# Enamel and Dentin Bond Durability of Self-Adhesive Restorative Materials

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**Purpose:** To use shear bond strength (SBS) and shear fatigue strength (SFS) testing to determine the durability of adhesion of self-adhesive restorative materials compared to composite resin bonded with a universal adhesive.

**Materials and Methods:** A universal adhesive, Prime & Bond Active, was used in self-etch mode to bond Z-100 composite resin to enamel and dentin. Three commercially available restorative materials and one experimental material with self-adhesive properties, Activa (A), Fuji II LC(F), and Equia Forte (E) and ASAR-MP4 (S) were also bonded to enamel and dentin. The SBS and SFS were determined for all materials. A staircase method was used to determine the SFS with 10 Hz frequency for 50,000 cycles or until failure occurred.

**Results:** On enamel, S generated similar values to the adhesive/composite materials and higher values than F, E, and A. On dentin, the composite/universal adhesive showed significantly higher SBS and SFS than the self-adhesive materials. S, F, and E generated higher values than A on dentin.

**Conclusion:** SBS and SFS values to enamel were similar for all materials tested except Activa which generated lower enamel values. On dentin surfaces, the self-adhesive materials generated similar SBS and SFS, with the exception of Activa. Those values were lower than that generated with composite resin and a universal adhesive.

**Keywords:** adhesion to dental hard tissues, fatigue testing, glass ionomers, bond durability, self-adhesive restoratives.

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Tooth-colored restorative materials are increasingly used for the repair of teeth damaged by caries.[4] The use of these materials affords the operator a more conservative clinical technique by allowing more tooth structure to be preserved and are better accepted by patients, as these materials provide a better shade and translucency match to natural teeth.[4] Bonding of tooth-colored materials to mineralized tooth structure can be accomplished by mediating the adhesive interface with a dental adhesive or by employing a "self-adhesive" restorative material, such as a glass ionomer, resin-modified glass ionomer or self-adhesive resin composite. Recent trends in adhesives have resulted in streamlined systems termed "universal adhesives," which can be employed in either etch-and-rinse self-etch mode or selective etch mode.[13] The selective enamel-etch technique made possible with these systems might be considered the best solution providing high bonding performance to both enamel and dentin.[5] However, the reality of some clinical situations may not allow sufficient time to navigate the enamel conditioning procedure (etch-and-rinse) without risking contamination of the bonding interface.

Both self-etching adhesives and self-adhesive restoratives contain acidic monomers and thus are able to solubilize the smear layer and underlying mineral component of enamel and dentin. This interaction produces micromechanical interlocking to promote bonding. In addition, self-etching adhesives can promote chemical bonding because they contain functional acidic monomers containing carboxylic, phosphonic or phosphate groups that facilitate chemical interaction with mineral apatite. Self-adhesive materials such as glass ionomers can also create ionic bonding between the polyacid molecules and calcium component of the enamel and dentin substrates.[26,27]

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**Table 1** Universal adhesive materials

| Adhesive | Manufacturer | Main components | Code |
|---|---|---|---|
| Prime & Bond active<br>Lot: 1712000006 | Dentsply Sirona;<br>Konstanz, Germany | Phosphoric acid modified acrylate resin, multifunctional acrylate, bifunctional acrylate, acidic acrylate, isopropanol, water, initiator, stabilizer | PB active |
| Resin composite | | | |
| Z100 Restorative<br>Lot No. N416713 | 3M Oral Care;<br>St Paul, MN, USA | Zirconia/silica, 0.01–3.5 µm<br>Filler load: 84.5% weight/66% volume matrix of this composite is a resin consisting of bis-GMA (bisphenol A diglycidyl ether dimethacrylate) and TEG-DMA (tri-ethylene glycol dimethacrylate) 2-benzotriazolyl-4-methyphenol 2 | |

**Table 2** Self-adhesive restorative materials

| Restorative | Manufacturer | Main components | Code |
|---|---|---|---|
| Experimental Material ASAR-MP4<br>Lot No. 1711004202 | Dentsply Sirona; Konstanz, Germany | Aluminum-phosphor-strontium-sodium-fluoro-silicate glass, water, highly dispersed silicon dioxide, acrylic acid, polycarboxylic acid, ytterbium fluoride, bifunctional acrylate, self-cure initiator, iron oxide pigments, barium sulfate pigment, manganese pigment, camphorquinone (photoinitiator), stabilizer | S |
| Fuji II LC<br>Lot No. 1707132 | GC; Tokyo, Japan | Fluoro-alumino-silicate glass, water, polyacrylic acid, HEMA, urethane dimethacrylate | F |
| Equia Forte<br>Lot No. 170807A | GC | Fluoro-alumino-silicate glass, water, polyacrylic acid, polybasic carboxylic acid, camphorquinone (photoinitiator) | E |
| Activa<br>Lot No. 171102 | Pulpdent; Watertown, MA, USA | Bioactive glass, silica, diurethane modified with hydrogenated polybutadiene, methacrylate monomers, modified polyacrylic acid, sodiumfluoride, camphorquinone (photoinitiator) | A |

Glass-ionomer cements, resin-modified glass ionomers, and self-adhesive resin composite materials are less technique sensitive than resin composite and resin adhesives in the presence of moisture in the cavity. While excessive moisture is contraindicated for adhesively bonded composite resins, self-adhesive materials may provide adequate adhesion to mineralized tooth structure in clinical situations where moisture control and isolation are difficult.[7] Unfortunately, there has been a relatively limited number of investigations on the adhesive performance of these self-adhesive materials.

The purpose of this in vitro study was to investigate the enamel and dentin bonding efficacy of four self-adhesive restorative materials compared to composite resin in combination with a "universal" adhesive. The null hypotheses tested were 1) there are no differences in bond durability to enamel among the materials tested and 2) there are no differences in bond durability to dentin among the materials tested.

## MATERIALS AND METHODS

### Study Materials

The composite and universal adhesive used in this study are shown in Table 1. The universal adhesive was used in a self-etch mode: Prime & Bond Active (PB active, Dentsply Sirona; Konstanz, Germany). The resin composite used was Z100 Restorative (3M Oral Care; St Paul, MN USA). The self-adhesive materials used in this study are shown in Table 2. These materials included 1. Fuji II LC (F, GC; Tokyo, Japan); 2. Equia Forte (E, GC); 3. Activa (A, Pulpdent; Watertown, MA, USA); and 4. an experimental material coded ASAR-MP4 (Dentsply Sirona).

### Specimen Preparation

Extracted human 3rd molars were randomly assigned to each test group. The bonding sites were prepared by sectioning the teeth mediodistally, and then removing approximately two-thirds of the apical root structure. The buccal and lingual tooth sections were mounted with dual-curing acrylic resin (Triad DuaLine, Dentsply Sirona) in 25-mm-diameter brass rings. The enamel and dentin bonding surfaces were ground flat to 4000-grit under water cooling and a sequence of carbide polishing papers (Struers; Cleveland, OH, USA). A 4000-grit surface minimizes the thickness of the smear layer on enamel and dentin and provides the best opportunity for self-etch and self-adhesive materials to bond to the substrate. Each enamel surface was evaluated using magnification to assure that the ground enamel sur-

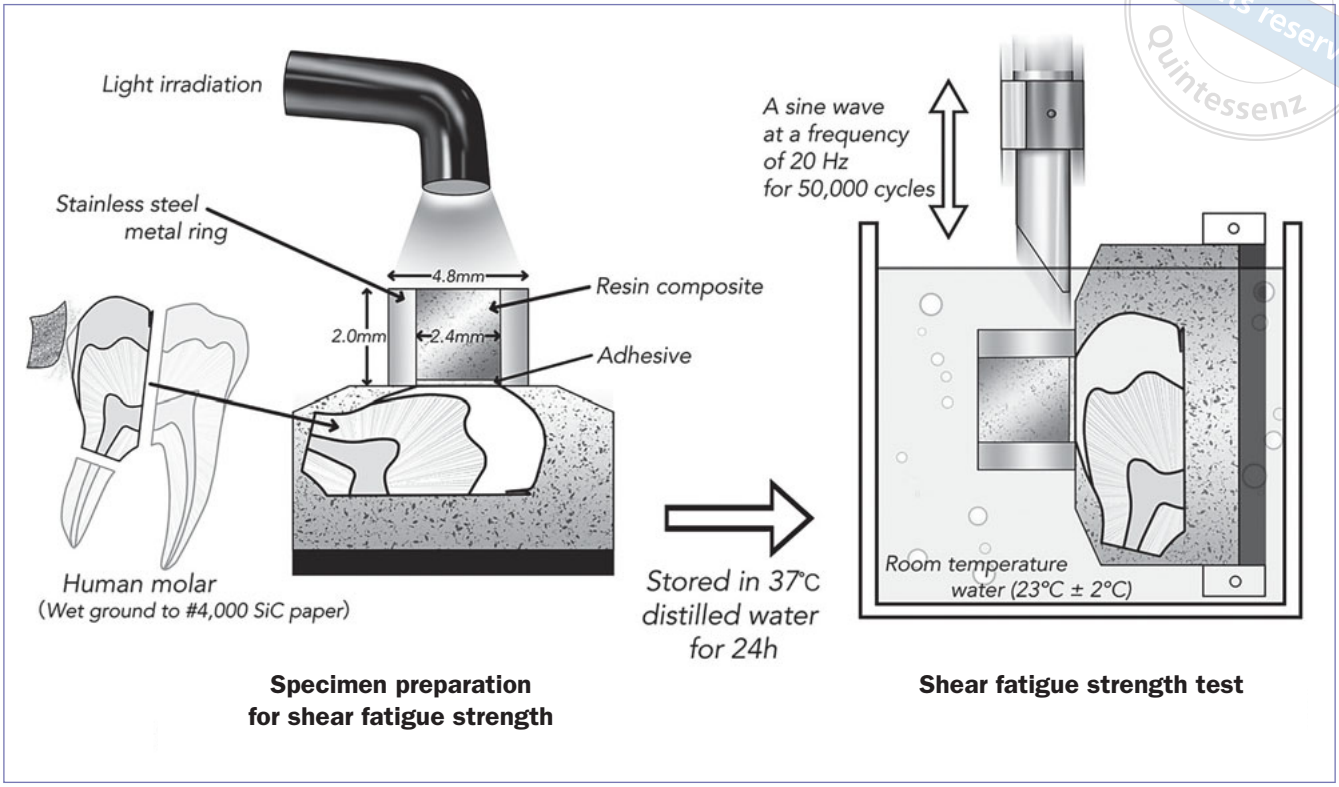


**Fig 1** Schematic illustration of the experimental model for shear bond testing and shear fatigue strength testing.

faces were intact and did not expose dentin. Using a 4000-grit final surface finish minimizes the smear layer thickness and reduces the influence of the smear layer on shear bond strength and shear fatigue strength.[22] Maintaining this surface finish also allows comparison to previous studies employing the same bonding and fatigue models. Stainless steel metal rings machined with an inner diameter of 2.4 mm, an outer diameter of 4.8 mm, and a height of 2.0 mm were used to confine resin composite on enamel and dentin surfaces for shear bond strength (SBS) and shear fatigue strength (SFS) tests. The resin composite cylinder inside the ring was approximately 2.36 mm in diameter and 2.0 mm in height. The rings were left in place during the tests.

### Shear Bond Strength Tests (SBS)

Twelve specimens each were used to determine the SBS to enamel and dentin. For the universal adhesive test group and both substrates, specimens were prepared without phosphoric acid pre-treatment (self-etch technique). Following the treatment of the flat-ground enamel and dentin surfaces with the adhesive agent, the metal rings were positioned over the bonding site and secured in place by clamping in a custom fixture. Z100 restorative resin composite was placed in one increment in the rings and polymerized for 40 s with a SmartLite Focus I (Dentsply Sirona) LED curing unit. The polymerization unit was evaluated using a radiometer before each group of specimens was cured. The output was measured at 1100 mW/cm$^2$. The tip of the light guide was positioned 1.5 mm above the top surface of the metal ring using a custom spacer. No surface conditioning or adhesive agent was used for the self-adhesive restorative materials. Following positioning of the bonding apparatus and metal ring, the restoratives were mixed for 10 s in a ProMix 2 mixing (Dentsply Sirona) device and placed directly in one increment onto the tooth substrate inside the metal ring. In the light-cured groups S, A, and F, the materials were allowed to self-cure at room temperature for 1 min to facilitate penetration and interaction with the substrate surface. The specimens in the light-cured groups were polymerized for 30 s each to assure a complete cure. For self-cured E and S, the specimens were allowed to self-cure for 6 min at room temperature. Following the curing protocols, the specimens were removed from the bonding apparatus, and the bonded specimens were stored for 24 h in distilled water at 37°C before testing.

The specimens were loaded to failure at a crosshead speed of 1.0 mm/min using an MTS Insight machine and TestWorks 4 software (MTS Systems; Eden Prairie, MN, USA). A metal rod with a chisel-shaped end was used to apply the load to the metal ring immediately adjacent to the flat-ground tooth surface. The shear bond strengths (MPa) were calculated from the peak load at failure divided by the bonded surface area.

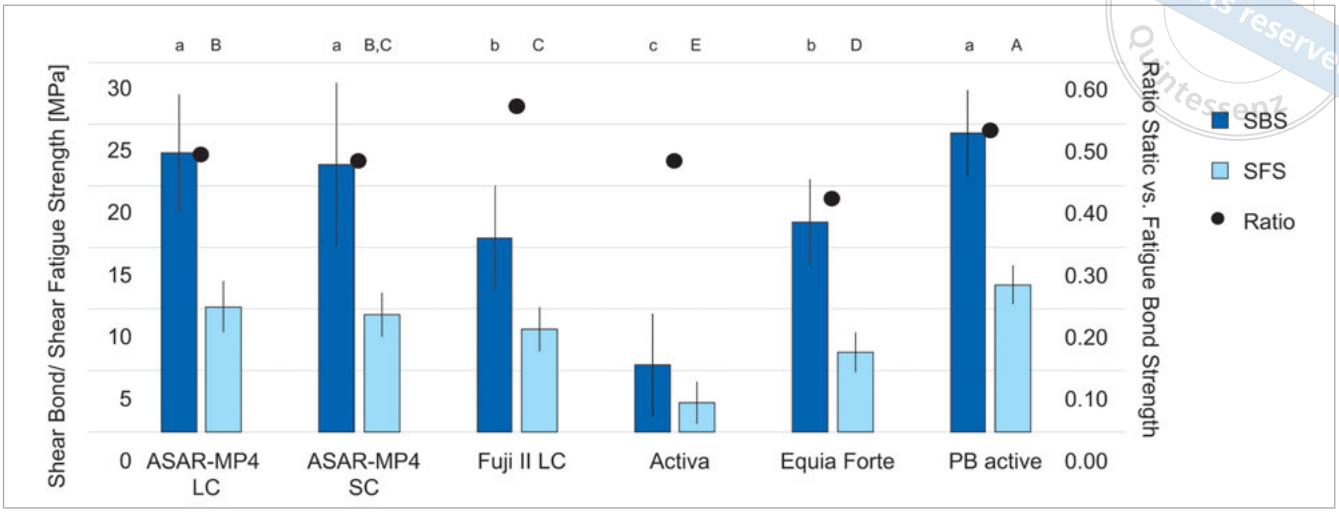


**Fig 2** Results for shear bond strength (SBS) and shear fatigue strength (SFS) to enamel. SBS groups marked with the same small letter were statistically similar ($p > 0.05$). SFS groups marked with the same capital letter were statistically similar ($p > 0.05$). The ratio of SFS to SBS was calculated by dividing the mean SFS by the mean SBS for each material. LC: light cured; SC: self-cured.

### Shear Fatigue Strength Testing (SFS)

The staircase method of fatigue testing introduced by Draughn[6] was used for SFS testing. Test specimens were made as described above for the SBS testing. The lower load limit was set near zero (0.4 N) and the initial maximum load applied was 50%–60% of the SBS previously determined for each of the adhesives tested. The load was applied at a frequency of 10 Hz with an ElectroPuls E1000 machine (Instron; Norwood, MA, USA) using a sine wave for 50,000 cycles or until failure occurred. The load was incrementally adjusted upward or downward (depending on survival or failure) by approximately 10% of the initial load[7,23] (Fig 1). For each test condition, 16 specimens were used to determine the SFS. The test specimens were immersed in room temperature water (23 ± 2°C) to minimize the influence of temperature increases on the bonded specimens. The mean shear fatigue strength (X) and standard deviation (S) were calculated using the formula below.[6]

$$X = X_0 + d\left(\frac{A}{N} - \frac{1}{2}\right)$$

$$S = 1.62\,d\left(\frac{NB - A^2}{N} + 0.029\right)$$

$$N = \Sigma n_i,\ A = \Sigma i n_i,\ B = \Sigma i^2 n_i$$

### Statistical Analysis

A one-way ANOVA followed by Tukey's significant difference (HSD) test ($\alpha = 0.05$) were used for analysis of the SBS data. A modified t-test with Bonferroni correction using a custom program in Excel was used for the SFS data.

## RESULTS

### SBS and SFS to Enamel

The results of the SBS and SFS to enamel for the materials tested are shown in Fig 2 and Table 3. PB-active generated SBS similar to those of S in both curing modes ($p > 0.05$). In both curing modes, S generated higher SBS ($p < 0.05$) compared to the other self-adhesive restorative materials. The universal adhesive generated the highest SFS, with S(LC) generating the highest values on enamel for fatigue of all the self-adhesive materials. The ratio of SFS to SBS was highest for F and lowest for E.

### SBS and SFS to Dentin

The results of the SBS and SFS to dentin for the materials tested are shown in Fig 3 and Table 4. The universal adhesive generated higher SBS and SFS compared to the self-adhesive restorative materials ($p < 0.05$). S in both curing modes as well as E and F generated similar values for SBS ($p > 0.05$). All of the materials generated higher SBS compared to A. F and S(LC) generated the highest SFS among the self-adhesive restorative materials. The ratio of SFS to SBS for F and S in both curing modes was similar to the ratio generated by the universal adhesive.

All debonded enamel and dentin surfaces were visually inspected along with the debonded assemblies following both tests. No evidence of cohesive failure in the substrate or restorative materials was noted, thus macroscopically characterizing the failure mode as adhesive.

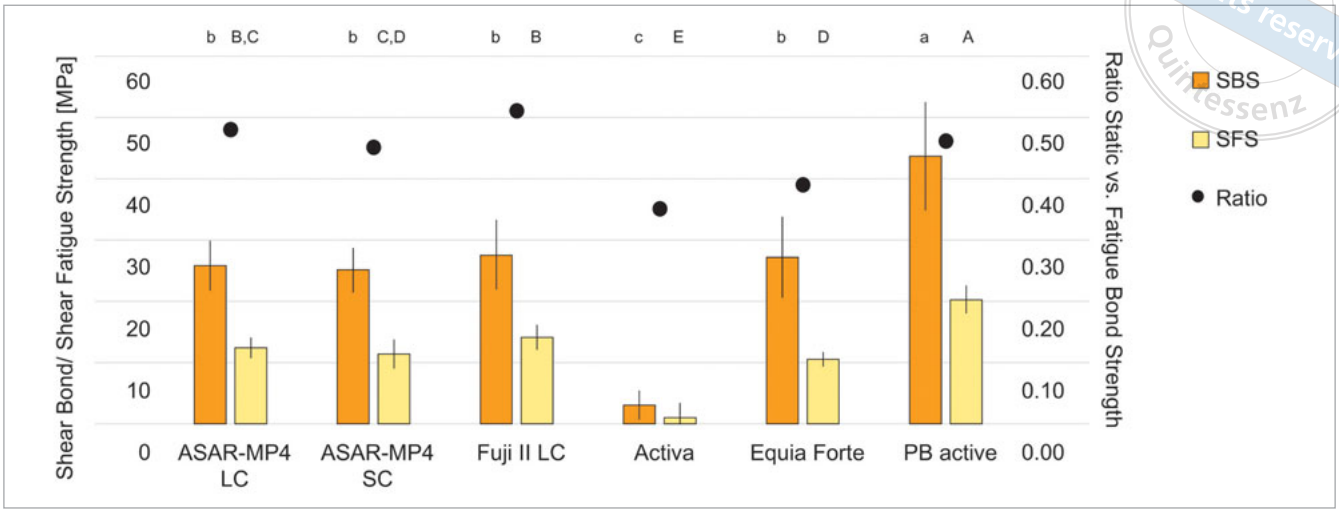


**Fig 3** Results for shear bond strength (SBS) and shear fatigue strength (SFS) to dentin. SBS groups marked with the same small letter were statistically similar ($p > 0.05$). SFS groups marked with the same capital letter were statistically similar ($p > 0.05$). The ratio of SFS to SBS was calculated by dividing the mean SFS by the mean SBS for each material. LC: light cured; SC: self-cured.

## DISCUSSION

Glass-ionomer based materials are widely used in dentistry because they possess a number of valuable properties, including chemical and diffusion-based adhesion to enamel and dentin as well as fluoride release.[16,29] Based on a review article that included thirty-two clinical studies of glass ionomer and resin-modified glass ionomer materials used to restore noncarious cervical lesions, these materials have shown the highest survival rates – which is a testament to the quality of the adhesion properties of these materials without using dental adhesives to promote bonding.[17] In load-bearing restorations in the permanent dentition, resin composites placed with 2- or 3-step adhesives have the highest chance of survival.[19] However, in the clinical environment, the management of the operative field can be challenging in posterior restorations, and the time required by the multiple steps of the adhesive is problematic. Thus, in an effort to increase clinical utility, particularly in posterior restorative applications, modifications have been made to enhance the physical properties of self-adhesive materials.

Most adhesive testing of materials is evaluated shortly after the creation of the bond. The interface between the tooth and the material, despite high initial bond strength, is subjected to mechanical forces and sustained exposure to moisture in the oral environment. Studies have observed the decline of bond strength as a result of hydrolytic degradation and mechanical stress.[1,11] Some authors[12] argued that results using static bond strength testing have limited clinical relevance and should not be used to make clinical recommendations. Theoretically, it should be clinically more relevant to test adhesive interfaces dynamically, as in the clinical situation tooth-resin composite bonds are seldom subjected to the acute shear and/or tensile stresses employed in static bond strength tests.[28] Thus, the use of a fatigue strength model may provide a more robust evaluation of the clinical adhesive potential of newer materials. In the past decade, dynamic adhesive bond strength testing assessing bond fatigue in terms of shear fatigue strength has been developed to better assess the ability of adhesive materials to bond to tooth structure.[10,15,20,23] A method for this bond fatigue strength testing was originally developed at the Academisch Centrum Tandheelkunde Amsterdam (ACTA) using the ACTA fatigue tester in 2006-2008, and was later modified[2,8-10] using a four-station fatigue cycler (Prototech; Portland, OR, USA). Later developments used a servohydrolic testing machine (MTS 858 Mini Bionix II, MTS Systems; Eden Prairie, MN, USA). Most recently, fatigue testing methods were further modified using an all-electric dynamic test instrument (ElectonPuls E1000, Instron).[2,20,23] The present method allows better assessment of a material's total-life tolerance to the repeated low-magnitude loads encountered in the oral cavity, as the cyclic stresses are thought to be more similar to the stresses generated during oral function than the continuous loading to failure applied with traditional shear bond strength testing.[8,9]

Laboratory studies have indicated that shear fatigue strength was not influenced by the frequency rate (5, 10, or 20 Hz with enamel[21] and dentin[18]) or by the number of cycles[24] (50,000, 100,000, or 1,000,000 cycles with enamel and dentin). As a result of these studies, the fatigue load for adhesively bonded resin composite to mineralized tooth structures was standardized using a sine-wave frequency of 20 Hz for 50,000 cycles or until failure occurs. This is a time-efficient approach for shear fatigue strength testing of rapidly advancing modern dental adhesives.

**Table 3** Mean and standard deviation for SBS and SFS to enamel of materials tested

| Material | SBS (MPa) | SFS (MPa) | Ratio SFS/SBS (%) |
|---|---|---|---|
| PB active | 24.3(3.5) | 12.0 (1.6) | 50.6% |
| S(LC) | 22.7(4.8) | 10.2 (2.1) | 44.7% |
| S(SC) | 21.8 (6.6) | 9.6 (1.8) | 44.0% |
| E | 17.1 (3.5) | 8.4 (1.8) | 49.1% |
| F | 15.8 (4.3) | 6.5 (1.6) | 41.1% |
| A | 5.5 (4.2) | 2.4 (1.7) | 43.6% |

**Table 4** Mean and standard deviation for SBS and SFS to dentin of materials tested

| Material | SBS (MPa) | SFS (MPa) | Ratio SFS/SBS (%) |
|---|---|---|---|
| PB active | 43.7 (8.9) | 20.3 (2.3) | 46.5% |
| F | 27.7 (5.7) | 14.1 (2.0) | 50.9% |
| E | 27.2 (6.6) | 10.6 (1.2) | 39.0% |
| S(LC) | 25.8(4.1) | 12.4 (1.7) | 48.1% |
| S(SC) | 25.1 (3.7) | 11.4 (2.3) | 45.1% |
| A | 3.1 (2.4) | 1.1 (2.4) | 35.5% |

The present study focused on investigating the static and dynamic adhesive potential of a newly developed material described as a self-adhesive composite hybrid (ASAR-MP4), with a glass ionomer (Equia Forte), resin-modified glass ionomer (Fuji II LC), a universal adhesive using the self-etching technique and a so-called bioactive self-adhesive material (Activa). Both Equia Forte and Fuji II LC have exhibited similar clinical success in Class V restorations compared to resin composite systems.[3,14,30] However, the most recent directions for use for Activa now recommend the use of a dental adhesive for clinical placement, no doubt due to the clinical evidence of the lack of self-adhesive properties of this material.[25]

On enamel, the universal adhesive and S in both modes generated statistically similar values for SBS. While the SFS for S/LC was statistically lower than the adhesive, this group generated the highest SFS compared to the other materials on enamel. The resin composite used with the universal adhesive is very stiff and has a very high modulus (greater than 15 GPa), which may influence the bonding values in both the static and dynamic test modes in this study.

Excluding Activa, SBS and SFS results in this study showed similar SBS for the self-adhesive materials to dentin. While there were some statistical differences in SFS among these materials, the values for F and S(LC) were statistically similar and may suggest that the new material might perform similarly, at least with respect to mechanical stresses, in a clinical situation. While the SBS and SFS for the universal adhesive to dentin were higher, the ratio of the SFS to SBS was similar for F and S in both self-curing and light-curing modes.

The results of this study confirm that Activa has a very low self-adhesive potential to enamel and dentin. The other self-adhesive materials generated values on enamel that were closer to those of the universal adhesive, compared to the larger differences that were noted on dentin. As dentin has less apatite compared to enamel, it is possible that the creation of micromechanical retention on dentin has a greater influence than does chemical bonding on that substrate, at least comparing the self-adhesive materials to the universal adhesive. Micromechanical retention is an important component in the resistance to mechanical stress, while chemical bonding likely increases resistance to hydrolytic degradation.[26,27] Further studies of the microstructure of the interface and the nature of the chemical interactions with enamel and dentin would help to better characterize the behavior of these materials. In addition, it would be useful to evaluate the shear fatigue strength of specimens after long-term water storage to evaluate the stability of the initial bond of these materials.

Both null hypotheses were rejected, as there were statistically significant differences in bond durability to both enamel and dentin among the materials tested in this study.

## CONCLUSION

The SBS and SFS values of the materials tested were found to vary depending upon the material system. The self-adhesive materials S, F, and E generated similar results in terms of static and dynamic bonding to dentin and enamel. The universal adhesive generated similar values to enamel regarding dynamic bonding. On dentin, the universal adhesive generated the highest values for both bond strength tests.

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**Clinical relevance:** Based on static and dynamic adhesion testing the clinical adhesive performance of a newly developed self-adhesive composite hybrid may equal that of glass ionomer and resin-modified glass ionomer restorative materials.