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Evaluation of micro-shear bond strength of composite to amalgam using different bonding systems

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ABSTRACT

Introduction: Recently, a new category of dental bonding agents called universal adhesives has been introduced to the market, which are claimed to be able to create a strong bond between composite resins and teeth, metal surfaces and all types of ceramics alone and without the use of other primers. The aim of this study was to evaluate the performance of universal adhesives in the repair of amalgam restorations with composite resins in comparison with the conventional method.

Materials & Methods: In this experimental in vitro study, 80 amalgam blocks were prepared and after abrading the surface with an intraoral sandblaster and performing the aging process, the specimens were divided into 4 groups of 20 each, depending on how each surface was prepared by different bonding in the following order: Single Bond2 (S), Alloy Primer and Single Bond (AS), G-Premio Bond (G) and Alloy Primer and G-Premio Bond (AG). The composite blocks were then cured on the surface of the specimens. Finally, the specimens were tested for micro-shear bond strength (μSBS) using a micro-tensile tester and their cross-section was examined for fracture pattern under a stereomicroscope. Data were analyzed using Kruskal-Wallis, Man Whitney, and chi-square tests. A value of $P<0.05$ was considered significant.

Results: The highest and lowest bond strengths belonged to the AG and S groups, respectively. The mean difference of μSBS in the four groups was significant, except in the AS and G groups.

Conclusion: G-Premio universal bond is able to create sufficient bond strength between composite and amalgam. However, it is recommended to use a combination of Alloy Primer with all bonding systems to improve bond strength.

Keywords: Dental Amalgam, Composite Resins, Shear Strength

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Introduction

Dental amalgam has been successfully used as a restorative material for over a century and accounts for approximately 75% of the restorative materials used by dentists. The advantages of amalgam include its low cost, good physical properties, ease of use, high durability, low technical sensitivity, high abrasion resistance, and inherent sealing ability. However, sometimes these restorations need to be replaced for reasons including marginal defects, recurrent carries, poor marginal integrity, fractured cusp, or the need to esthetically conceal the metal color of the amalgam. [1-10] In such cases, the idea of repairing defective areas with materials such as composite is more conservative compared to complete replacement and spares the dental tissue. Therefore, this method causes less iatrogenic injuries and the dental pulp is protected. It is also less time-consuming and more cost-effective. [2,4-8] In this context, extensive research has been conducted to achieve an optimal bond between composite and amalgam. The results of these studies have indicated that various methods can be used to improve such a bond. These include mechanical methods such as surface preparation through roughening by sandblasting [2,5,6,8,11,12] as well as methods for creating a chemical bond with the metal surface by applying a resin or multi-purpose adhesives or agents known as primers. [4, 8, 12-17] These materials are able to form ionic bonds with metal oxides or active metal compounds of the amalgam. However, there are still conflicting results regarding the formation of true chemical bonds between metals, especially amalgam and resin composites. Recently, a new generation of dental adhesives has appeared on the market, called multimode or universal adhesives. [4, 18,19] These adhesives contain a phosphate monomer and a silane monomer in addition to the conventional functional monomer. The phosphate esters, which are present in all universal adhesives, have the potential to form chemical bonds with metals, zirconia and dental tissue by forming insoluble Ca^{+2} salts. Furthermore, the silane in these compounds leads to better bonding between adhesive resin components and composites and silica-based ceramics. [4, 18, 20-22] Considering the above explanations and according to the manufacturers of these adhesives, it seems that the use of these bonding systems eliminates the need to use separate primers such as silane and metal primers to form a bond to different surfaces.

Bond strength is a very important mechanical property to predict the clinical durability and success of tooth-colored restorations. [3, 23] Moreover, previous studies have shown a positive correlation between this property and the extent of polymerization shrinkage, which is considered to be the major challenge in composite resin restorations. [3] Therefore, if the manufacturers' claim is confirmed, the use of universal adhesives is less time-consuming and less costly for both the patient and the dentist compared to conventional adhesives, as the technical steps are shorter and no separate primers have to be used. Considering that there are few [4, 16, 17, 19, 23-26] and inadequate studies on the efficiency of these adhesives in bonding to metal surfaces, especially amalgam, the aim of the present study was to investigate the bond strength of composite resin to amalgam restorations using universal adhesives and to compare it with conventional methods involving the use of an Alloy Primer with a conventional two-step etch-and-rinse adhesive. The null hypotheses of the present study are as follows:

1. There is no difference between universal bonding and single bonding in terms of micro-shear bond strength (μSBS) of the specimens.
2. The application of an Alloy Primer has no effect on the μSBS of the specimens.

Materials & Methods

Ethical approval was obtained from the Ethics Committee of Zanjan University of Medical Sciences (IR.ZUMS.REC.1397.143).

Sample Preparation

A total of 80 amalgam blocks (dimensions 7 × 5 × 2 mm) (sample size was calculated according to a previous article [23] $n=\frac{(Z_{1-\frac{\alpha}{2}}+Z_{1-\beta})^2}{(\mu_1-\mu_2)^2}\times(SD_1^2+SD_2^2)$ $\alpha=0.05, \beta=0.2, \mu_1=5.26, \mu_2=4.04, SD_1=1.49, SD_2=1.26, n=20$) were made in acrylic molds. To make these blocks, the acrylic molds were placed on glass slabs. The amalgam (ANA 2000Duet, Nordiska Dental AB, Angelholm, Sweden, extra high copper, admixed alloy, containing: 26.1% copper, 43% silver, and 30.8% tin) was then triturated in the amalgamator (FAGHIHI, FD-5000-A, Iran) and filled into the cavities with a condenser. Excess amalgam at the margins was removed with a glass slide pressed firmly against the cavity. The amalgam specimens were stored in distilled water in an incubator at 37 °C and 100% humidity for 24 hours to complete the curing process. Subsequently, the top of the amalgam blocks was polished with a 600-grit silicon carbide abrasive paper (Roeko) under running tap water to prevent overheating. The samples were then placed in an ultrasonic bath (EUROSONIC 4D, Italy) with distilled water for 10 minutes to remove possible impurities. They were then dried and placed in artificial saliva solution (RGS, Iran) containing mucin, lysozyme, α-amylase, and albumin in buffered phosphate saline to simulate the protein coat available under clinical conditions and stored at 37 ± 1 °C for two weeks for aging [4]. The surface of the specimens was then dried and cleaned with an alcohol-impregnated cotton ball for 5 seconds. The contact surface of the samples was sandblasted with 50µm aluminum oxide particles using an intraoral sandblaster (Micro Etcher Danville IIA, USA) at a pressure of 2 MPa from a distance of 10 mm for 10 seconds at a speed of 12 mm /s and an angle of 90 °. They were randomly divided into the following four groups of 20 amalgam blocks each, using different bonding systems on their surface:

Single Bond2 (3M, ESPE, USA) (S), Alloy Primer (Clearfil, Kuraray Medical, Inc.Okayama, Japan) + Single Bond (AS) (applied, respectively), G-Premio Bond (GC, America, USA) (G), Alloy Primer + G-Premio Bond (AG) (applied, respectively) (Table1). All primers and adhesives were used according to the manufacturer's instructions. Next, the composite cylinders (Kerr, point4 A1, Italy) (dimensions 0.7 × 1 mm) -in Tygon tubes (Tygon, Norton Performance Plastic Co., Cleveland, OH)- were placed on the amalgam blocks with the help of a plugger and cured with a light curing device (LED, woodpecker, China) at an intensity of 1000 $\frac{mw}{cm^2}$ for 30 seconds (Figure 1). Then, the tubes were carefully removed with a bistoury blade, and the samples were kept in distilled water at 37 ° C in an incubator for 24 hours.

Bond strength test

A micro-tensile tester (Bisco, USA) was used to evaluate the µSBS (Figure 1). To attach the specimens to the instrument, the base of the amalgam specimens was glued to the table provided for placement of the specimens using a cyanoacrylate adhesive. A wire with a semicircular loop (diameter 0.25 mm) was placed around the adhesive surface between the amalgam and composite resin. A micro-shear force was applied to the specimens at a speed of 0.5 mm/min via the crosshead of the unit. The values determined in newtons were divided by the cross-section in mm^2 of the composite cylinders and the values were expressed in Mpa.

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Table 1. Materials used and manufacturer's instructions

Restorative materials and factory	Type of restorative material	Ingredient	Factory instructions
ANA 2000 (Duet, Nordiska Dental AB, Angelholm, Sweden)	Amalgam	Non-gamma2,lathe-cut, high-copper alloy with 43% Ag, 25.4% Cu	In an amalgamator, at 5,000 rpm for 10 seconds
G-Premio Bond (GC,America,USA)	Universal adhesive	4-MET*, MDP, MDTP, acetone, water, initiator	Apply The bonding agent to the surface by an applicator. Wait for 10 seconds and then air dry for 5 seconds with high air pressure - cure for 10 seconds.
SingleBond (3M, ESPE,USA)	Two-Step etch and rinse adhesive	BisGMA, HEMA, dimethacrylates, ethanol, water, Photoinitiator	Apply on the surface in two layers, gently air dry each layer for 5 seconds, cure for 10 seconds.
Alloy Primer (Clearfil, Kuraray Medical, Inc. Okayama, Japan)	Metal conditioner	VBATDT,MDP, acetone	Apply on a sandblasted metal surface. Wait for 5 seconds, apply dental adhesive on the surface
Kerr, point4A_1 (Italy)	light-cured, resin-based composite	BIS-GMA, TEGDMA and BIS-EMA Filler: Barium glass and Silica Average particle size 0.4 microns	light cure for 20 seconds

* 4-MET (4-methacryloxyethyl trimellitic acid), MDP (methacryloyloxydecyl dihydrogenphosphate), MDTP (methacryloyloxydecyl dihydrogen triphosphate), BisGMA (Bisphenol glycidyl methacrylate), HEMA (hydroxyethyl methacrylate), VBATDT (6- (4-vinylbenzylpropyl) amino-1,3,5-triazine-2,4-ditione), TEGDMA (triethylene glycol dimethacrylate and BIS-EMA (ethoxylated bisphenol-A dimethacrylate)

Figure 1. **Micro-tensile tester**

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The fracture pattern

The cross-sectional area of the fractured specimens was examined under a stereomicroscope (Nikon's SMZ800 Japan) at 20x magnification. The type of fracture at the surface between resin and amalgam was indicated as adhesive, cohesive, or combined. Fractures that occurred only at the adhesive surface were classified as adhesive. If the fractures involved only the substrate or the restorative composite, they were designated as cohesive, and if both types of adhesive and cohesive fractures occurred simultaneously, they were classified as mixed fractures (Figure 2).

Statistical analysis

Statistical analysis was performed using SPSS Inc. (SPSS Inc., Chicago, IL, USA) 24SPSS. The Kolmogorov-Smirnov one-sample test showed that the data distribution was not normally distributed. Therefore, the Kruskal-Wallis test was applied to compare the bond strength, and the Mann-Whitney test was used for pairwise comparison. The chi-square test was utilized to evaluate the fracture types in the groups.

Figure 2. Different fracture patterns under a stereomicroscope

Results

The mean μSBS is given in the table (Table 2). The Kruskal-Wallis test showed a significant difference between the four groups in terms of the mean value of bond strength ($P < 0.001$). The bond strength of the AG group (12.50 ± 4.92 MPa) was significantly higher than that of the other groups ($P \leq 0.036$). The results of the Mann-Whitney test represented a significant difference in all groups except in the AS and G groups ($P > 0.05$) (Table 3). The results of the chi-square statistical analysis indicated that the frequency distribution of the different fracture patterns among the 4 groups was not statistically significant ($P > 0.05$) (Table 4). Adhesion fracture accounted for the majority of fractures in each of the groups. The percentage and frequency of fracture patterns of the specimens are illustrated in Figure 3 and Table 4.

Table 2. Mean and standard deviation of bond strength values in four studied groups

Group	Bond strength	Sample size
Singlebond + Alloyprimer	9.32 ± 32.05	20
G-Premio bond + Alloyprimer	12.4 ± 50.92	20
Singlebond	6.3 ± 23/47	20
G-Premio bond	8.3 ± 44.68	20

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Table 3. Comparison of bond strength of different groups

Group	1 Alloyprimer+Single bond	2 Alloyprimer+Gpremio bond	3 Singlebond	4 G-Premio bond
Singlebond + Alloyprimer	-	0.036(S)	0.011(S)	0.465(NS)
G-Premio bond+Alloyprimer	-	-	0.000(S)	0.007(S)
Singlebond	-	-	-	(S)0.014
G-Premio bond	-	-	-	-

Mann Whitney Statistical Test, S: Significant, NS: Non-significant

Table 4. Failure modes

Group	Failure Modes(%)		
	Adhesive	Cohesive	Mixed
AlloyPrimer+ Single Bond	85%	10%	5%
Alloyprimer+ Gpremio bond	75%	10%	15%
Single bond	70%	10%	20%
Gpremiobond	75%	15%	10%

Chi-Square Statistical Analysis (p=0.825)

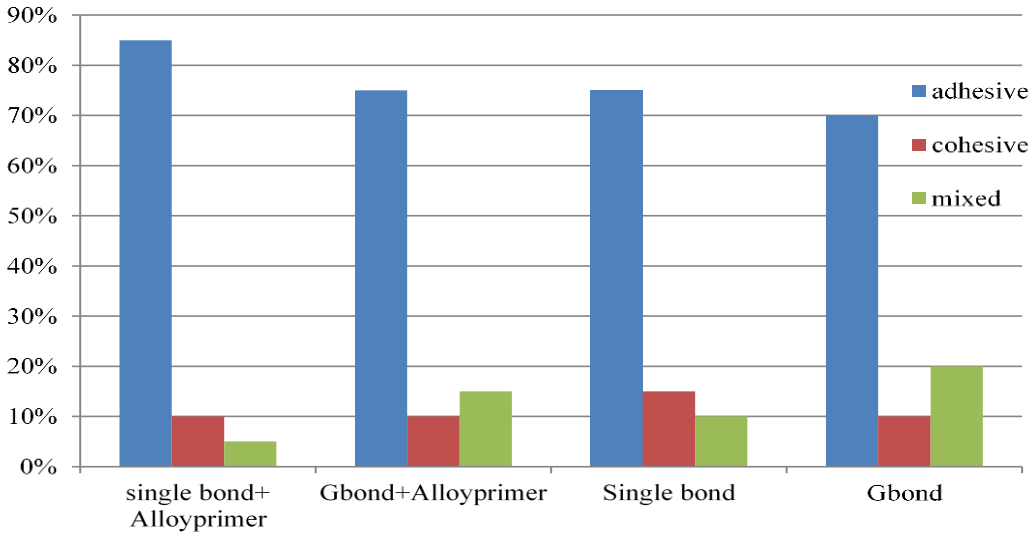

Figure 3. Frequency of the fracture patterns

Discussion

The results showed that, the universal adhesive had a higher bond strength compared to conventional adhesives and that the use of metal primer together with these bonding systems helped to improve the bond as much as possible.which is consistent with the results of some studies. [4,15, 23, 24, 27] This finding may be due to higher phosphate monomer concentration in metal primers compared to G premio bond and its intact structure over time in Alloy Primer bottles; in single-bottle bonding systems where pH is between 1 and 2 (G-Premio Ph=1.5), the

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effectiveness of functional phosphate monomers (such as MDP) and carboxylates (e.g., 4 MET) decreases slightly during storage due to the degradation and hydrolysis of ester bonds (methacryloxy esters) near the acidic monomers and the water content of the adhesive.[28] Balkaya et al. reported similar SBS for conventional and universal bonding agents [4]; however, in the present study, the universal adhesive provided higher bond strength than the conventional bonding system even without metal primer. This difference may be attributed to the type of adhesives used and the presence of two types of phosphate monomers, i.e., 4MET and MDP, in the G-Premio bond structure while only one type of phosphate acid monomers is used in the structure of Single Bond Universal, Futurabond Universal, and Clearfil Universal. Furthermore, the solvent in the G-Premio Bond is acetone. As the results of the study by Nishiyama et al. suggested, the ester bonds in functional methacrylate monomers are hydrolyzed in the presence of acetone as a solvent.[29] However, in all three types of adhesives mentioned in Balkaya's study, alcohol was used as a solvent. The results of carbon-13 nuclear magnetic resonance (CNMR)transmission electron microscopy (TEM) and Fourier transform infrared spectroscopy (FTIR) demonstrated that in the presence of alcoholic solvent hydrolytic degradation of functional monomers, esterification of acidic groups also occurs, even much more frequently than the first type. [28] Therefore, the effectiveness of this type of adhesive is more likely to be affected by time.

Another reason for the higher bond strength in Alloy Primer+ bonding groups is that Alloy primers increase the wettability of the adhesive and thus increase the bond strength by enhancing the diffusion of the functional monomers into the micro- and nano-retentions formed by sandblasting [30], and consequently reducing the contact angle for the bond between the resin adhesive and amalgam.

Similar to some studies [4, 26] ,the present study showed that adhesive fracture was the most common fracture pattern in all specimens. However, there was no significant statiscal difference between the 4 groups($P>0.05$). It can be pointed out that despite the progress made in improving composite-amalgam bonding, the bond strength is still lower than the cohesive strength of the composite. In the ongoing study, amalgam specimens were stored in artificial saliva at 37 ° C for two weeks to simulate the clinical conditions of the oral environment [4] and the time interval between restoration placement and fracture occurrence. A micro-shear test was used to evaluate the bond strength in this study due to these advantages: 1. Its ease of use, and 2.No need to trim specimens, thus avoiding missing a number of specimens during preparation. In addition, microtesting is much less likely to concentrate structural defects in any single specimen due to their small bonding areas. [23, 31,32] resulting in more adhesive fractures which contributes to higher validity of test results.[4] This study was performed in a laboratory environment free of oral fluids, and occlusal pressure. the aging process was performed on amalgam by only storing in artificial saliva, and only the immediate bond strength was investigated ,so that further long term in vitro and in vivo studies are required.

Conclusion

Considering the limitations of the present study, it can be concluded that when repairing a fractured amalgam, the bond strength can be improved by using Alloy Primer before applying dental adhesives to the amalgam surface.

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Conflicts of Interest: There are no conflicts of interest

Authors' Contribution

A.Naghili, E.Zajkani helped with study design , data analysis writing and editing the manuscript. A. Naghili, M.Mirzania, E.Zajkani helped with data collection. All authors read and approved the final manuscript.

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