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EARLY SHEAR BOND STRENGTH OF EXPERIMENTAL AMALGAM-BONDING COMBINATIONS WITH AND WITHOUT THERMOCYCLING

Nadim Baba* | Fred Berry | Monica Lehnhoff*****

Abstract

The purpose of this study was to evaluate the shear bond strength (SBS) of amalgam to dentin using four different bonding systems. Eighty extracted human third molars of approximately the same size were selected. Teeth were sectioned parallel to the occlusal plane to expose mid-coronal dentin, and then one-half of each tooth was embedded in acrylic resin. Four bonding systems were used following manufacturers' recommendations. Enamel and dentin surfaces on teeth being restored with amalgam bonding were etched, rinsed, single-coated with primer/adhesive, restored with Valiant PhD-XT using condensation to intermingle amalgam with setting adhesive, light cured (30 sec) and conditioned (distilled water, 37°C, 24h). Half of all specimens were thermocycled for 24 hours (5°C/55°C, 1 min dwell times, 500 cycles). All specimens were macro shear bond strength tested (loading rate of 1mm/min, 25°C, knife edge texted). Results (MPa = failure load/ bond area) for groups (n=10) were statistically analyzed (2-way ANOVA, Tukey-Kramer post hoc tests, $p \leq 0.05$). Shear bond strength of thermocycled groups was the greatest ($p<0.05$). Results for groups 1-4 for storage only (6.7±1.6, 6.5±1.0, 3.6±0.9, 6.4±2.5) versus thermocycled (13.3±3.0, 15.1±4.9, 15.4±4.7, 18.2±5.8) showed essentially no bonding system effect and no interaction of bonding system with thermocycling. After thermocycling, the adhesive mode of failure was most common.

Keywords: amalgam bonding - shear strength - adhesives.

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FORCES DE CISAILLEMENT D'UNE COMBINAISON EXPÉRIMENTALE D'ADHÉSIFS D'AMALGAMES AVEC ET SANS THERMOCYCLAGE

Résumé

Le but de cette étude était d'évaluer la résistance au cisaillement de l'amalgame collé à la dentine en utilisant quatre systèmes de collage différents. Quatre-vingt dents de sagesse humaines extraites, d'environ la même taille, ont été sélectionnées. Les dents ont été sectionnées parallèlement au plan occlusal à mi- hauteur coronaire pour exposer la dentine, puis la moitié de chaque dent a été incluse dans de la résine acrylique. Quatre systèmes de collage ont été utilisés selon les recommandations des fabricants. Les surfaces de l'émail et de la dentine des dents en cours de restauration avec un amalgame collé ont été mordancées, rincées, recouvertes d'une couche de primer/adhésif, restaurées avec du Valiant PhD-XT avec condensation pour entremêler l'amalgame à l'adhésif en cours de prise, photopolymérisées (30 sec) et conditionnées (l'eau distillée, 37°C, 24h). La moitié de tous les échantillons ont été thermocyclés pendant 24 heures (5°C/ 55°C, le temps de séjour: 1 minute, 500 cycles). La résistance au cisaillement a été testée pour tous les échantillons (taux de chargement=1mm/min, 25 °C). Les résultats (MPa=charge de rupture/zone de collage) pour les groupes (n=10) ont été statistiquement analysés (2-way ANOVA et Tukey-Kramer post hoc, $p \leq 0{,}05$). La résistance au cisaillement des groupes thermocyclés était plus élevée ($p < 0{,}05$). Les résultats pour les groupes 1-4 pour le stockage seulement (6,7 ± 1,6, 6,5 ± 1,0, 3,6 ± 0,9, 6,4 ± 2,5) par rapport aux groupes thermocyclés (13,3 ± 3,0, 15,1 ± 4,9, 15,4 ± 4,7, 18,2 ± 5,8) n'a montré pratiquement aucun effet du type de collage ainsi qu'aucune interaction du type de collage avec le thermocyclage. Le mode d'échec le plus commun était l'échec adhésif après thermocyclage.

Mots clés : amalgame - collage - résistance au cisaillement - adhésifs.

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Introduction

Amalgam restorations have been used for more than a century and have proven to be a valuable restorative material. They are relatively inexpensive, versatile and can provide long-lasting results when appropriately placed [1, 2]. Although the quality of the amalgam alloy available has improved, marginal sealing remains a challenge for the clinician [3, 4]. Factors such as thermal conductivity [5], thermal expansion coefficient and lack of adhesion facilitate initial microleakage soon after placement of amalgam restorations [6]. Sealing improves with aging due to the corrosion process resulting in releasing oxides that will be deposited into the amalgam-tooth interface [7, 8]. When cavities are more extensive, dentists rely on several retentive features to provide additional retention for the amalgam (pins, boxes, grooves, etc.) [9,10]. To minimize tooth reduction and yet provide additional retention to compromised tooth structure, bonding amalgam using various adhesive agents has been proposed to improve the bond strength of amalgam to tooth structure [11,12]. The mechanism of amalgam bonding to tooth structure was first explained in 1983 by Ziardiackas and Stoner [13]. They described the formation of a micro-mechanical bond between the amalgam and the adhesive material before polymerization and showed that the bond of the adhesive material to dentin and enamel occurs through dentin-calcium ion linkage. In an *in vitro* study, Setcos et al. [14] demonstrated that bonding amalgams provides retention that is equivalent or better than the use of mechanical undercuts. Several studies [14-16] have described the potential advantages of amalgam-bonded restorations: decreased microleakage, decreased post-operative sensitivity, increased bond strength and enhanced fracture resistance of restored teeth. Several types of bonding agents using different curing modes (light, chemical and dual-curing) have been proposed [15, 17, 18].

It was hypothesized that thermocycling would have no statistically significant effect on the bond strength of bonded amalgam to dentin. The objective of this *in vitro* study was to evaluate the shear bond strength of amalgam bonded to dentin using four different bonding systems.

Materials and methods

Eighty extracted human third molars of approximately the same size were collected. Teeth were numbered and randomly divided into 2 groups: 1) 24-hour shear bond strength without thermocycling (WTC) and 2) with thermocycling (TC).

The teeth were sectioned parallel to the occlusal plane to expose mid-coronal dentin using a low speed diamond saw (South Bay Technology Inc., Model 650, Temple City, CA, USA) with water coolant. The sectioned teeth were mounted inside auto-polymerizing cylindrical shaped acrylic resin blocks (1x1 inches) (Neocryl Splint, Bosworth, Skokie, IL, USA) with the exposed dentin surface placed parallel to the surface of the block. The dentin surface was finished using no.600 silicone-carbide abrasive paper under running water to provide a uniformly textured surface and ensure that no acrylic resin was present on the bonding surface (Fig.1). Four bonding systems were used in this study (Table 1): 1) All-Bond 2® Primer A&B with Panavia 21, 2) All-Bond 2® Primer A&B with Pre-Bond & D/E resin, 3) Optibond® All-in-one with NX3 DC, and 4) Adper™ Scotchbond™ Multi-Purpose Plus adhesives.

Each group was divided randomly into 4 subgroups. Mixing some of the materials was performed due to the availability of a resin cement (Panavia) and the desire to evaluate its bond to dentin utilizing a dentin bonding agent.

All teeth were etched with 37% phosphoric acid gel for 15 seconds (Scotchbond™ etchant, 3M ESPE, St Paul, MN, USA) and then rinsed using a combination of distilled water and filtered air for 10 seconds. Excess moisture was removed from the prepared surfaces using a cotton pellet while ensuring that the etched dentin remained moist. For each group, a primer/adhesive solution was applied to the surface in thin coat(s) according to established protocols (Table 2). All teeth were then placed in an Ultradent bonding jig (Ultradent Products Inc., South Jordan, UT, USA) and a size no.1 hard copper band (Henry Schein, Melville, NY, USA) was used to allow firm amalgam condensation (Valiant PhD-XT, Ivoclar Vivadent Inc., Amherst, NY, USA) onto the resin-treated surface (dentin only). The amalgam was allowed to set undisturbed for 30 minutes.

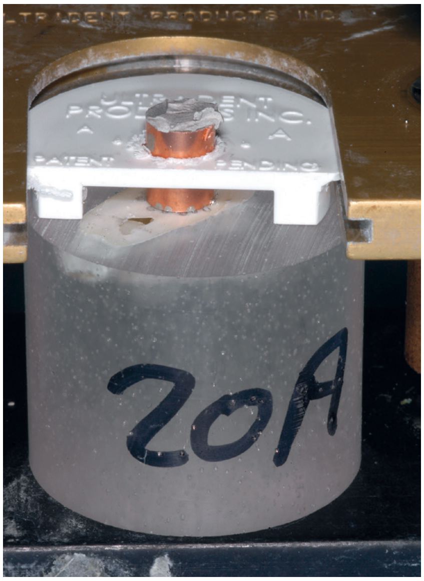

Fig. 1: Bonding jig holding the copper band for amalgam condensation.

The specimens of the WTC group were then immersed in distilled water at 37°C for 24 hours after which the shear bond strength was tested (25°C, dry specimen) (VWR, model 1520, Batavia, IL, USA).

The specimens of the TC group were placed in a thermocycling apparatus for 24 hours (Thermo NESLAB, Portsmouth, NH, USA) and cycled in water between 5°C and 55°C with a dwell time of 1 minute at each temperature for 500 cycles.

Trade Name	Material	Manufacturer	Lot number
Valiant PhD-XT	Amalgam	Ivoclar Vivadent Inc., Amherst, NY, USA	DO302X1
Optibond® All-in-One	Dental adhesive	Kerr, Orange, CA, USA	2763994
All Bond 2®	Dental adhesive	Bisco, Schaumburg, IL, USA	0700003799 0700004831
Adper™ Scotchbond™	Dental adhesive	3M ESPE, St. Paul, MN, USA	7RA/7BA/7KY/7BK
Nexus 3 DC	Resin cement	Kerr, Orange, CA, USA	2857172
Panavia 21	Resin cement	Kuraray Medical, Okayama, Japan	1 1/130
Pre-Bond resin	Bonding agent	Bisco, Schaumburg, IL, USA	800002438
D/E resin	Bonding agent	Bisco, Schaumburg, IL, USA	800002438

Table 1: Materials selected for the study.

Material used	Mixing technique
Optibond® All-in-one with Nexus 3 DC	Etch, apply 1st coat of adhesive, scrub 20 seconds, apply 2nd coat, scrub, air dry, mix and apply a thin coat of the NX3 DC cement, condense amalgam.
All-Bond 2® Panavia 21	Etch, mix one drop A & B primers and apply 5-6 coats, lightly dry, mix and apply thin coat of Panavia 21, condense amalgam.
Adper™ Scotchbond™ Multipurpose Plus	Etch, mix one drop of activator and primer, apply 15 seconds, dry 5 seconds, mix one drop of adhesive and catalyst, apply thin coat to all surfaces, condense amalgam .
All-Bond 2® Prebond resin D/E resin	Etch, mix one drop A & B primers and apply 5-6 coats, mix one drop of Prebond resin and one drop of D/E resin, apply thin coat, condense amalgam.

Table 2: Mixing of materials used in the study.

Two trained and calibrated operators performed all restorative procedures.

A screw-driven universal testing machine (ReNew Model 1125, MTS Systems Corporation, Eden Prairie, MN, USA) was used to evaluate the shear bond strengths at a constant loading rate of 1.0mm/minute. The value recorded was the maximal load at failure in MPa for each specimen (Fig. 2).

Results for the shear bond strength values were tabulated and an initial comparison of the groups was made using 2-way analysis of variance (ANOVA). Significance was set at an p-value of 0.05. A Tukey-Kramer multiple comparison test was then performed at a significance level p=0.05. Debonded surfaces were viewed under a microscope (Model DX; Global, St Louis, Mo) under ×30 magnification to determine modes of failure.

Failures were classified as adhesive, cohesive or mixed. Adhesive failures were defined as having 75% of the debonded amalgam surface area free of resin bonding agent. Cohesive failures were defined as having 75% of the amalgam surface area covered with resin. Mixed failures were defined as the amalgam surface exhibiting less than 75% adhesive or cohesive failure.

Results

The means and standard deviations of SBS of bonded amalgam to dentin are listed in table 3. Two-way ANOVA and the Tukey-Kramer test revealed that there were significant differences in the SBS of amalgam to dentin due to thermocycling ($p<0.05$), whereas the interaction between the bonding systems and the test condition was not statistically significant ($p>0.05$). Before thermocycling, the mean bond strengths varied from the highest value of 6.7 MPa for All-Bond® Pre-bond to the lowest value of 3.6 MPa for All-Bond® Panavia 21. The mean SBS in all groups increased after 500 thermocycles. The increase in bond strength calculated by difference between thermocycle 0 and thermocycle 500 values was the least for group 1 (198%) and the greatest for group 3 (428%).

Fracture sites were evaluated under a dissecting microscope for each of the adhesive systems within the study groups. Failure modes of all specimens are presented in table 3. In the 24-hour storage protocol with no thermocycling, groups 1 and 3 had seven specimens with adhesive failures whereas three underwent mixed failure. In group 2, half of the specimens underwent adhesive failure and the other half underwent mixed failure. In group 4, two specimens underwent adhesive failure; three specimens underwent cohesive failure, whereas five specimens underwent mixed failure. Among the thermocycled groups, adhesive failure occurred in all the groups 1 and 2 specimens. In group 3, two speci-

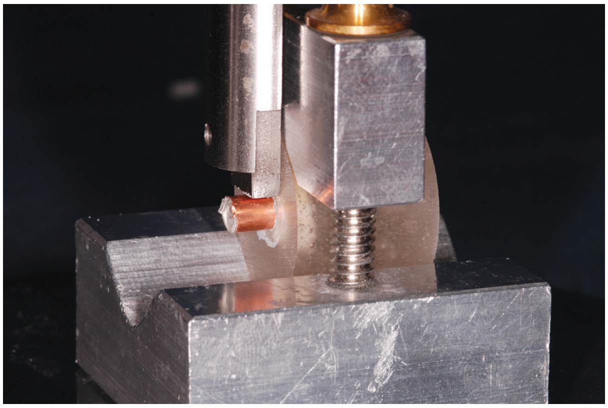

Fig. 2: Device for the shear bond strength test.

Shear bond strength (MPa) Mean ± SD			**Failure Mode**	
Group	No thermocycling	Thermocycled (500 cycles)	No thermocycling	Thermocycled (500 cycles)
1- All-Bond® 2 Prebond resin D/E resin	6.7 ± 1.6	13.3 ± 3.0	7/0/3	10/0/0
2- Adper™ Scotchbond™ Multipurpose Plus	6.5 ± 1.0	15.1 ± 4.9	5/0/5	10/0/0
3- All-Bond 2® Panavia 21	3.6 ± 09	15.4 ± 4.7	7/0/3	2/3/5
4- Optibond® All-in-one Nexus 3 DC	6.4 ± 2.5	18.2 ± 5.8	2/3/5	7/0/4

n = 10
All specimens were stored 24 hours prior to testing
SD = standard deviation
A/C/M (adhesive failure/cohesive failure/Mixed failure)

Table 3: Early shear bond strength and failure mode.

mens underwent an adhesive failure; three specimens underwent a cohesive failure, whereas five specimens underwent mixed failure. In group 4, seven specimens underwent adhesive failure whereas three underwent mixed failure.

Discussion

The results of the present study showed that thermocycling did increase the bond strength of bonded amalgam to dentin.

SBS of amalgam to dentin without thermocycling ranged between 3.6 and 6.7 MPa. These results are consistent with those of McComb et al. [19], Bagley et al. [20] and Barkmeier et al. [21] who concluded that the strength of amalgam bonded to tooth is in the range of 3 to 10 MPa.

After thermocycling, the SBS of amalgam bonded to dentin ranged between 13.3 and 18.2 MPa. These results are coherent with those of Varga et al. [22] who stated that the bond strength of amalgam to dentin increased after thermocycling. However, McComb et al. [19] reported that there was a reduction in the SBS after thermocycling. Additional previous studies have shown no effect of thermocycling on bonded amalgam restorations [23, 24].

The current study indicates that thermocycling significantly improved the bond strength between amalgam and dentin. This result could be due to the fact that thermocycling enhances the maturation of the bond between the amalgam and the resin. Unpolymerized resin is incorporated into the condensed amalgam and thermocycling can mature the interlocking bond between the resin and the amalgam [25]. These results show that bonding of amalgam to dentin is possible and can enhance the longevity of extensive amalgam restorations even without the addition of retentive features.

Adhesive failure occured after thermocycling in all specimens of the groups 1 and 2 and in 70% of the specimens of group 4. However, mixed failure mode was more common in group 3 after thermocycling. The types of bond failure obviously support the results of the SBS test. These results are consistent with those of Van Meerbeek et al. [26] and Eakle et al. [27].

Resin did not remain totally confined to the bonded area and, during amalgam condensation, it became part of the set copper band cylinders. This additional surface attachment might have increased the SBS results obtained after thermocycling.

Results may vary in *in vivo* situations where bonded amalgams are subjected to intraoral forces and thermocycling. Other factors such as the age of dentin and the confinement of the amalgam within a cavity would also likely influence the SBS of bonded amalgam.

The results of this study have produced further inconsistency between the published studies, highlighting the need for additional studies that compare methodologies and materials to determine the impact of thermocycling on the bonding of amalgams to dentin.

Conclusion

Based on the conditions and limitations of this *in vitro* study, the following conclusions may be drawn:

1-There was a significant increase in the SBS of amalgam bonded to dentin due to thermocycling ($p<0.05$).

2- The results were not statistically significant for the four bonding types as well as for the interactions between bonding systems under these test conditions ($p>0.05$).

3-After thermocycling, the adhesive fracture was the most common mode among three of the tested groups.

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