

# Shear Bond Strength of Aged Dental Amalgam Repaired with Composite

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**Resumo:** *Objetivos:* Avaliar a influência de três sistemas adesivos e de dois tratamentos de superfície sobre a resistência adesiva a forças de corte da interface de união compósito-amálgama. *Materiais e métodos:* Foram preparados 60 espécimes de amálgama (Tytin) e armazenados em água (37°C) durante 365 dias. Em 30 espécimes, a superfície do amálgama envelhecido foi sujeita à acção abrasiva de uma pedra verde. Nos restantes espécimes, o tratamento da superfície foi realizado com um jacto de óxido de alumínio. O compósito (Tetric) foi colado à superfície de amálgama utilizando três sistemas adesivos (Amalgambond, All-Bond2 e Scotchbond1) (n=10). Os espécimes foram armazenados em água (37°C) durante 7 dias e termociclados. Os ensaios mecânicos foram realizados com uma máquina de testes universal Instron. O tipo de falha de união foi avaliado com um estereomicroscópio. Os dados obtidos foram analisados com ANOVA e testes post-hoc segundo Student-Newman-Keuls ( $p<0.05$ ) *Resultados:* O tratamento de superfície apresentou uma influência estatisticamente significativa ( $p<0.001$ ) sobre os valores de resistência adesiva, tendo a abrasão com pedra verde produzido valores mais elevados. O Scotchbond1 produziu valores de resistência adesiva estatisticamente ( $p=0.047$ ) mais elevados que o All-Bond2, nos espécimes submetidos ao jacto de óxido de alumínio. A falha de união foi predominantemente do tipo adesivo. *Conclusões:* O tratamento da superfície do amálgama envelhecido com jacto de óxido de alumínio permitiu duplicar a resistência adesiva relativamente à abrasão com pedra verde. Apenas foram encontradas diferenças estatisticamente significativas entre os adesivos, nos espécimes condicionados com jacto de óxido de alumínio.

**Palavras-Chave:** Amálgama; Compósito; Materiais dentários; Reparação; Adesão; Tratamento de superfície

**Abstract:** *Objectives:* The objective of this study was to evaluate the effect of surface treatment and adhesive agent on the shear bond strength between a resin composite and an aged dental amalgam (356-days). *Materials and Methods:* Sixty amalgam (Tytin) disks were stored in water at 37°C for 365 days. Half of the specimens were airborne particle abraded and the remaining half was roughened with a greenstone. Resin composite cylinders (Tetric) were bonded onto the amalgam surfaces using Amalgambond, All-Bond 2 or Scotchbond 1 (n=10). Specimens were stored in water at 37°C for 7 days and thermocycled. Shear bond strength testing was carried on an Instron Universal Testing Machine. Stereomicroscope examination was carried out to determine the bond failure sites. Results were analyzed by ANOVA followed by Student-Newman-Keuls' post-hoc tests and the level of statistical significance was set at 5%. *Results:* Surface treatment significantly affected the shear bond strengths ( $p<0.001$ ) with airborne particle abrasion producing the highest bond strengths. Significantly higher shear bond strengths ( $p=0.047$ ) were found with Scotchbond 1 in comparison with All-Bond 2, in the airborne particle abraded specimens. Bond failures were predominantly of the adhesive type. *Conclusions:* Airborne particle abrasion resulted in a twofold increase in shear bond strength compared with roughening with a greenstone. Significant differences were found between the adhesives, in the airborne particle abraded specimens.

**Key-words:** Amalgam; Composite resin; Dental materials; Repair; Bonding; Surface treatment

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## INTRODUCTION

Amalgam-composite interfaces are created by several dental procedures<sup>(1,5)</sup>. Cementing fixed prostheses and orthodontic brackets to dental surfaces restored with amalgam has become common, with the modern resin composite cements and bonding techniques<sup>(1,6,7)</sup>. Repairing amalgam restorations, by adding fresh amalgam or resin composite, can provide a less invasive procedure than its complete removal and replacement, whenever it is desirable to reduce further trauma to the tooth or to avoid additional stress to the patient<sup>(4,8)</sup>. Finally, since esthetics has become a strong concern to both clinicians and patients, the application of resin composite veneers over existing amalgam restorations has been mentioned by several authors<sup>(3,9-14)</sup>.

Several systems have been described to provide bonding of resin composites to amalgam. Macromechanical retention techniques have been initially suggested such as undercuts, grooves and pins<sup>(9,11-13,15)</sup>. More recently, the use of microretention and bonding agents has been proposed<sup>(2,3)</sup>. Several authors have studied the influence of different adhesive systems and amalgam surface treatments, such as roughening and airborne particle abrading, on the adhesive strength between resin composite and amalgam, with varying results<sup>(4,8,16-19)</sup>. Most studies suggest that airborne particle abrading the amalgam surface results in higher shear bond strengths than grinding with conventional abrasive instruments<sup>(5,7,19-20)</sup>. However, another study did not find any bond strength improvement by abrading the amalgam surface<sup>(8)</sup>.

The influence of the aging period of the existing amalgam on the bond strengths has been previously investigated<sup>(8)</sup>. However, most previous studies used short-term aging periods, usually ranging between freshly condensed unset amalgam and 21 days<sup>(1,6,8,16-19)</sup>. Since amalgam alloys are subjected to continuous changes as a result of mechanical forces, corrosion and slow solid-state phase changes<sup>(21)</sup>, older amalgams may behave in a different manner. The adhesive strength between resin composite and amalgam with a longer aging period has not been previously investigated.

The objectives of this in-vitro study were<sup>(1)</sup> to compare two different surface treatment techniques and<sup>(2)</sup> to evaluate the effect of three different adhesive agents on the shear bond strength between a resin composite and a dental amalgam submitted to a long term aging period (365 days).

## MATERIALS AND METHODS

In this study, the shear bond strength between old amal-

gam and composite was investigated. Dentists commonly use a variety of methods to improve the retention between two different materials. Accordingly, in order to produce micro-retentions, old amalgam was airborne particle abraded or roughened with a greenstone<sup>(19)</sup>. To further promote the adhesion, three adhesive systems were used before the application of the resin composite on the treated amalgam surface.

A total of 60 flat cylindrical amalgam specimens were prepared with a spherical amalgam alloy (Tytin, batch n° 51066, Sybron/Kerr, Romulus, MI, USA). The amalgam was triturated according to the manufacturer's recommendations regarding time and speed and then manually condensed into cylindrical retentive cavities, with a diameter of 7.5 mm and 4 mm deep, made in standardized polymethylmethacrylate cylindrical blocs (13 mm in diameter and 12 mm in height). The cavities were overfilled and the amalgam excess was immediately removed with a cutting instrument. After a 24 hour initial setting period, the amalgam surfaces were flattened with 220, 320, 500 and 1000 grit silicon carbide grinding paper (Struers, DK-2610 Rodovre, Copenhagen, Denmark). The final polishing step was made with a green rubber point (ref 4572, Dedeco International Inc, Long Eddy, NY, USA). All specimens were then stored in distilled water at 37° C for 365 days, with the water being changed every month<sup>(22)</sup>.

Prior to resin composite bonding, half of the specimens were airborne particle abraded with 50  $\mu$ m aluminum oxide using a Microetcher Erc (Danville Engineering, San Ramon, CA, USA) and the other half was roughened with a greenstone (Shofu Inc., Kyoto 605, Japan) using a handpiece (Faro F632, Faro USA, Burlingame, CA, USA) at 30.000 rotation per minute. The load on the bur was standardized by the operator performing the procedure with very light hand pressure to achieve a visually roughened surface<sup>(23)</sup>. All specimens were etched with 35% phosphoric acid (Scotchbond Etchant, batch n° 4AT, 3M Dental Products, St Paul, MN, USA) in order to remove the abrasive particles from the amalgam surface.

The specimens were divided in 6 groups of 10 specimens each, according to the possible combinations between the two surface treatments and the three bonding agents. The sample size (n=10) was determined using an estimated significance level of 0.05 and a power of 0.8. The adhesive systems used were Amalgambond, All-Bond 2, and Scotchbond 1 (Table 1). All adhesives were used according to the manufacturer's instructions. The resin composite (Tetric, batch n° 805372, Vivadent Ets, FL-9494, Schaan, Liechtenstein) was polymerized for 40 seconds with a 3M Curing Light 3000XL unit (3M Dental Products, St Paul, MN, USA), using a gelatin capsule with an internal diameter of 4.9 mm (Torpac Inc., Fairfield, NJ, USA) as matrix.

Material	Component	Batch n°	Manufacturer
Amalgambond Plus	Dentin Activator	60402	Parkell Products Inc, Farmingdale, NY, USA
	Base	60601	
	Adhesive Agent	62009	
	Universal Catalyst	605022	
	HPA	60405	
All-Bond 2	Primer A	29177	Bisco Inc, Itasca, IL, USA
	Primer B	29187	
	Dentin / Enamel Bonding Resin	39047	
	Prebond	39037	
Scotchbond 1	Adhesive	3BB	3M Dental Products, St Paul, MN, USA

Table 1 - Adhesive systems used for bonding the composite resin to the aged amalgam

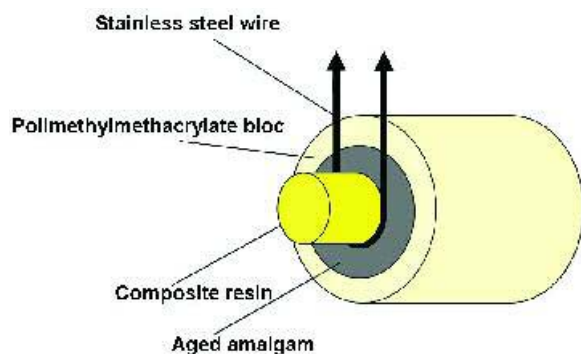


Figure 1 - Shear bond strength testing design

The specimens were stored in water at 37°C for 7 days, and thermocycled for 500 cycles between 5°C and 55°C with a 20-second dwell time. Shear bond strength testing was carried on an Instron Universal Testing Machine, model 4502 (Instron Ltd, Bucks, HP12 3SY, UK), at a crosshead speed of 2 mm/min (Figure 1). The specimens were positioned in the base of the machine with the amalgam surface parallel to the direction of the force produced by the Instron. The force was applied at the amalgam-composite interface using a 20 cm loop of stainless steel wire ( $\phi$  0.8 mm), suspended from the movable crosshead. The force required to split the specimens was recorded in Newton and later converted to stress (MPa).

The fracture surfaces were examined using a Nikon SMZ-2 stereomicroscope (Nikon Europe BV, P.O.B. 7609, Netherlands), under x20 magnification, and classified as:<sup>(1)</sup> adhesive failure -

failure at the amalgam-adhesive interface,<sup>(2)</sup> cohesive failure of the resin composite, and<sup>(3)</sup> adhesive-cohesive failure - failure at the amalgam-adhesive interface with partial cohesive failure of the resin composite.

After abrading the old amalgam surface and prior to bonding, representative specimens of both amalgam surface treatments were prepared, observed and photographed using a scanning electron microscope (Hitachi S-450, Hitachi Ltd., Tokyo, Japan).

The differences in mean shear bond strength among the different groups were determined initially using a two-way analysis of variance (ANOVA). Given cross-product interactions, two one-way ANOVA for each surface treatment were conducted, along with Student-Newman-Keuls' post-hoc tests to detect which means differed. The normality of the data distribution and the equality of variance were evaluated using Kolmogorov-Smirnov and Bartlett tests, respectively. Surface treatment and adhesive system were used as the independent variables and the level of statistical significance was set at 5%.

## RESULTS

Mean shear bond strengths ranged from 12.3 MPa to 16.8 MPa, for the airborne particle abraded specimens, and from 6.9 MPa to 8.2 MPa, when roughening with a greenstone (Figure 2).

Two-way ANOVA showed that the surface preparation technique significantly affected the shear bond strengths ( $p < 0.001$ ). The effect of the bonding agent was not significant ( $p = 0.168$ ).

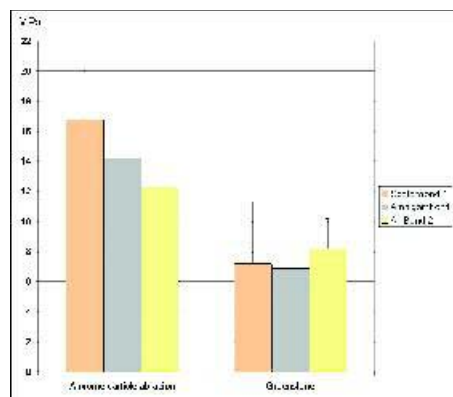


Figure 2 - Mean shear bond strengths and standard deviations of composite resin bonded to aged amalgam, using three adhesive systems and two surface treatments (MPa). (Means connected by horizontal bars were not significant different).

A statistically significant interaction ( $p=0.025$ ) was also found between these two factors, indicating that the effect of the adhesive system differed by method of surface treatment. Accordingly, the data for each surface treatment were analyzed in two separate one-way ANOVAs followed by Student-Newman-Keuls' post-hoc tests. These analyses showed statistically significant differences ( $p=0.047$ ) between Scotchbond 1 and All-Bond 2 in the airborne particle abraded specimens, but no significant differences ( $p=0.371$ ) between the specimens roughened with a greenstone.

Bond failures (Table 2) occurred predominantly at the amalgam-adhesive interface. A small percentage (8%) of adhesive-cohesive failures was also found, in the airborne particle abraded groups. Cohesive failures, either at the resin composite or the aged amalgam, were not found.

Scanning electron microscope evaluation showed that aluminum oxide airborne particle abrasion created a rougher (significantly more retentive) surface than abrading with a greenstone which creates mainly grooves and ridges on the amalgam surface (Figures 3 and 4).



Figure 3 - SEM micrograph of the amalgam surface roughened with a greenstone



Figure 4 - SEM micrograph of the amalgam surface after aluminum oxide airborne

## DISCUSSION

In the present in-vitro study, using an aging period of 365 days, the mean shear bond strengths ranged between 12.3 and 16.8 MPa when the specimens were submitted to airborne parti-

Experimental sequence	Bond Failure Site		
	Adhesive	Adhesive-cohesive	Cohesive
Airborne particle abrasion, Amalgambond	8	2	0
Airborne particle abrasion, All-Bond 2	8	2	0
Airborne particle abrasion, Scotchbond 1	9	1	0
Greenstone, Amalgambond	10	0	0
Greenstone, All-Bond 2	10	0	0
Greenstone, Scotchbond 1	10	0	0
<b>Total</b>	<b>55</b>	<b>5</b>	<b>0</b>

Table 2 - Distribution of bond failure sites of the 60 composite resin cylinders bonded to aged amalgam using three adhesive systems and two surface treatments.



cle abrasion, and between 6.9 and 8.2 MPa for the specimens roughened with a greenstone. These values are close to the bond strength to etched enamel which range from 15 to 25 MPa, depending on the resin and the testing method used<sup>(24)</sup>.

Under the conditions of the present investigation, surface treatment was the most influent factor on the bond strength between resin composite and an aged dental amalgam. When the two types of surface treatment were compared, the mean values obtained with airborne particle abrasion showed an 89% increase. These results corroborate the findings of previous studies<sup>(5,7,19-20)</sup>, showing that airborne particle abrading the amalgam surface using a 50  $\mu$ m aluminum oxide powder can dramatically increase the shear bond strength of several adhesive systems to old amalgam.

The increased surface area and an improved mechanical interlocking of the adhesive system have been suggested as the main factors leading to this bond strength increase<sup>(19)</sup>. A previous study showed that airborne particle abrading with aluminum oxide produces an increase of 30% to 90% of the surface area of nickel-chromium and palladium-silver alloys, increasing simultaneously the surface wettability by reducing the contact angle between several adhesive systems and these alloys<sup>(25)</sup>.

The present data suggest that amalgam surface characteristics have more influence in the bond strength between amalgam and composite than the adhesive system used. In this study,

the general effect of the adhesive system on the shear bond strengths was a minor one. However, a significant difference was found between Scotchbond 1 and All-Bond 2, in the airborne particle abraded specimens. These results are in agreement with previous publications<sup>(5,16,18)</sup>.

The stereomicroscope observation of the fractured specimens showed adhesive failures in most specimens. A combination of adhesive-cohesive fractures was found in 16% of the airborne particle abraded specimens, suggesting that the adhesive strengths approached the cohesive resistance of the resin composite itself.

## CONCLUSIONS

It was concluded that under the conditions of the present study, airborne particle abrading the amalgam surface significantly increases the shear bond strength, when compared with roughening with a greenstone. The adhesive system used did not significantly influence the results to the same degree, with the exception of the difference between Scotchbond 1 and All-Bond 2, in the airborne particle abraded specimens.

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