

BASIC RESEARCH

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Evaluation of Effectiveness of Self-Adhesive Composites for Amalgam Repair with or Without Alloy Primer, in Terms of Shear Bond Strength

Evaluación de la efectividad de los compuestos autoadhesivos para la reparación de amalgamas con o sin imprimación de aleación, en términos de resistencia al cizallamiento

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ABSTRACT: Purpose: The purpose of the present research was to evaluate the effectiveness of self-adhesive composites in amalgam repair as time-saving alternatives to universal bonding-universal composite materials, with or without the usage of Alloy Primer in terms of shear bond strength. Materials and Methods: Forty-two disc-shaped amalgam samples were prepared by condensing into 6×2 mm holes in acrylic resin blocks, sandblasted with 50 µm Al₂O₃ and randomly divided into 6 groups according to repair material [Constic (Group C), Fusio Liquid Dentin (Group FLD), Universal bonding+Universal composite (Group Control), Alloy Primer+Constic (Group APC), Alloy Primer+Fusio Liquid Dentin (Group APFLD), Alloy Primer+Universal bonding+Universal composite (Group APControl)]. After shear bond strength test, the fracture modes were determined under a digital microscope. Statistical analysis was performed using one-way analysis of variance and Tukey's post hoc test. Results: The highest shear bond strength values were obtained for Fusio Liquid Dentin, among the three repair materials when Alloy Primer was not applied ($p < 0.05$). Usage of Alloy Primer increased shear bond strength values of the investigated repair materials to amalgam, except Fusio Liquid Dentin. Conclusion: Within the limitations of the present research, it can be concluded that; the investigated self-adhesive composite materials could be time-saving alternatives to the Universal bonding+Universal composite for the purpose of amalgam repair, in terms of shear bond strength. The clinicians could prefer Fusio Liquid Dentin self-adhesive composite material without Alloy Primer application for the purpose of amalgam repair in non-cooperative patients.

KEYWORDS: Amalgam repair; Self-adhesive composite; Shear bond strength; Alloy primer; Amalgam; Composite.

RESUMEN: Propósito: El propósito de la presente investigación fue evaluar la efectividad de las resinas compuestas autoadhesivas en la reparación de amalgamas como alternativas que ahorran tiempo con respecto a las resinas compuestas universales de adhesión universal, con o sin el uso de Alloy Primer en términos de resistencia al cizallamiento. Materiales y métodos: Se prepararon 42 muestras de amalgama en forma de disco condensándolas en orificios de 6×2 mm en bloques de resina acrílica, arenadas con 50 µm de Al₂O₃ y divididas al azar en 6 grupos según el material de reparación [Constic (Grupo C), Fusio Liquid Dentin (Grupo FLD), Adhesivo universal+Resina universal (Grupo Control), Imprimación de aleación+Constic (Grupo APC), Imprimación de aleación+Fusio Liquid Dentin (Grupo APFLD), Imprimación de aleación+Adhesivo universal+Resina universal (Grupo APControl)]. Después de la prueba de resistencia al cizallamiento, los modos de fractura se determinaron bajo un microscopio digital. El análisis estadístico se realizó mediante análisis de varianza unidireccional y la prueba post hoc de Tukey. Resultados: Se obtuvieron los valores de resistencia al cizallamiento más altos para Fusio Liquid Dentin, entre los tres materiales de reparación cuando no se aplicó Alloy Primer ($p < 0.05$). El uso de Alloy Primer aumentó los valores de resistencia al cizallamiento de los materiales de reparación investigados para la amalgama, excepto Fusio Liquid Dentin. Conclusión: Dentro de las limitaciones de la presente investigación, se puede concluir que; los materiales compuestos autoadhesivos investigados podrían ser alternativas que ahorran tiempo con respecto al adhesivo universal+resina universal con el fin de reparar las amalgamas, en términos de resistencia a la cizalladura. Los clínicos podrían preferir el material compuesto autoadhesivo Fusio Liquid Dentin sin la aplicación Alloy Primer con el fin de reparar amalgamas en pacientes poco cooperadores.

PALABRAS CLAVE: Reparación de amalgama; Resina autoadhesiva; Resistencia al cizallamiento; Imprimación de aleación; Amalgama; Resina.

INTRODUCTION

When defect occurs in amalgam restorations, repair is preferred over renewing these restorations, because some of the adjacent dentin could be damaged in the latter procedure (1). There are various mechanical and chemical repair techniques used to repair amalgam restorations (2,3). Mechanical techniques include threading of pins (3) and chemical techniques involve the use of adhesive agents (4,5). Airborne particle abrasion, a mechanical technique, modifies the metal surface, thereby enhancing

micromechanical bonding (3,4,6). On the other hand, primers and adhesives produce ionic bonds with amalgam surfaces (6). The effect of Alloy Primers (AP), metal primers, opaquers, airborne particle abrasion, fibre sheets, bonding systems, silica coating and silanization on composite and amalgam bonding have been reported in the literature (1,6-9). However, these complex methods are not usually suitable for amalgam repair in non-cooperate patients.

New self-adhesive flowable resin composites, such as Constic (C) (DMG, Germany) and Fusio

Liquid Dentin (FLD) (Pentron Clinical, USA) were recently introduced to the dental market (10). The use of these materials eliminates the need for a separate bonding procedure, improves the ease of use and saves time (11,12). Self-adhesive resin composites contain self-etching and/or self-adhesive monomers those etch enamel and dentin; they may also chemically bond to hydroxyapatite (13). A previous study (14) reported a self-adhesive composite to be adequate for the repair of noncarious amalgam defects, in terms of microleakage. Moreover, studies have focused on the bonding strength of self-adhesive flowable resin composites to dentin, enamel, nano-composites or ceramics (10,15-20); however, information on the ability of these composites to bond with amalgam remains lacking.

FLD contains 4-methacryloxyethyl trimellitic acid (4-META) and contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP). 4-META chemically bonds to hydrophobic amalgam surface via 4-META (21, 22), while MDP acts as a functional monomer for base alloys (23, 24). Furthermore, AP that also contains MDP was reported to increase the micro shear bond strength (μ SBS) values between amalgam and universal composite in a previous research (8). The information on the effect of AP application on the shear bond strength (SBS) values of these self-adhesive composites with amalgam remains lacking.

This *in vitro* study aimed to evaluate the efficiencies of self-adhesive composites with or without AP application for amalgam repair, in terms of SBS. The tested first null hypothesis of the present study was that there would be no differences among the SBS values between the different composite materials and amalgam. And the second null hypothesis was that AP application would not affect the SBS values between the composite materials and amalgam.

MATERIALS AND METHODS

Two commercially available self-adhesive composites, a universal bonding (UB) material, a universal composite (UC) material, amalgam and AP were used in the present study. The brand names, manufacturers and chemical compositions of the materials are listed in Table 1.

PREPARATION OF AMALGAM SAMPLES

A non-gamma 2, lathe cut, high copper amalgam (Rubydent, İnci Dental, İstanbul, Turkey) (50%Ag, 30%Sn, 20%Cu) was triturated according to manufacturers' instructions and condensed into a 6x2 mm hole in polymerized poly methyl methacrylate (PMMA) (Meliodent, Vario, HeraeusKulzer, Wehrheim, Germany) until they were overfilled (Figure 1A).

Forty-two amalgam-PMMA specimens were finished with 1200-grit silicon carbide abrasive papers (Carbimed Paper Discs, Buehler, Lake Bluff, Illinois) after setting for 24h in an incubator at 23°C. All of the amalgam samples were sandblasted with 50 μ m Al₂O₃ for the purpose of standardizing amalgam surfaces using an intraoral sandblaster (Kavo RONDOflex Plus 360; KaVo Dental GmbH) at a pressure of 2 bars from a distance of 10mm for 10s (Figure 1B).

REPAIRING PROTOCOLS

The PMMA-amalgam specimens were cleaned in an ultrasonic bath for 10 min and air-dried, then randomly divided into 6 groups (n=7) (Table 2). The sample size was calculated according to a previous article (25) and it was determined that 7 specimens per group provided a power of 0,95 at a significance level of 0,05.

Following repair protocols were preformed for the relevant groups:

- Group C: C (DMG, Germany) was injected to the polyethylene mold (inner diameter, 3mm; height, 4mm) incrementally in two layers measuring not more than 2mm. Each layers of C were dispensed to the surface for 25 s using the dispensing tip provided by the manufacturer and light polymerized (at 800 mW/cm², Elipar Trilight, 3M ESPE, Seefeld, Germany) for 20 s. A distance of 2mm (9) between each layers of C and light-tip was provided; a glass plate (2mm in thickness) was placed on top, before light polymerization of the last layer of C for the same purpose. After light polymerization of the last layer of C, polyethylene moulds were removed gently from the specimens.
- Group FLD: FLD (Pentron Clinical, USA) was injected to the polyethylene mold (inner diameter, 3mm; height, 2mm) incrementally in two layers measuring not more than 1mm. Each layers of FLD were dispensed to the surface for 20s and light polymerized (at 800 mW/cm², Elipar Trilight, 3M ESPE, Seefeld, Germany) from a distance of 2mm for 10s (9). Another polyethylene mold (inner diameter, 3mm; height, 2mm) was placed upon the first one, and the same procedure was repeated. A glass plate (2 mm in thickness) was placed on top of the last layer before polymerization. Polyethylene moulds were removed gently from the specimens.
- Control Group: The following protocol was used for specimens repaired by UB (All Bond Universal, Universal bonding resin, Bisco, Schaumburg, IL, USA) + UC (Aelite All Purpose Body, microhybrid composite, Bisco, Schaumburg, IL, USA). The UB was applied to the prepared amalgam surface using the brush tip, and then air-dried with an air-water syringe. The bonding agent was then cured for 20s. The UC was condensed incrementally into the polyethylene mold (inner diameter, 3mm; height, 4mm) in two layers measuring not more than 2mm. Each layers of UC were light cured (at 800 mW/cm², Elipar Trilight, 3M ESPE, Seefeld, Germany) from a distance of 2 mm for 40s (9). A glass plate (2mm in thickness) was placed on top of the second layer before polymerization. Polyethylene moulds were removed gently from the specimens.
- Group APC: One layer of AP (Z-PRIME, Bisco, Schaumburg, IL, USA) was applied to the amalgam surface using single brush and then air-dried with an air-water syringe. C (DMG, Germany) was injected to the polyethylene mold (inner diameter, 3mm; height, 4mm) incrementally in two layers measuring not more than 2mm. Each layers of C were dispensed to the surface for 25 s using the dispensing tip provided by the manufacturer and light polymerized (at 800 mW/cm², Elipar Trilight, 3M ESPE, Seefeld, Germany) from a distance of 2mm (9) for 20 s. A glass plate (2mm in thickness) was placed on top of the second layer before polymerization. Polyethylene moulds were removed gently from the specimens.
- Group APFLD: One layer of AP (Z-PRIME, Bisco, Schaumburg, IL, USA) was applied to the amalgam surface using single brush and then air-dried with an air-water syringe. FLD (Pentron Clinical, USA) was injected to the polyethylene mold (inner diameter, 3mm; height, 2mm) incrementally in two layers measuring not more than 1mm. Each layer of injected composite was dispensed to the surface for 20s and light polymerized (at 800 mW/cm², Elipar Trilight, 3M ESPE, Seefeld, Germany) from a distance of 2mm (9) for 10s. Another polyethylene mold (inner diameter, 3mm; height, 2mm) was placed upon the first one, and the same procedure was repeated. A glass plate (2mm in thickness) was placed on top of the last layer before polymerization. Polyethylene moulds were removed gently from the specimens.
- Group APControl: One layer of AP (Z-PRIME, Bisco, Schaumburg, IL, USA) was applied to the amalgam surface using single brush and then air-dried with an air-water syringe. The UB (All Bond Universal, Universal bonding resin, Bisco, Schaumburg, IL, USA) was applied to the prepared amalgam surface using the brush tip, and then air-dried with

an air-water syringe. The bonding agent was then cured for 20s. The UC (Aelite All Purpose Body, microhybrid composite, Bisco, Schaumburg, IL, USA) layers were condensed incrementally into the polyethylene mold (inner diameter, 3mm; height, 4mm) in two layers measuring not more than 2mm. Each layers of UC were light cured (at 800 mW/cm², Elipar Trilight, 3M ESPE, Seefeld, Germany) from a distance of 2mm for 40s (9). A glass plate (2mm in thickness) was placed on top of the second layer before polymerization. Polyethylene moulds were removed gently from the specimens.

The prepared amalgam-composite specimens were stored in 100% humidity at room temperature for 24h before (25) the SBSt (Figure 1C).

SHEAR BOND STRENGTH TEST

Amalgam-composite specimens were mounted in the custom-made jig. A universal testing machine (Shimadzu Corporation, Kyoto, Japan) with chisel format shear load applier was loaded at crosshead speed of 0.5 mm/min to perform SBSt. The force was applied to the amalgam-composite adherent interface until fracture occurred (Figure 1D), and the value was recorded as Newtons (N). The load values were expressed into megapascals (MPa) by dividing recorded peak load at the failure to the adhesive surface area (mm²).

In addition to quantitative analysis, failure modes were evaluated separately by two evaluators (FAS and DG) using a digital microscope (Shuttleplex p-400r, Nikon, Tokyo, Japan) at 40× magnification. Failure modes were classified as: (a) adhesive (b) mixed, (c) cohesive within the ceramic material, or (d) cohesive within the repair material.

STATISTICAL ANALYSIS

The SBS values of the tested groups were normally distributed according to Kolmogorov-Smirnov test. Two-way ANOVA was used to analyze the means of each group. Multiple comparisons between the groups were performed using Tukey's post hoc tests because statistically significant differences were noted ($p < 0.05$).

RESULTS

The results of two-way ANOVA for SBS values are shown in Table 3; means (SDs) of SBS values, group differences and failure modes are listed in Table 4. The lowest SBS values were calculated for Group Control, this difference was significant for Group FLD ($p < 0.05$) (Table 4). Mean SBS values of Group FLD was higher than that of Group APFLD ($p \geq 0.05$), while mean SBS value of Group C was lower than Group APC ($p \geq 0.05$) (Table 4). Group APControl had higher SBS values than Group Control. This difference was significant ($p < 0.05$) (Table 4).

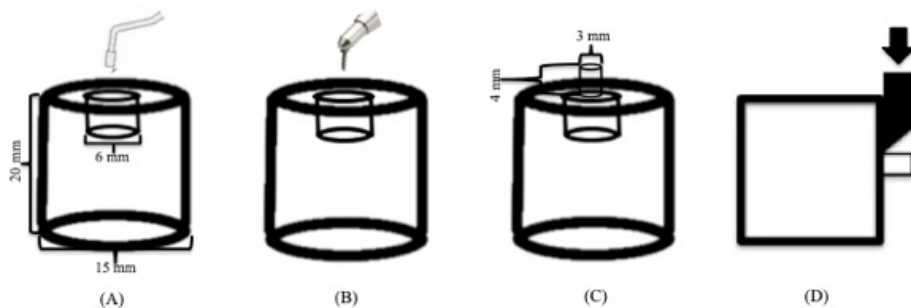


Figure 1. A) Amalgam was condensed into a 6×2 mm hole in PMMA; B) amalgam surface was sandblasted with 50 μm Al₂O₃ using an intraoral sandblaster; C) 4×3 mm composite build-ups were prepared for the investigated repair materials; D) A shear load at crosshead speed of 0.5 mm/min was applied to the amalgam-composite adherent interface until fracture occurred.

Table 1. Materials and their compositions.

Material and manufacturer	Composition
Fusio Liquid Dentin, Self-adhesive flowable composite, Pentron Clinical, USA	UDMA, TEGDMA, HEMA, 4-META
Constic, Self-adhesive flowable composite, DMG, Germany	Bis-GMA, EBADMA, UDMA, HEMA, TEGDMA, HDMA, MDP
Aelite All Purpose Body, microhybrid composite, Bisco, Schaumburg, IL, USA	Bis-EMA, TEGDMA, glass filler, amorphous silica
All Bond Universal, Universal bonding resin, Bisco, Schaumburg, IL, USA	MDP, dimethacrylate resins, HEMA, ethanol, water, initiator
RubyCap Ng, Capsule Amalgam, Rubydent, İnci Dental, İstanbul, Turkey	50% Ag, 30% Sn, 20% Cu
Alloy primer, Bisco, Schaumburg, IL, USA	BPDM, HEMA, ethanol, MDP

Bis-GMA: bisphenol A glycidylmethacrylate, EBADMA: ethoxylated bisphenol A dimethacrylate, HDMA: 1,6-hexanediol dimethacrylate, HEMA: hydroxyethyl methacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, TEGDMA: triethylen glycol dimethacrylate, UDMA: urethane dimethacrylate, 4-META: 4-methacryloxyethyl trimellitic acid, Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate, Ag: silver, Sn: tin, Cu: copper, BPDM: dimetakrylan bifenylyu.

Table 2. Experimental groups and repair protocols.

Group name	Repair protocol
C	Constic
FLD	FLD
Control	Universal Bonding+Universal Composite
ApC	AP+Constic
ApFLD	AP+FLD
ApControl	AP+ Universal Bonding+Universal Composite

Table 3. Two-way ANOVA results.

Source	SS	df	MS	F	P
Composite	178.557	2	89.278	4.077	0.025
Alloy Primer	54.329	1	54.329	2.481	0.124
Composite x Alloy Primer	245.475	2	122.738	5.605	0.008
Error	788.333	36	21.898		
Total	10068,569	42			

Table 4. Mean SBS values (MPa) (Mean \pm SD) and failure modes of the Groups.

Groups		SBS			Failure Modes		
Composite	AP	Mean \pm SD	Lower bound	Upper bound	Adhesive	Cohesive	Mixed
C	-	12.21 \pm 4.02a, #	8.48	15.92		20%	80%
	+	16.47 \pm 6.96A, #	10.04	22.91		50%	50%
FLD	-	19.26 \pm 2.78b, *	16.68	21.83		60%	40%
	+	14.87 \pm 4.55A, *	10.65	19.08		50%	50%
BComp	-	8.54 \pm 5.13a, ®	3.81	13.29	100%		
	+	15.49 \pm 3.45A, ©	12.30	18.69	10%	40%	50%

Lower superscript letters (a, b): different letters in the same column indicate a significant difference in mean SBS scores for AP- different composite groups ($p < 0.05$). Uppercase superscript letters (A, B): different letters in the same column indicate a significant difference in mean SBS scores for AP+ different composite groups ($p < 0.05$). The symbols (#*®©): different letters for the AP +/- groups of the same composite material indicate a significant difference ($p < 0.05$).

DISCUSSION

Total replacement of amalgam restorations could cause excessive tooth tissue loss and pulpal damage (26,27). Repair is recommended instead of total replacement for damaged amalgam restorations. Several repair techniques, including airborne particle abrasion, AP application, metal primer application, use of opaquers, use of fibre sheets, use of bonding systems, silica coating, silanization on composite and amalgam bonding have been reported in the dental literature (1, 6-9).

This *in vitro* study aimed to evaluate the efficiencies of self-adhesive composites with or without AP application for amalgam repair, in terms of SBS. The highest SBS values were observed in Group FLD (19.26 \pm 2.78) ($p < 0.05$). Composite materials influenced the SBS values according to two-way ANOVA results ($p < 0.05$). Therefore, the first null hypothesis that there would be no differences among the SBS values between the different composite materials and amalgam was rejected. According to results of two-way ANOVA, AP application did not affect the SBS values ($p \geq 0.05$). Therefore, the second null hypothesis that AP application would not affect the SBS values between the composite materials and amalgam was accepted.

Roughening amalgam surface by air abrasion increases the bond strength between amalgam and repair materials (6,21). In the present study, air abrasion was performed in all of the prepared amalgam specimens to create standardized and rough adhesive surfaces. The adhesive system is important for the roughened amalgam surface (28).

The minimum accepted SBS value between resin-based materials and substrate is 5 MPa according to ISO 10477 (29). In the present study, the SBS values were higher than 5 MPa for all of the investigated groups, despite the lower bound value was 3.81 MPa for Group Control.

C and FLD are self-adhesive composites and thus do not require adhesive applications on the surface (11,12). The present results show that when AP was not applied to the amalgam surface, FLD (which chemically bonds to hydrophobic amalgam surface via 4-META) (21,22) caused higher SBS values than C (which contains MDP monomers) (30) ($p \geq 0.05$). Although making exact comparisons between the results is impossible, the obtained SBS value for Group FLD in the present study was similar to that obtained by Machado *et al.* (21) for the bonding of 4-META containing adhesive to an amalgam surface (16.82 \pm 3.60).

AP could condition both noble and base alloys. AP bonds to an alloy via hydrophilic carboxylate groups, wherein the hydrophobic component interacts with the resin composite. MDP acts as the functional monomer for base alloys (23,24). Balkaya *et al.* (8) have demonstrated that the use of AP before the use of universal adhesives increases the microshear bond strength to amalgam. The present results except those for FLD, agree with the results reported by Balkaya *et al.* (8). This can be explained by the positive interaction of MDP in the C and All Bond Universal with MDP in AP. The SBS value of Group FLD was higher than that of Group APFLD ($p \geq 0.05$). This result may have been caused by the negative relationship between 4-META in FLD and MDP monomer in AP.

In the present study, calculated SBS values for APControl were slightly lower than a previous finding (22.60 ± 4.30) (9); but considerably higher than another finding (4.40 ± 2.00) (6). Different types of amalgam materials were used in those studies. According to Ozcan *et al.* (6), the type of amalgam may influence the results.

Korkmaz *et al.* (31), have investigated the repair bond strength of resin composites with nickel-crom (NiCr) and titanium (Ti) alloys. They reported SBS values of 7.96 ± 1.19 and 6.98 ± 1.70 MPa for the bonding of resin composites with sandblasted dental casting NiCr and Ti alloys, respectively. Although it is not possible to make exact comparisons between the results because of different metallic substrates were used, the calculated SBS value in the present study for Group Control was similar to those reported by Korkmaz *et al.* (30).

Specimens were not subjected to thermal aging in the present study. This is a limitation and long term interactions between self-adhesive composites and amalgam should be investigated in further studies.

CONCLUSIONS

Within the limitations of the present research, it could be concluded that the investigated self-adhesive composites are strong and time saving alternatives for the purpose of amalgam repair, on the basis of SBS values. AP application increased the SBS values between C and amalgam, thus C should be used with AP when repairing amalgam restorations. FLD seems to be a better alternative to the UB+UC and C because of not requiring AP application and reducing the steps of amalgam repair.

CONFLICT OF INTEREST

The authors of the present study declare that they have no conflict of interest.

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None.

ETHICAL APPROVAL

This article does not contain any studies with human participants or animals performed by any of the authors.

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