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Effects of Different Surface Conditioning Methods on the Bond Strength of Composite Resin to Amalgam

M Özcan • C Koolman • A Aladag
M Dündar

Clinical Relevance

Repair of defective amalgam restorations, especially including cusp fractures, is clinically time-saving and is not invasive. Surface conditioning with silicacoating and opaquer treatment before composite resin application adds to increased bond strengths of such restorations.

SUMMARY

Repairing amalgam restorations with composite resins using surface conditioning methods is a conservative treatment approach. This study investigated the effects of different conditioning methods that could be used for repair of amalgam fractures. Amalgam (N=96) was condensed into cavities within autopolymerizing polymethylmethacrylate (PMMA), and the exposed surface of each specimen (diameter, 6 mm; thickness, 2 mm) was ground finished. The specimens were randomly divided into nine experimental groups (n=12 per group), depending on the conditioning method used. The control group had natural central incisors with amalgam (n=12). The combination of the following conditioning methods was tested: silicacoating (Sc), sandblasting (Sb), metal primers, coupling agents, fiber (Fb) application, and opaquers (O). Five types of

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*Mutlu Özcan, Dr Med Dent, PhD, professor and research associate, University of Zurich, Center for Dental and Oral Medicine, Dental Materials Unit, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland

Carolien Koolman, DDS, private practice, Groningen, The Netherlands

Akin Aladag, DDS, PhD, assistant professor, University of Ege, Department of Prosthodontics, Izmir, Turkey

Mine Dündar, DDS, PhD, associate professor, University of Ege, Department of Prosthodontics, Izmir, Turkey

*Corresponding author: University of Zurich, Center for Dental and Oral Medicine, Dental Materials Unit, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Plattenstrasse 11, CH-8032, Zurich, Switzerland; e-mail: mutluozcan@hotmail.com

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silanes, metal primers, or adhesives (Visiobond [V], Porcelain Photobond [PP], Alloy Primer [AP], Unibond sealer [Us], ESPE-Sil [ES]), and four opaquers, namely, Clearfil St Opaquer (CstO), Sinfony (S), Miris (M), and an experimental Opaquer (EO-Cavex), were used. The groups were as follows: group 1, Sc+ES+S+V; group 2, Sc+ES+CstO+V; group 3, Sc+ES+M+V; group 4, Sc+ES+EO+V; group 5, Sb+AP+S; group 6, Sb+AP+PP+CstO; group 7, Sc+ES+S+Fb+V+Fb; group 8-control, SC+ES+V; and group 9, Etch+Sc+ES+S+Us. One repair composite was used for all groups (Clearfil Photo Bond Posterior, Kuraray, Tokyo, Japan). Shear bond strengths (SBSs) (MPa ± SD) were evaluated after 5 weeks of water storage (analysis of variance [ANOVA], Tukey honestly significant differences [HSD], α=0.05). Group 1 exhibited significantly higher values (35.5 ± 4.1) than were seen in group 4 (19.4 ± 8.9), group 6 (19.1 ± 7.8), and group 8 (20.1 ± 4.1) (p<0.05). Group 9 exhibited significantly lower values (8.3 ± 3.4) than were noted in groups 1 to 3 (35.5 ± 4.1; 27 ± 12.5; 24.4 ± 5.1, respectively) (p<0.05). Group 7 (16.4 ± 5.9) showed significantly lower values than were observed in group 1 (35.5 ± 4.1) (p<0.05). Surface conditioning techniques affected the bond strengths of composite adhesion to amalgam. Experimental opaquer exhibited lower values. Leaving a small border of enamel around the restoration decreased the bond strength.

INTRODUCTION

Defective dental amalgam restorations are usually treated by replacement with a restorative material.[1] As a viable conservative technique, the repair of defective areas has been suggested because it is simple and quick.[1-3] Repair also preserves the tooth, as it is difficult to remove a restoration without removing an integral part of the tooth itself. If not necessary, sound enamel and dentin removal is avoided, strength is maintained, and longevity of the tooth is increased.[1] Cusp fractures or fractures within amalgam restorations are usually due to impact load, fatigue load during mastication, microdefects in the amalgam, secondary caries, technical errors during cavity preparation, incompatible thermal expansion coefficients, history of endodontic treatment, insufficient sound tooth material available such as undermining cusps, number of surfaces restored, or occlusal prematurity.[2,3]

A few reports in the literature describe the incidence of tooth fracture.[2,4,5] In one study, the rate of cusp fracture was reported to be 5 teeth per 100 adults per year.[4] In another study, the incidence of cusp/incisal edge fracture per 100 subjects was reported as 14 teeth over the course of 2 years.[5] Cusp fractures are a significant dental health problem. In many cases, they are caused by the conventional preparation technique required for amalgam restorations, wherein large undercuts are needed to attain macromechanical retention. Continuous occlusal loading of such weakened cusps could result in horizontal crack formation and cusp fractures.[6] In a previous study on the incidence of cusp fracture over a 3-month period, molars were more frequently registered with cusp fractures than were premolars.[2] Although maxillary molars presented a greater number of fractures of buccal cusps, mandibular molars presented more fractures of the lingual cusps. Mastication was most frequently reported as the cause of fracture.

The more surfaces that were restored and/or the wider the isthmus, the greater was the risk of cusp fracture.[2,7] Thus, it is likely that the restorative status of the tooth has an influence on the incidence of fracture. Most fractures were observed in the supragingival location, suggesting that fractures could be restored easily.[3] Although sparse literature on the subject has been published, repair of a restoration is more cost beneficial than total replacement, wherever appropriate.[8] A properly and skillfully manipulated amalgam restoration that replaces one or two cusps or restores an entire occlusal surface may serve for many years. However, in visible areas of the mouth, an amalgam restoration may present an esthetic problem.[9] Fracture and the esthetic appearance of amalgam restorations present a clinical problem and have created a demand for the development of a simple, efficient intraoral repair method that does not necessitate removal and remaking of the entire restoration.

Recently, veneering the amalgam restoration with resin materials was done in an attempt to solve the problem.[10] Various repair techniques have been suggested in the literature, many of which are based on mechanical and/or chemical means.[9,11] Mechanical means require threading of pins to provide retention for resin composite veneers.[11] Chemical means, on the other hand, use multipurpose adhesive agents.[1,12] Several studies have shown that airborne particle abrasion modifies the metal surface and provides micro-roughness that is essential for mechanical bonding,[1,10,11,13] while primers and adhesives form ionic bonds with metal oxides or with the active metal compounds of the amalgam.[13] However, the existence of a true chemical bond between amalgam and resin composites is controversial.[9]

The literature contains many reports on bonding of resin composites to alloy surfaces,[14] but these methods have not been investigated for the purpose of bonding composites to amalgam. Although the concept of veneering composites to amalgam restorations is not new to restorative dentistry, there seems to be no consensus in the literature regarding the best method for repairing amalgam restorations. Therefore, the objective of this study was to evaluate the effects of various surface conditioning methods, silane coupling agents, metal primers, and fibers, in combination with various opaquers, on the bond strength of resin composite to amalgam.

MATERIALS AND METHODS

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

Specimen Preparation

Eight experimental groups (n=12) used one type of fresh amalgam (Cavex Amalgam, Cavex Holland BV, Haarlem, The Netherlands). The amalgam used was a non-gamma 2, high-copper, dispersed phase amalgam alloy composed of a mixture of lathe-cut particles and microfine eutectic spherical particles (69.2% Ag, 18.6% Sn, 11.9% Cu, 0.3% Zn). It was triturated according to the manufacturer's recommendations. The control group (n=12) consisted of natural central incisors. The amalgam was condensed with a hand instrument into a cylindrical cavity (diameter, 6 mm; thickness, 2 mm) prepared in autopolymerizing polymethylmethacrylate (PMMA) (Palapress, Vario, Hereaus Kulzer, Wehrheim, Germany) until it was slightly overfilled (N=108). One day after condensation, the exposed surface of each specimen was ground finished to 400-grit silicone carbide abrasive (Struers RotoPol 11, Struers A/S, Rodovre, Denmark), except for the control group, which was ground finished with 800-grit silicone carbide abrasive.

The specimens were randomly assigned to one of the combinations of silicacoating (Sc), sandblasting (Sb), coupling agents, metal primers, adhesives, fibers (Fb), and opaquers (O) as shown in Table 2. The control (clinical) group contained natural central incisors that had cavities on their labial surfaces filled with amalgam. The preparations and undercuts for retention were made with a diamond bur (1014-KG Sorensen, Barueri, SP, Brazil) with an air turbine. Then the amalgam was condensed in the same way as in the other groups.

Amalgam Surface Modification

Five types of silane coupling agents, metal primers, and adhesives, namely, Visiobond (V), Porcelain Photobond (PP), Alloy Primer (AP), Unibond sealer (Us), and ESPE-Sil (ES), and four opaquers (Clearfil St Opaquer [CStO], Sinfony [S], Miris [M], and an Experimental Opaquer [EO]) were included in the study (Table 1). In one additional group, woven fiber was used to increase the interfacial strength for the repair joint.[13]

Bonding Procedures

The specimens were randomly assigned to various surface conditioning methods (Table 2). For surface roughening, silicacoating with 30 μm SiO_2 (CoJet, 3M ESPE, St Paul, MN, USA) or sandblasting with 50 μm Al_2O_3 was used. Then the silicacoated specimens were silanized (ESPE-Sil). After sandblasting, Alloy Primer was used, and opaquers and bonding agents were applied. In the fiber group, two pieces of preimpregnated woven glass-fiber sheets (Everstick Net, StickTech, Turku, Finland) were cut circular (diameter 3.6 mm) and were adhered with Visio-bond onto the conditioned amalgam.[13] The control group contained coronal parts of natural central incisors with an amalgam restoration in the middle portion of their labial surfaces. In this group, enamel was etched with 35% phosphoric acid, and then a bonding agent was applied.

Bonding procedures were carried out in accordance with the manufacturers' instructions. All materials were mixed and applied by one operator. Composite resin (Clearfil Photo Bond Posterior, Kuraray) was applied to the conditioned amalgam surfaces using polyethylene moulds (inner diameter, 3.6 mm; height, 4 mm). The composite resin was packed against the substrate incrementally with a composite-filling instrument (Ash 49, Dentsply, Weybridge, UK) and was subsequently photopolymerized in two layers measuring not more than 2 mm. Each layer was polymerized for 40 seconds from a distance of 2 mm. Light intensity was between 350 and 375 mW/cm^2 (SDS Kerr, Orange, CA, USA). After photopolymerization, the polyethylene moulds were gently removed from the test specimens. All specimens were stored in water for 5 weeks at 37°C prior to testing.

Table 1: *Brand, Manufacturer, Chemical Composition, and Batch Number of Materials Used in This Study*

Product Name	Company Name	Chemical Composition	Batch Number
Palapress Vario	Hereaus Kulzer, Hanau, Germany	Polymethylmethacrylate	F42028
CoJet-Sand (Sc)	3M ESPE AG, Seefeld, Germany	Aluminium trioxide particles coated with silica; particle size: 30 µm	165092
ESPE-Sil (ES)	3M ESPE AG, Seefeld, Germany	3-Methacryloxypropyltrimethoxysilane, ethanol	152745
Visio-Bond (V)	3M ESPE AG, Seefeld, Germany	Bisacrylate, aminodiol methacrylate, camphorquinone, benzyl dimethyl ketale, stabilizers	260950
Alloy Primer (AP)	Kuraray, Tokyo, Japan	Methacryloyloxydecyl dihydrogen phosphate (MDP) 6-(4-vinylbenzyl-n-propyl) amino-1,3,5- trizine-2,4-dithio (VBATDT)	00494
Unibond sealer (Us)	Cavex, Haarlem, The Netherlands	Maleic acid, methacrylate monomers	060043
Porcelain Bond Activator (PP)	Kuraray, Tokyo, Japan	Bisphenol, a polyethoxy demethacrylate, 3-methacryloyloxypropyl trimethoxy silane	001248
ClearfilSt Opaquer (CstO)	Kuraray, Tokyo, Japan	Bisphenol a diglycidylmethacrylate, triethyleneglycol dimethacrylate	413277
Sinfony (S)	3M ESPE AG, Seefeld, Germany	$>$90 wt% (octahydro-4,7-methano-1H-indeniyl)bis(methylene)diacrylate monomers	03344609
Miris (M)	Coltène, Whaledent, Cuyahoga Falls, OH, USA	Resin matrix: aromatic and aliphatic dimethacrylates, inorganic filler: Ba-Al-glass, pyrogenic, SiO_2, photoinitiator, camphorquinone/ synergist: NI	0189343
Experimental Opaquer (EO)	Cavex, Haarlem, The Netherlands	—	-
EverstickNet	Stick Tech Ltd, Turku, Finland	Silicium oxide, calcium oxide, aluminium oxide, natrium oxide, magnesium oxide, kalium oxide, barium oxide = E-glass (electric glass, silanated); bis-GMA, PMMA	CE0510
Clearfil Photo Bond Posterior	Kuraray, Tokyo, Japan	Bisphenol diglycidylmethacrylate, 10-methacryloyloxydecyl dihydrogen, 2-hydroxyethyl methacrylate	9594
Ultra-Etch	Ultradent Products Inc, South Jordan, UT, USA	35% phosphoric acid	L117

Abbreviations: bis-GMA, bisphenol A diglycidyl ether dimethacrylate; PMMA, autopolymerizing polymethylmethacrylate.

Testing Procedure

Specimens were mounted in the jig (Bencor Multi-T shear assembly, Danville Engineering Inc, San Ramon, CA, USA) of the Universal Testing Machine (Lloyd LRX, Lloyd Instruments Ltd, Fareham, UK), and force was applied to the substrate-adherent interface until fracture occurred. The specimens were loaded at a crosshead speed of 1 mm/min, and

Table 2: *Experimental Groups and Sequence of Applied Surface Conditioning Methods*

Step	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9
1	Sc	Sc	Sc	Sc	Sb	Sb	Sc	Sc	Etching enamel
2	ES	ES	ES	ES	AP	AP	ES	ES	Bonding agent on enamel
3	S	CstO	M	EO	S	PP	S	V	Sc the amalgam
4	V	V	V	V	Composite	CstO	Fiber	Composite	ES
5	Composite	Composite	Composite	Composite		Composite	V (no light curing)		S
6							Apply another fiber and light cure		Us
7							Composite		Composite

Abbreviations: AP, Alloy Primer; CstO, Clearfil St Opaquer; EO, Experimental opaque; ES, Espe-Sil-silane; M, Miris; PP, Porcelain Photobond; S, Sinfony; Sb, Sandblasting; Sc, Silica coating; V, Visio-Bond-bonding agent; Us, Unibond sealer.

the stress-strain curve was analyzed with Nexygen 2.0 software (Lloyd LRX, Lloyd Instruments Ltd).

Statistical Analysis

Statistical analysis was performed using Statistical Package for the Social Sciences (SPSS), version 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). One-way analysis of variance (ANOVA) was used to analyze the means of each group. Because statistically significant differences were noted between groups ($p<0.05$), multiple comparisons were made with the Tukey honestly significant differences (HSD) adjustment test to determine the effect of individual surface conditioning; p values less than 0.05 were considered statistically significant for all tests.

RESULTS

Shear bond strengths (SBSs) were significantly affected by the conditioning methods used ($p<0.05$). Mean bond strength values of the experimental groups are shown in Figure 1.

In groups 1, 2, 3, and 4, different opaquers were used, and group 9 was the control. In group 1, Sinfony Opaquer (S) exhibited the highest results (35.5 ± 4.1), and these results were significantly higher than those of groups 4 (19.4 ± 8.9), 6 (19.1 ± 7.8), and 8 (20.6 ± 4.1) ($p<0.05$). The difference between groups 2 (Clearfil St Opaquer-CstO) (27 ± 12.5) and 3 (Miris Opaquer-M) (24.4 ± 5.1) was not significant ($p>0.05$).

In group 5, Alloy Primer (AP), and in group 6, after AP, Porcelain Bond Activator (PP) were used. No significant differences were noted between groups 5 and 6 (22.6 ± 4.3 and 19.1 ± 7.8, respectively) ($p>0.05$). Group 9, containing enamel at the borders of the amalgam restoration, exhibited significantly lower SBSs (8.3 ± 3.4) than were seen in groups 1 to 3 (35.5 ± 4.1, 27 ± 12.5, 24.4 ± 5.1, respectively)

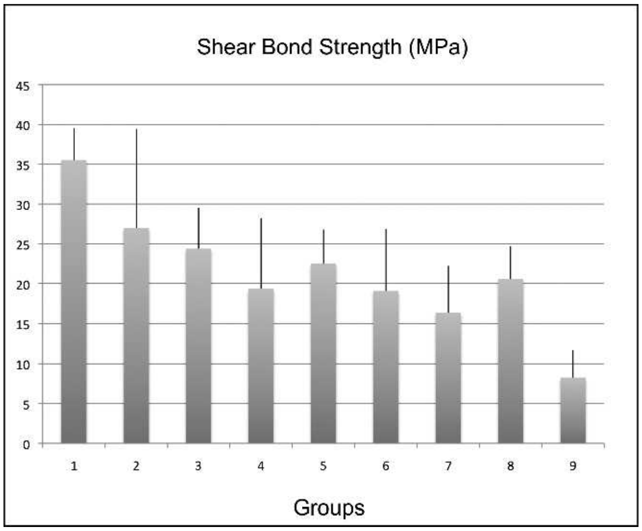

Figure 1. *Mean bond strength and standard deviations of experimental groups after various surface conditioning methods were used.*

($p<0.05$). In group 7 (16.4 ± 5.9), where fiber sheets were used, SBSs were significantly lower than in group 1 (35.5 ± 4.1) ($p<0.05$).

DISCUSSION

The repeatedly removed amalgam restoration is accompanied by associated loss of tooth tissue through progressive cavity enlargement and repeated insults to the pulp.[8] Because the addition of amalgam to amalgam restorations is not reliable,[15] experience indicates that whatever the circumstances of the amalgam restoration to be repaired, an adhesive approach should be considered. Moreover, for the repaired restoration to withstand functional loads, a strong and durable bond is desirable, to maintain the remaining restoration in function.[13]

In this study, different types of surface conditioning methods were used to adhere a composite resin to dental amalgam. Techniques used included surface roughening by silicacoating with 30 μm SiO_2 or sandblasting with 50 μm Al_2O_3. Then the silicacoated specimens were silanized, or Alloy Primer was applied to the sandblasted surfaces. Opaquers, bonding agent, and then composite were applied. One group was fiber-treated, and in another group, an extending border of enamel was present.

A significant difference was observed between the Sinfony opaquer applied group and the control group, and between the Sinfony group and the experimental opaquer. Opaquers have been advocated to mask the amalgam prior to composite bonding to simulate clinical situations wherein fracture of amalgam involved esthetics, such as in some visible areas of the mouth.[1,3] In this study, one powder-liquid opaquer (Sinfony) and three paste opaquers were used. It is likely that bond strengths produced by opaquers, besides being dependent on chemical composition, can depend on consistency and thus handling characteristics. When they were mixed in proportions specified by manufacturers, the consistency of the opaquers showed slight differences from one another; this may have contributed to differences in thickness between the resin and the conditioned amalgam, thereby affecting bond strength. High viscous paste opaquers possibly did not wet the surfaces evenly.[16] For this reason, a powder-liquid mixture with its low viscosity showed better results.

It might be difficult to get an equally thin layer in paste opaquers because the paste comes out of a tube, and air bubbles can be entrapped. The powder-liquid opaquer is easier to handle in terms of amount, consistency, and thickness.[1,3] Probably as a result of the thickness of the paste opaquer applied, the deepest part of this layer was not fully polymerized.[13,16-18] On the other hand, a powder-liquid opaquer can be easily controlled during application by using the tip of a probe. The experimental opaquer in the present study could not mask the metal surface; therefore it could not fulfill the requirement of masking amalgam.

The woven fiber–applied group showed lower bond strength than group 1, in which a bonding agent was also applied. However, in a previous study, higher bond strengths were attained when a preimpregnated glass fiber was used.[13] This might have resulted from use of another type of composite for repair. Although a small portion of fibers was extending at the borders, this did not increase bond strength.

In Group 9, simulating the possible clinical situation, the adhered area contained a small portion of enamel at the margins of the amalgam. Although it could be postulated that this would lead to an increase in bond strength due to enamel etching, group 9 showed significantly lower bond strengths when compared with groups 1, 2, and 3. This might be attributed to the small area of enamel left at the margins. Larger areas of enamel surrounding amalgam may enhance these results.

In groups 5 and 6, Alloy Primer was used, but no significant differences were noted between these groups and the other groups. In this study, higher SBS results (22 MPa) were attained than in a previous study (2.2 MPa)[13]; however, lower SBS results were attained from another study (27 MPa), wherein bond strengths of composite resins to cast titanium alloy with different surface conditioning methods were studied using this alloy primer.[17] In those studies, different types of amalgam or cast metal alloy substrates were used, and an opaquer was not included. Significant differences between groups 1 and 6 could be due in part to differences in surface roughening (silicacoating or sandblasting). The particle morphology of these two sand types is different. Particles used for sandblasting were ordinary alumina particles with 50 μm particle size, whereas silicacoating was achieved using alumina particles coated with silica, with particle size of 30 μm. The rough surfaces may not allow for good wettability of the silane or the primer in the pores.[16] One other reason could be greater hydrolytic degradation of methacryloyloxydecyl dihydrogen phosphate (MDP)-containing primers and adhesives versus methacrylate-based silanes and adhesives.[12,16,17] In fact, Alloy Primer is claimed to enhance the bond strength to both noble and base metal alloys because it contains both 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-trizine-2,4-dithio (VBATDT), the functional monomer for noble alloy, and MDP, the functional monomer for base metal alloy.[18] Long-term hydrolytic durability of such adhesive promoters needs further investigation.

A change in the surface roughness of high-copper amalgams occurs over time because of the formation of Cu_6Sn_5 crystals that provide micro-roughness essential for mechanical bonding to occur.[9] Future studies should investigate the effects of aging of amalgam and surface roughness on adhesion; this could be considered as the limitation of this study.

Shear bond testing is one convenient method of testing adhesive interfaces that is well established in biomaterials research.[19,20] However, the actual mode of disruption of the interface is virtually never a pure shear deformation. Nevertheless, the resultant data could be used for ranking materials and protocols within a single study.

CONCLUSIONS

From this study, the following could be concluded:

1. Bond strength of the resin composite to amalgam substrates varies in accordance with the surface conditioning methods used.
2. The Sinfony opaquer used to mask the amalgam showed significantly higher bond strength than the experimental opaquer.
3. Leaving a small border of enamel around the amalgam restoration led to significantly lower bond strengths.

Note

Part of this study was presented at the ConsEuro Meeting in Sevilla, Spain, which was held from March 12–14, 2009.

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