comprehensively highlight that these outcomes are deeply related to adhesive composition, substrate type (permanent vs. primary), bonded area, loading method, and thermocycling protocol.

The  $\mu$ SBS test was selected for this study due to its use of small bonded areas, which results in more uniform stress distribution, fewer specimen defects, reduced cohesive failures in the substrate or composite, and a higher rate of adhesive failures at the interface. This enhances the reliability of assessing true interfacial bond strength, particularly for brittle substrates such as enamel [44-45].

Regarding the reliability of the current findings, the precision and accuracy of the measurement instruments play a critical role in  $\mu$ SBS evaluations. Given the minute bonded surface areas (approximately 1 mm<sup>2</sup>) involved in  $\mu$ SBS testing, even minor instrumental deviations or calibration errors can significantly skew the calculated stress values. In this study, the use of a rigorously calibrated universal testing machine in strict accordance with ISO 29022:2013 guidelines ensured high sensitivity and accuracy in fracture load recording. By minimizing equipment-related variability and operator-induced errors, the observed differences in bond strength among the E&R, SE, and universal adhesive systems can be confidently attributed to their distinct chemical and micromechanical interactions with the primary enamel, rather than methodological artifacts.

The values obtained in this *in vitro* study may not precisely reflect the clinical performance of these dental adhesives due to the effects of saliva, masticatory forces, and pH alterations in the oral environment. Therefore, clinical studies are recommended to verify the present findings. Further studies are also required on other physical properties of adhesives such as their bond strength to dentin and microleakage. Also, the effect of prolonged water storage and higher frequency of ther-

mal cycles on bond strength should be investigated.

## Conclusion

It appears that Scotchbond Multipurpose three-step E&R adhesive still has the best performance in bonding to primary enamel. G-Premio universal adhesive can yield a  $\mu$ SBS comparable to that of Clearfil SE Bond two-step SE adhesive to enamel.

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None

## Conflict of Interest

There is no conflict of interest to declare.

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