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Micro-shear bond strength of universal adhesives used for amalgam repair with or without Alloy Primer

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Abstract

Aim: The aim of this study was to investigate the adhesive performance of three different universal adhesives to repair aged amalgam by composite resins with or without Alloy Primer.

Materials and Methods: Sixty amalgam samples were prepared, aged, and randomly divided into 12 main groups according to adhesive procedures used. Composite buildups were placed on amalgam surfaces. After micro-shear bonding test, the fracture surfaces were examined under the scanning electron microscopy statistical analysis was performed using two-way analysis of variance and Tukey's *post hoc* tests.

Results: Without use of Alloy Primer, all of the universal adhesives provided similar bond strength values with conventional adhesives ($P > 0.05$); however, an Alloy Primer significantly increased the bond strength values of universal adhesives ($P < 0.05$). No significant difference in bond strength values was noted for conventional adhesives with or without Alloy Primer ($P > 0.05$) except for Clearfil SE Bond ($P < 0.05$).

Conclusions: Within the limitations of this study, it can be concluded; using Alloy Primer before universal adhesives increased the bond strength significantly.

Keywords: Amalgam repair; composite; current adhesives; micro-shear bond strength; universal adhesive

INTRODUCTION

Replacement of defective restorations is sometimes required in general dental practice due to secondary caries, marginal defects, cuspal fracture, and insufficient marginal integrity.[1,2] Repairing of defective areas instead of replacement protects both the dental tissues and restorative material because less amount of hard tissues are removed.[3] The repairing of an aged restoration causes less destruction compared to the replacement of restoration, with a reduced risk of tooth fracture and pulp damages.[4] The previous studies showed that alternative treatments to replacement of defective restorations, such as marginal sealing, refurbishment, and repair improved the clinical properties and increased their quality and longevity significantly for defective restorations with minimal interventions.[5,6]

Repair is the first order among minimally invasive treatment methods, especially in recent years. Instead of completely removing the old restoration in this treatment option, the defective area is completely removed. The retentive areas are formed by applying various roughening processes to the amalgam surface and repair with resin composite using dentin or amalgam bonding systems.[7-10]

Different repair methods are available in the literature, including mechanical and/or chemical bonding techniques.[9-12] Mechanical methods include roughening the amalgam surface, forming retentive areas and grooves, or

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placing amalgam pins.[10] It has been suggested that chemical bonding may be achieved by the use of multipurpose adhesive systems or agents called metal primers.[13] In addition, it has been argued that it is necessary to provide surface retention by spraying aluminum oxide particles with compressed air (air abrasion) for good mechanical bonding.[8,14,15] However, there are conflicting conclusions about the real chemical bonding between amalgam and resin composites.[16]

Recently, a new category of adhesives called multimode or universal adhesives have been marketed. They can be used in etch-and-rinse, self-etch, or selective-etch mode depending on the specific clinical situation and preferences of the practitioner.[17,18]

These adhesives contain a phosphate monomer (methacryloyloxydecyl dihydrogen phosphate [MDP]) and silane in addition to conventional functional monomers. The phosphate esters, such as 10-MDP form in all current universal adhesive systems, have many positive attributes including the potential to bond chemically to metals, zirconia, and to tooth tissues through the formation of nonsoluble Ca^{++} salts.[17] In addition, it is stated that universal adhesives can be used not only to bond to dentin and enamel but also on substrates such as zirconia, noble and nonprecious metals, composites, and various silica-based ceramics, as adhesive primers. Hence, this will provide adhesion to these surfaces without the need for dedicated and separately placed primers such as silane and various products marketed as metal and zirconia primers.[17] Metal primers are used in the surface conditioning of both noble metals and base metals, and it is suggested that these agents increase connectivity. Metal primers are bound to the alloy by hydrophilic carboxyl groups and are linked to the resin component by the exposed hydrophobic parts. Choo *et al*. reported that a metallic primer, Alloy Primer, was bound to the noble metals by the 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione functional monomer, and to the base metals by the 10-MDP monomer.[19] MDP forms a strong chemical bonding with oxide layer on the surface of the alloy and thus forms reliable bond of the resin to the alloy.[20]

In literature, there was no study on adhesive performance of universal adhesives in repairing of damaged or aged restorations. For this reason, the aim of this study was to evaluate the adhesive performance of universal adhesives to repair aged amalgam restorations by composite resins. The null hypothesis of present study was that the there is no difference between universal adhesives when used with or without Alloy Primer to repair aged amalgam restorations.

MATERIALS AND METHODS

The brand name, manufacturer, chemical composition, and batch number of the materials used in the current study are listed in Table 1.

Preparation of amalgam specimens

Sixty disc-shaped amalgam specimens, 6 mm diameter 2 mm thickness were prepared in polyvinyl chloride (PVC) rings for this study. The PVC rings were placed on a glass microscope slide, the amalgam was condensed, a second microscope slide pressed firmly onto the amalgam to remove excess. The amalgam used in this study was a nongamma 2, high-copper, dispersed phase amalgam alloy composed of a mixture of spherical, and irregular particles. The each prepared amalgam sample was mounted on a PVC ring filled with acrylic resin (Imicryl, Konya, Turkey). Specimens were allowed to set for 24 h in an incubator at 37°C. The upper surface of each specimen was ground finished to 400-grit silicon carbide abrasive (Struers RotoPol 11, Struers A/S, Rodovre, Denmark). The specimens were then aged using 5000 thermal cycles between 5°C and 55°C. The surface of all samples was sandblasted with 50 µm Al_2O_3 (KaVo RONDOflex2015 Powder; KaVo Dental GmbH, Biberach, Germany) using a intraoral sandblaster (Kavo RONDOflex Plus 360; KaVo Dental GmbH) at a pressure of 2 bar from a distance of 10 mm for 10 s.

For the half of the groups, Alloy Primer (Clearfil, Kuraray Medical, Inc., Okayama, Japan) was applied to surface of aged amalgams according to manufacturers' instructions [Table 1].

Experimental groups and adhesive applications

The amalgam specimens were divided into 12 main groups according to adhesive applications as mentioned belove:

- Group 1 – Single Bond Universal (3M ESPE)
- Group 2 – Alloy Primer (Kuraray) + Single Bond Universal (3M ESPE)
- Group 3 – Futurabond U (Voco)
- Group 4 – Alloy Primer (Kuraray) + Futurabond U (Voco)
- Group 5 – Clearfil Universal (Kuraray)
- Group 6 – Alloy Primer (Kuraray) + Clearfil Universal (Kuraray)
- Group 7 – Single Bond 2 (3M ESPE)
- Group 8 – Alloy Primer (Kuraray) + Single Bond 2 (3M ESPE)
- Group 9 – Clearfil Tri-S Bond (Kuraray)
- Group 10 – Alloy Primer (Kuraray) + Clearfil Tri-S Bond (Kuraray)
- Group 11 – Clearfil SE Bond (Kuraray)
- Group 12 – Alloy Primer (Kuraray) + Clearfil SE Bond (Kuraray).

Preperation of composite cylinders

Three cylindrical composite buildups (Charisma, Heraeus Kulzer) were created on surfaces of five amalgam discs in each group thus 15 test samples were obtained (n = 15) using tygon tubes (0.75 mm internal diamaterx1 mm length) (Tygon, Norton Performance Plastic Co, Cleveland,

Table 1: Materials, composition, and application modes used in present study

Materials	Composition	Application mode	
Charisma Heraeus Kulzer, Hanau, Germany Batch# 010417A	Bis-GMA, TEGDMA, barium aluminum fluoride glass, silicium dioxide	1. Apply resin composite to the surface 2. Light polymerize for 20 s	
Clearfil SE Bond Kuraray, Osaka, Japan Primer Batch# 01041A Bond Batch# 01552A	Primer: MDP, HEMA, hydrophilic dimethacrylate, dl-camphorquinone, N, N-diethanol-p-toluidine, water. Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N, N-diethanol-p-toluidine, silanated colloidal silica	Without Alloy Primer Apply primer to the surface and leave in place for 20 s Dry with air stream to evaporate the volatile ingredients Apply bond to the surface and then create a uniform film using a gentle air stream light polymerize for 10 s	With Alloy Primer Apply alloy primer to surface Apply primer to the surface and leave in place for 20 s Dry with air stream to evaporate the volatile ingredients Apply bond to the surface and then create a uniform film using a gentle air stream Light polymerize for 10 s
Adper Single Bond 2 3M ESPE, St. Paul, MN, USA. Batch# N151635	HEMA, Bis-GMA, ethanol, dimethacrylate, methacrylate functional copolymer of polyacrylic and polytaconic acid, water, photoinitiator	Without Alloy Primer Apply etchant for 15 s Rinse for 10 s Blot excess water Apply 2-3 consecutive coats of adhesive for 15 s with gentle agitation Gently air dry for 5 s Light polymerize for 10 s	With Alloy Primer Apply etchant for 15s Rinse for 10 s and gently air-dry Apply Alloy Primer to surface Apply 2-3 consecutive coats of adhesive for 15 s with gentle agitation Gently air dry for 5 s Light polymerize for 10 s
CLEARFIL TRI-S BOND Kuraray Medical Inc., Okayama, Japan Batch# 000004	MDP, Bis-GMA, HEMA, colloidal silica, ethanol, water, dl-camphorquinone, Initiators, accelerators	Without Alloy Primer Apply adhesive for 20 s Air dry for >5 s Light polymerize for 10 s	With Alloy Primer Apply Alloy Primer to surface Apply adhesive for 20 s Air dry for >5 s Light polymerize for 10 s
Futurabond U Voco, Cuxhaven, Germany Batch# 1415274	Liquid 1: Acidic adhesive monomer, HEMA, BISGMA, HEDMA, UDMA, catalyst. liquid 2: ethanol, ınitiator, catalyst.	Without Alloy Primer Mix and stir thoroughly both liquids with the single tim applicator Apply the adhesive homogenously to the surface and rub for 20 s using the single tim Dry off the adhesive layer with dry, oil-free air for at least 5 s Light cure the adhesive layer for 10 s	With Alloy Primer Apply Alloy Primer to surface. Mixture and stir thoroughly both liquids with the Single tim applicator Apply the adhesive homogenously to the surface and rub for 20 s using the single tim Dry off the adhesive layer with dry, oil-free air for at least 5 s Light cure the adhesive layer for 10 s
Single Bond Universal Adhesive 3M ESPE, Neuss, Germany Batch# 535812	10-MDP, Vitrebond, Copolymer, HEMA, BISGMA, dimethacrylate resins, filler, silane, initiators, ethanol, water	Without Alloy Primer Apply the adhesive with the applicator to the entire surface and rub for 20 s Dry gently for about 5s until it no longer moves and the solvent has evaporated completely Harden the adhesive with a curing light for 10 s	With Alloy Primer Apply Alloy Primer to surface Apply the adhesive with the applicator to the entire surface and rub for 20 s Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely Harden the adhesive with a curing light for 10 s
Clearfil Universal Bond Kuraray Noritake Dental Inc., Okayama, Japan Batch# 2B0005	Bis-GMA, HEMA, ethanol, 10-MDP, hydrophilic aliphatic dimethacrylate, colloidal silica, camphorquinone, silane coupling agent, accelerators, initiators, water	Without Alloy Primer Apply Clearfil Universal Bond to the surface with the applicator brush and rub it in for 10 s Dry sufficiently by blowing mild air for >5 s until the adhesive shows no movement. Use a vacuum aspirator to prevent the adhesive from scattering Light cure for 10 s	With Alloy Primer Apply Alloy Primer to surface. Apply Clearfil Universal Bond to the surface with the applicator brush and rub it in for 10 s Dry sufficiently by blowing mild air for >5 s until the adhesive shows no movement. Use a vacuum aspirator to prevent the adhesive from scattering Light cure for 10 s
Alloy Primer Kuraray, Okayama, Japan Batch# 2P0046	Methacryloyloxydecyl dihydrogen phosphate (MDP), 6-(4-vinyl benzyl-n-propyl) amino-1,3,5-triazine-2,4-dithio (VBATDT)	Apply with a microbrush	
RubyCap Ng Capsule Amalgam Rubydent, İnci dental, İstanbul, Turkey Batch# P464	50% Ag, 30% Sn, 20% Cu	Triturate 7 s with high-power amalgamator	

Bis-GMA: Bis-phenol A diglycidylmethacrylate, HEMA: 2-hydroxyethyl methacrylate, TEGDMA: Triethylene glycol dimethacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, UDMA: Urethane dimethacrylate, HEDMA: 1,6-hexanediol dimethacrylate, Al_2O_3: Aluminum oxide, VBATDT: 6-(4-vinylbenzyl-npropyl) amine-1,3,5-triazine-2,4dithione

OH, USA) under pressure with nonsticky hand instrument and bulk-cured for 20 s using a light-emitting diode (LED) light-curing unit (Elipar FreeLight 3, St. Paul, 3M ESPE, MN, USA) at 1200 mW/cm2 output [Figure 1]. The output energy of the LED-curing device was measured periodically with a radiometer (Demetron/Kerr, Danbury, CT, USA). The failed samples before the test were replaced with new samples.

Micro-shear bond strength test

After storage in distilled water (37°C/24 h), the Tygon tubes were removed using a scalpel, and the specimens were subjected to a universal testing machine (Instron, Model 4444, Instron Corporation, Canton, MA, USA). A 0.25 mm thick wire loop was placed around the composite cylinders contacting semicircularly. Microshear bond test was performed at a crosshead speed of 0.5 mm/min. The microshear forces were recorded in Newtons (N) and calculated as megapascals (MPa) by dividing to the bonding area (mm^2).

Scanning electron microscopy analysis of debonded surfaces

All fracture surfaces of debonded samples were coated with gold-palladium and examined under scanning electron microscopy (SEM) (LEO-440, Zeiss, Cambridge, England). Failure modes were classified as follows: adhesive failure (A); at resin-amalgam interface, mixed failure (M); where adhesive failure occurred with a thin layer of composite material remaining on the amalgam surface, cohesive failure in composite resin material (C).

Statistical analysis

Statistical analysis was accomplished using SPSS 10.0 software (SPSS Inc., Chicago, IL, USA). Kolmogorov–Smirnov analysis was used to test normality and decided to perform parametric test for analysis of the data. For this purpose, two-way analysis of variance (ANOVA) and Tukey's *post hoc* test were used (α =0.05). Fracture modes were analyzed using the Chi-square test (α =0.05). The pretesting failures were not included in the statistical analysis.

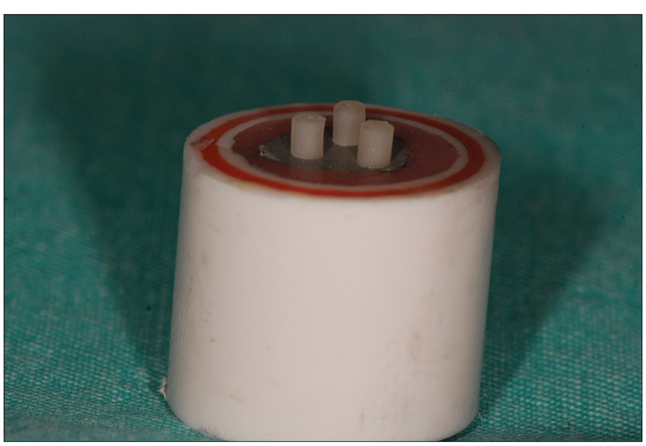

Figure 1: Preparation of sample

RESULTS

The results of the micro-shear bond strength tests are presented in Table 2.

According to two-way ANOVA, using of Alloy Primer before adhesive application significantly increased the bond strength of all universal adhesives ($P = 0.00$). Among universal adhesives used with Alloy Primer, Single Bond Universal showed the highest mean bond strength values (20.2 ± 5.9 MPa) followed by Futurabond U (18.2 ± 5.9 MPa), Clearfil Universal (18.1 ± 4.3 MPa), respectively. However, these adhesives did not show statistically significant difference when compared to each other ($P > 0.05$). In current adhesives used with Alloy Primer, Clearfil SE Bond was found statistically higher (17.1 ± 5.1 MPa) than own control group (11.6 ± 2.4) ($P < 0.05$). Other two conventional adhesives Single Bond 2 and Clearfil Tri-S Bond did not increase the bond strength when used with Alloy Primer (P = 1,00).

SEM analysis of debonded surfaces showed that adhesive failing mode was most common for all groups. Cohesive failing mode was rarely seen in groups [Figure 2 and Table 2]. Chi-square test showed that no statistical differences ($P > 0.05$) were reported among all tested groups in terms of failing modes.

DISCUSSION

Since the conventional adhesives required time-consuming additional surface procedures to repair fractured amalgam restorations, in repair protocol of amalgam restorations, using universal adhesives may be useful to increase the bond strength of composite resins to amalgam surfaces. In the present study, the effect of universal adhesives on bond strength of composite resin to amalgam was tested. It was found that universal adhesives did not increase the bond

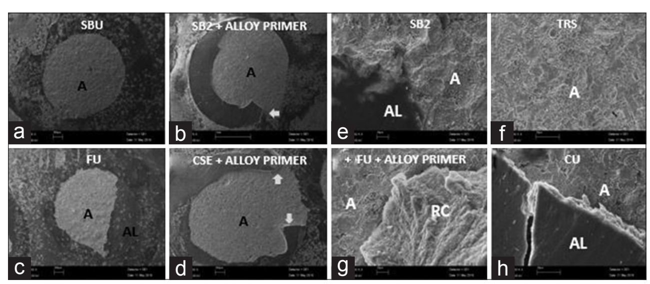

Figure 2: Scanning electron microscopy images of debonded surfaces (×100 and ×2500). (a) Adhesive failing: Completely removed adhesive layer, (b and d) mix failing: Small amount of remained adhesive layer and composite resin (white arrows), (c) adhesive failing: Adhesive layer in the half of debonded surface. (e-h) Scanning electron microscopy images of debonded surfaces (×2500). SBU: Single Bond Universal, FU: Futurabond Universal, CSE: Clearfil SE Bond, CU: Clearfil Universal, TSB: Tri-S Bond, SB2: Single Bond 2, A: Amalgam, AL: Adhesive layer, RC: Resin composite

Table 2: Mean, upper, lower bond strength values, standard deviation, and failure modes of groups

	Bond strength (MPa)			Failure modes, n (%)			
	Mean±SD	Upper	Lower	Adhesive	Cohesive	Mixed	PTF
SBU	10.8±2.9	17.1	5.7	13 (86.7)	0	0	2 (13.3)
SBUa	20.2±5.9	30.0	12.3	14 (93.3)	0	1 (6.7)	0
CU	11.7±2.1	14.3	8.1	14 (93.3)	0	0	1 (6.7)
CUa	18.1±4.3	24.6	11.3	14 (93.3)	0	0	1 (6.7)
FU	11.8±3.4	16.5	7.3	15 (100)	0	0	0
FUa	18.2±5.9	28.4	9.9	13 (86.7)	0	1 (6.7)	1 (6.7)
CSE	11.6±2.4	15.9	6.7	15 (100)	0	0	0
CSEa	17.1±5.1	28.9	9.4	13 (86.7)	0	1 (6.7)	1 (6.7)
TSB	10.0±3.5	15.7	4.3	15 (100)	0	0	0
TSBa	11.2±3.6	17.6	6.4	13 (86.7)	0	1 (6.7)	1 (6.7)
SB2	9.2±2.9	14.3	4.7	14 (93.3)	0	0	1 (6.7)
SB2a	10.3±3.6	16.3	4.0	14 (93.3)	0	1 (6.7)	0

SBU: Single Bond Universal, SBUa: Single Bond Universal with Alloy Primer, CU: Clearfil Universal, CUa: Clearfil Universal with Alloy Primer, FU: Futurabond Universal, FUa: Futurabond Universal with Alloy Primer, CSE: Clearfil SE Bond, CSEa: Clearfil SE Bond with Alloy Primer, TSB: Tri-S Bond, TSBa: Tri-S Bond with Alloy Primer, SB2: Single Bond 2, SB2a: Single Bond 2 with Alloy Primer, PTF: Pretesting failure, SD: Standard deviation

strength values without use of Alloy Primer, but when used Alloy Primer before adhesives applications, it improved the bond strength of composite. For this reason, the null hypothesis of present study was rejected.

It is difficult to make direct comparisons with the results of other studies due to design and new adhesives used in this study. However, the previous studies have shown that the roughening of the amalgam surface increased the surface area and mechanically locked the adhesive to the amalgam.[21-23] For this reason, air abrasion was applied to the amalgam surfaces in our study.

In a study using a conventional adhesive (Scotchbond Multipurpose Adhesive) by Ozcan *et al*., it was reported that Alloy Primer, air abrasion with Alloy Primer, silica coating, and silanization showed similar bond strength.[24]

In a microleakage study by Cehreli *et al*., it was demonstrated that using a self-etch adhesive system (Clearfil SE Bond) accompanying Alloy Primer did not significantly improve the sealing of restorative complex.[25] Unlike, in the present study, the use of Alloy Primer with Clearfil SE Bond increased the micro-shear bond strength.

The manufacturers claim that universal adhesives can be bonded to any substrate such as zirconia, noble and nonprecious metals, composite resins, and various silica-based ceramics without the need for a separate silane or primer application.[17] However, there was no difference between universal adhesives and conventional adhesives when used without Alloy Primer in the present study. However, when Alloy Primer was used before adhesive protocol, it significantly increased the bond strength of universal adhesives, while the bond strength of conventional adhesives did not affect except for Clearfil SE Bond. This can be explained by the positive interaction of 10 MDP in the universal adhesives (Single Bond Universal, Futurabond U, Clearfil Universal) and Clearfil SE Bond (in both primer and bond), with 10 MDP in the Alloy Primer. In addition, Clearfil Tri-S Bond contains MDP, but its bond strength did not increase when Alloy Primer was used. This cannot explain within the limitations of this study; further research needs to be done.

In the present study, a microshear test was used to evaluate the bond strength of materials tested. This technique has some superiorities because it is easier, does not need to cut sample after bonding of the composite resin as in "micro-tensile," and this means bonding strength will not be reduced due to the slicing procedure.[26] More than one sample can be applied solely to one substrate surface; therefore, it requires a fewer number of total samples for the study. The SEM examination of a larger number of samples at the same time may be easier than microtensile and macro-shear tests. The bonding area is smaller than that of the other shear bond test method, and this may result in more "adhesive" type of failings and consequently more valid assessment. The "cohesive" fractures do not represent the clinically relevant failure mechanism in real cavities. This particular problem can be prevented with micro-shear testing because the predominant failure is also "adhesive" during the investigation. Similarly, in the present study, SEM images showed that most common failing type was the adhesive failing.

The present study was conducted in an *in vitro* environment and several factors such as oral fluids, occlusal forces, and thermal changes were not taken into account, and only amalgam samples were tested. Therefore, further *in vivo* and *in vitro* studies are needed to validate the results of the present study.

CONCLUSIONS

Within the limitations of the present study, the following can be concluded:

To repair a fractured amalgam, using an Alloy Primer before application of universal adhesives increases the bond strength of adhesives used in the present study.

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Nil.

Conflicts of interest
There are no conflicts of interest.

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