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Comparative Evaluation of Microtensile Bond Strength of Three Adhesive Systems

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ABSTRACT

Objectives: The aim of this study was to assess the microtensile bond strength of three universal adhesives to dentin and enamel.

Materials and Methods: Sixty extracted human third molar teeth were chosen and divided into six groups regarding the adhesive (G-Premio Bond, Clearfil S3 Bond or Single Bond) and tooth surface. All the applied bonding agents were universal adhesives. The teeth were polished and the adhesives were applied; then the teeth were restored with composite resin. The samples were mounted in acrylic resin and sectioned. The specimens were subjected to a universal testing machine and the microtensile bond strength was measured. The failure mode of each specimen was determined under a stereomicroscope. Data were analyzed using two-way ANOVA ($\alpha=0.05$).

Results: The microtensile bond strength of G-Premio Bond to enamel and dentin was 11.79±8.27 and 17.55±9.47 MPa, respectively which was not significantly different from the values in Single Bond group (15.59±10.66 and 17.19±10.09 MPa to enamel and dentin, respectively; $P>0.05$). However, the values for Clearfil S3 Bond were 7.11±4.23 and 7.88±8.83 MPa to enamel and dentin, respectively, which were significantly lower than the values for G-Premio Bond ($P<0.05$). Scanning electron microscopic (SEM) images showed that the adhesive failure was dominant in both enamel and dentin groups and in all adhesive systems.

Conclusion: G-Premio Bond and Single Bond provided higher microtensile bond strength compared with Clearfil S3 Bond. Universal adhesives with their acceptable performance can be applied in self-etch mode on both enamel and dentin.

Keywords: Dental Cements; Dental Bonding; Dental Enamel; Dentin; Composite Resins

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INTRODUCTION

Adhesive systems have become a common technology for composite resin bonding to the tooth structure in the recent years. Clinical performance of composite resin restorations depends on the adhesives to form an acceptable and durable bond to the tooth structure [1,2]. However, some complications occur after restorative treatment, such as microleakage of bacteria and fluids through the tooth-restoration interface. These complications make adhesive application a challenge. The bacteria, molecules, and ions penetrate through the restoration and dentin or enamel interface and cause secondary caries, discoloration, and pulpitis, affecting restoration durability [3]. Thus, an ideal bond to dentin and enamel in composite restorations is necessary [4].

Adhesive application for dental restorations was started in 1955. At the same time, Buonocore found that acids could be used for tooth surface preparation before adhesive application [5]. Besides, adhesive systems that can easily bond to enamel and dentin make the cavity preparation less invasive [6]. In general, adhesives increase composite resin bond strength to enamel and dentin, and enhance restoration performance [7,8]. Universal adhesive systems were recently introduced, which can be used in different etching modes such as self-etch, selective-etch, and total-etch [7]. These adhesives have all the components in one bottle. Universal adhesives have the ability to bond the dental substrate to direct restorative materials and indirect restorations such as zirconia, alumina, metals, and ceramics. The majority of these adhesives have special monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and nanofillers which promote bonding performance to dental substrate and restorations [9]. There is a debate among the scientists about the efficacy of universal adhesives, especially in self-etch mode. Currently, there is not enough information regarding the effectiveness of universal adhesives, and also comparison of their adhesive strength. Microtensile bond strength test is one of the reliable techniques for evaluation of the bonding performance of adhesive systems. The aim of this study was to evaluate the microtensile bond strength of three commercially available adhesives to enamel and dentin.

MATERIALS AND METHODS

Specimen preparation:

Sixty intact human third molars that had been extracted for orthodontic reasons or due to periodontal disease were used in this study (ethical approval code: 9011272032).

The teeth were stored in 0.5% chloramine-T for one week according to ISO 11405. Then, they were kept at 4°C distilled water. Teeth were divided into 3 groups based on the type of adhesive (n=20). Since the main goal of the current study was to compare the bond strength of adhesives from the same generation, all the selected bonding agents were universal adhesives. G-Premio Bond (GC Corporation, Tokyo, Japan), Clearfil S3 Bond (Kuraray Medical Inc., Okayama, Japan), and Single Bond Universal (3M ESPE, St Paul, MN, USA) were evaluated in this study (Table 1).

Table 1. Specifications of adhesives used in this study

Adhesive system	Manufacturer	Contents
G-Premio Bond	GC Corporatio, Tokyo, Japan	10-MDP, 4-META, 10-methacryoyloxydecyl dihydrogen thiophosphate, methacrylate acid ester, distilled water, acetone, photo-initiators, silica fine powder
Clearfil S3 Bond	Kuraray	10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, ethanol, water, silanated colloidal silica
Single Bond Universal	Medical Inc.,	10-MDP, phosphoric acid ester monomer, HEMA, silane, dimethacrylate, Vitrebond copolymer, filler, ethanol, water, initiators, silane

10-MDP: 10-methacryloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; 4-META: 4-methacryloyloxy-ethyl trimellitate anhydride

Each adhesive group was divided into dentin and enamel subgroups. In dentin subgroups, the enamel surface was cut to expose dentin (2mm from the occlusal surface). In enamel groups, the buccal surface was polished with #600 grit abrasive paper (Acqua Flex, Norton, Brazil) under

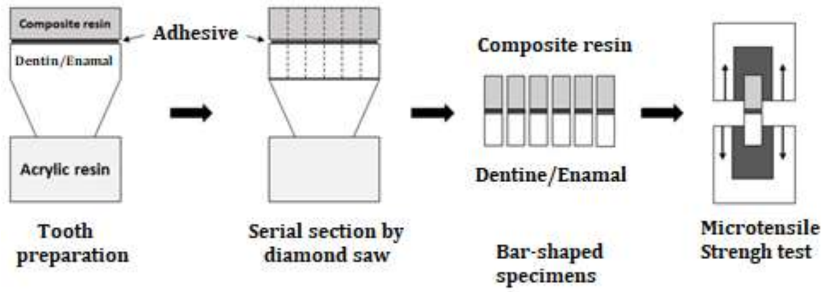

Fig. 1. Schematic view of the experimental design

water in a circular pattern to create a flat surface. The self-etch mode was used for all adhesives; therefore, no acid etchant was used. All the adhesives were applied according to the manufacturers' instructions as follows:

In group one, G-Premio Bond was applied and after 10 seconds, the surface was air-dried for 5 seconds. The adhesive was photo-polymerized by a LED curing unit (Blue phase G2; Ivoclar-Vivadent Schaan, Liechtenstein) for 10 seconds. In group two, Clearfil S3 Bond was applied on the tooth surface without rubbing, as instructed by the manufacturer. After 10 seconds, the adhesive was air-dried for 5 seconds and light-cured for 10 seconds.

In the third group, Single Bond was applied on the tooth surface and rubbed for 20 seconds. The bonding agent was air-dried for 5 seconds and then light-cured for 10 seconds. After adhesive application, composite resin was applied on the tooth surface (Gradia Direct, GC, Tokyo, Japan) with the incremental technique.

Each layer had 2 mm height and the whole composite resin block was build up to reach 5 mm thickness. Each increment of composite was cured with a LED unit for 20 seconds. The samples were kept in saline for 24 hours to complete the polymerization. After preparation, all teeth were mounted in acrylic resin and cut with a low-speed diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA) to create specimens with 1 mm^2 diameter.

Microtensile bond strength test and failure analysis:

A universal testing machine (5566S, Instron, Canton, MA, USA) was utilized for the microtensile test with load applied at a crosshead speed of 0.5 mm/minute. Each specimen was fixed to a jig on the machine and tested until fracture. The force at the time of fracture was recorded in Newtons (N). The microtensile bond strength was calculated by dividing the recorded force by the surface area of each specimen. All microtensile bond strength values were expressed in megapascals (MPa) (Fig. 1).

Failure mode analysis:

To determine the classification of fracture, the specimens were removed from the jig, and the failure mode was classified under a stereomicroscope (SZX9; Olympus, Tokyo, Japan) as adhesive failure (fracture at the adhesive bonding interface), mixed failure (fracture of adhesive and dentin or resin margin) and cohesive failure (fracture within dentin or resin). The frequency of each fracture type was reported as percentage.

Scanning electron microscopic (SEM) observation:

The specific features of adhesive and mixed failures in each group were observed by SEM assessment (JCM-6000 Plus; Joel, Tokyo, Japan). The selected samples were mounted on an aluminum slab and underwent SEM assessment with an accelerating voltage of 20 kV at x100 magnification.

Statistical analysis:

To assess the effect of substrate and adhesive system on microtensile bond strength, two-way ANOVA followed by the Tukey's post-hoc test (for pairwise comparisons) were applied (α=0.05).

RESULTS

Microtensile bond strength:

In the enamel groups, the measured microtensile bond strength in use of G-Premio Bond, Clearfil S3 Bond and Single Bond was

11.79, 7.11 and 15.59 MPa, respectively. In the dentin groups, the microtensile bond strength values in use of G-Premio Bond, Clearfil S3 Bond, and Single Bond were 17.55, 7.88 and 17.19 MPa, respectively (Table 2, Fig. 2).

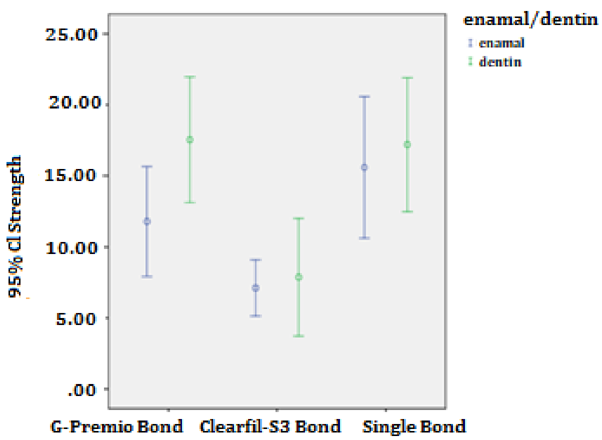

Fig. 2. Microtensile bond strength after using each adhesive on the enamel and dentin substrates

According to two-way ANOVA, the effect of bonding system on microtensile bond strength was significant ($P<0.00$); however, the effect of dental substrate ($P=0.09$) and the interaction effect of bonding agent and substrate on microtensile bond strength ($P=0.41$) were not significant.
This results indicated that the adhesive used for tooth preparation had a significant effect on the bond strength of restoration to tooth surface.
However, the effect of dental substrate (dentin or enamel) was not significant ($P>0.05$).
The Tukey's test showed that there was a significant difference in microtensile bond strength between G-Premio Bond and Clearfil S3 Bond ($P<0.00$). Also, the microtensile bond strength was significantly different between Clearfil S3 Bond and Single Bond ($P<0.00$). The difference between G-Premio Bond and Single Bond was not significant ($P=0.66$).

Failure mode:
Table 3 presents the frequency of failure modes as observed under a stereomicroscope.

SEM observation:
Representative SEM images of adhesive and mixed failures in the enamel and dentin after microtensile bond strength test are shown in Figure 3.

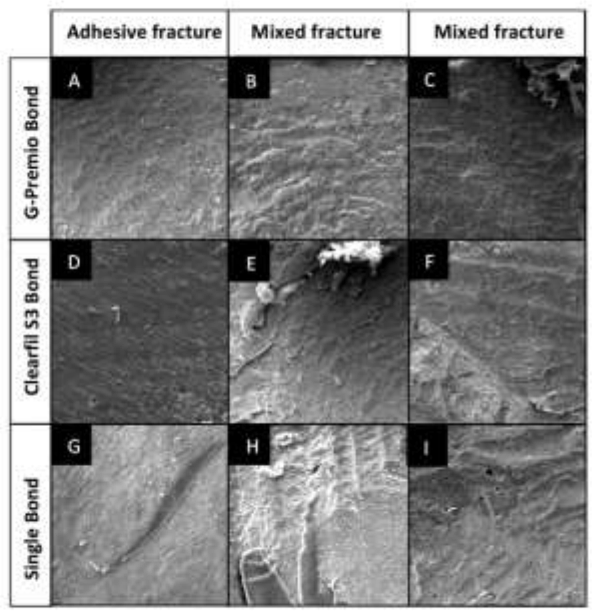

Fig. 3. Representative SEM images of adhesive and mixed fractures, left column: adhesive fracture, middle column: mixed fracture in dentin, right column: mixed fracture in enamel (A, B, C): G-Premio Bond, (D, E, F): Clearfil S3 Bond, (G, H, I): Single Bond

Table 2. Microtensile bond strength values (MPa) after using each adhesive system on dentin and enamel substrates

Adhesive system	Substrate	Upper limit	Lower limit	Mean±SD
G-Premio Bond	Enamel	30.28	1.76	11.79±8.27
	Dentin	49.03	7.48	17.55±9.47
Clearfil S3 Bond	Enamel	18.37	0.81	7.11±4.23
	Dentin	43.25	0.61	7.88±8.83
Single Bond	Enamel	48.19	4.47	15.59±10.66
	Dentin	35.93	3.20	17.19±10.09

SD: standard deviation

Table 3. Frequency and percentage of fracture modes after using each adhesive on enamel and dentin substrates (N=20)

Adhesive system	Substrate	Number (percent)		
		Adhesive fracture	Cohesive fracture	Mixed fracture
G-Premio Bond	Enamel	10 (50)	5 (25)	5 (25)
	Dentin	11 (55)	5 (25)	4 (20)
Clearfil S3 Bond	Enamel	13 (65)	2 (10)	5 (25)
	Dentin	12 (60)	3 (15)	5 (25)
Single Bond	Enamel	13 (65)	4 (20)	3 (15)
	Dentin	10 (50)	7 (35)	3 (15)

As shown in Figures 3A, 3D and 3G, the specimen with adhesive failure had a smooth and homogenous debonded surface. As shown in Figures 3B, 3E, and 3H, the specimen with mixed failure in dentin showed composite residues and a dentinal tubule clogged with smear layer. As indicated in Figures 3C, 3F, and 3I, the specimen with mixed fracture in the enamel showed that part of the enamel surface was covered with composite while exposed enamel was seen in some areas.

DISCUSSION

Universal adhesives are among the recent advancements in adhesive dentistry. These materials have all the ingredients in one bottle, which makes their application simple. Universal adhesives can be used in self-etch, selective-etch, and total-etch modes [7].

The self-etch adhesive system formula contains water-based acidic monomers like ester phosphate or carboxylic acid with hydrophilic monomers like 2-hydroxyethyl methacrylate mostly in 35% and 55% concentrations [10]. Universal adhesives have some functional monomers such as MDP monomers which can improve the long-term bond strength of restoration to the tooth surface. Also, addition of these monomers to adhesives increases the bond strength in dentin bonding [11]. In self-etch adhesives, the smear layer plays the role of an intermediate bonding layer [12].

When a primer or self-etch adhesive is applied on the dentin, it penetrates through the inner layer of mineralized dentin by dissolution of mineral crystals and smear layer [13]. This acidic nature is one of the factors that enhances the durability of restorations [14].

Commonly, 30-40% phosphoric acid is used in total-etch systems. Although the phosphoric acid is not applied in self-etch adhesive systems, the acidic monomers in their composition make the bond strength of restoration to the tooth surface reliable [15, 16]. The resin tags are thicker after using phosphoric acid as an enamel etchant in comparison with acidic primer which may affect the bond strength in total-etch systems. The difference in the quality of resin tags is because of the lower pH of phosphoric acid [17].

In fact, higher pH and lower power of self-etch adhesives compared with total-etch adhesives can affect the quality of bonding [18]. In self-etch adhesives, the etch and rinse step is skipped; thus, the concentration of calcium and phosphorous ions after the dissolution of hydroxyapatite crystals prevents further dissolution and may decrease demineralization [19]. However, some studies showed that the bond strength of self-etch and total-etch adhesives was similar and can provide a reliable bond specially in dentin where achieving an acceptable bond strength is more challenging [20–22]. Other investigations reported that the bond strength of one-step self-etch adhesives was lower than that of total-etch and also two-step self-etch adhesives [23,24].

Although self-etch adhesives make thinner resin tags, the bond strength of restorations to enamel when applying these adhesives is the same as total-etch adhesive systems [25-28].

In self-etch application mode of universal adhesives, although hydroxyapatite crystals are exposed to acidic monomers, their degradation is not complete.

This partial degradation is one of the advantages of universal adhesives. The crystals around collagen fibrils have an important role in bond strength to the surface of the tooth since they have a chemical reaction with functional monomers in adhesives. The results of this study showed that Clearfil S3 Bond and Single Bond had almost the same microtensile bond strength to dentin and enamel. Nevertheless, while using G-Premio Bond, the microtensile bond strength to dentin (17.55 MPa) was more than that to enamel substrate (11.79 MPa). In general, the type of substrate did not have a significant effect on microtensile bond strength. However, the effect of using a different type of adhesive system was prominent. In a study by Yazici et al, [29] it was shown that in each adhesive system, the microtensile bond strength to enamel was significantly more than that to dentin; their results were different from the present findings. In the present study, the substrate type did not have a significant effect on microtensile bond strength. We believe that the main reason for this difference could be related to the type of adhesive system used in the two studies.

G-Premio Bond is a universal light-cure adhesive that is used as a bonding agent for direct composite resin and compomer restorations. It is commonly used for dual-cure bonding of core build-up restorations, repair of porcelain, composite restorations, and treatment of tooth hypersensitivity [30]. This adhesive has three functional monomers; the first one is 4-methacryloyloxyethyl trimellitate anhydride which is responsible for bond strength of restoration to dentin and enamel surface. The second functional monomer is MDP which results in bond strength to zirconia, alumina, and non-precious metals in addition to dentin and enamel. The third functional monomer is methacryloyloxydecyl dihydrogen thiophosphate; this monomer enables acceptable bond strength to precious metals. High penetration and wettability of this adhesive results in favorable infiltration into dentinal tubules.

In our study, G-Premio Bond and Single Bond had the highest bond strength to dentin. However, Clearfil S3 Bond did not provide a reliable bond strength compared with the other two adhesives, which can be related to its functional monomers. Cevik et al. [31] reported that self-etch adhesives containing MDP and 4-methacryloyloxyethyl trimellitate anhydride showed more favorable bond strength to dentin compared with other types [31]. The result of their study was in accordance with our study as G-Premio Bond containing the afore-mentioned monomers showed higher bond strength to dentin in comparison with two other groups. However, in the study by Fabiao et al, [32] it was indicated that G-Premio Bond presented lower bond strength to dentin compared with two-step self-etch adhesives. This finding was different from the results of the present study. The differences between the results of these studies can be related to several factors such as the methods of evaluation and the applied adhesives.

The performance of adhesives for bonding to the tooth surface is based on the enamel and dentin demineralization and monomer infiltration [33]. Besides, the technique of adhesive application adopted by the clinician, and its formulation play important roles in the final outcome [34]. Also, chemical composition, monomer content, and percentage and type of fillers differ among different adhesives, and can influence the performance of adhesive systems [35,36].

SEM fractographic images were used for assessment of the fracture patterns in the present study. SEM images showed that the adhesive failure was dominant in both enamel and dentin groups and among all the tested adhesive systems. In the study by Gre et al, [37] the microtensile bond strength of a total-etch adhesive and universal adhesives was analyzed and it was indicated that most of the fracture modes were adhesive in all techniques. Further studies are recommended to assess the long-term durability of universal adhesive systems. Also, clinical studies are recommended to compare other clinical factors that influence the restorations to cast a final judgment.

CONCLUSION

Based on the results of the current study, it was shown that G-Premio Bond and Single Bond provided higher microtensile bond strength compared with Clearfil S3 Bond in both enamel and dentin surfaces. Since the performance of universal adhesives on dentin and enamel is similar, these adhesives can be used in self-etch mode on tooth surfaces. It can also be concluded that universal adhesives can be a candidate for satisfactory restorations due to their reliable microtensile bond strength.

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CONFLICT OF INTEREST STATEMENT

None declared.

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