Original Article

Effects of Silica Coating and Silane Surface Conditioning on the Bond Strength of Metal and Ceramic Brackets to Enamel

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ABSTRACT

Objective: To evaluate the effect of tribochemical silica coating and silane surface conditioning on the bond strength of metal and ceramic brackets bonded to enamel surfaces with light-cured composite resin.

Materials and Methods: Twenty metal and 20 ceramic brackets were divided into four groups (n = 10 for each group). The specimens were randomly assigned to one of the following treatment conditions of the metal and ceramic brackets' surface: (1) tribochemical silica coating combined with silane and (2) no treatment. Brackets were bonded to the enamel surface on the labial and lingual sides of human maxillary premolars (20 total) with a light-polymerized resin composite. All specimens were stored in water for 1 week at 37°C and then thermocycled (5000 cycles, 5°C to 55°C, 30 seconds). The shear bond strength values were measured on a universal testing machine. Student's *t*-test was used to compare the data ($\alpha = 0.05$). The types of failures were observed using a stereomicroscope.

Results: Metal and ceramic brackets treated with silica coating with silanization had significantly greater bond strength values (metal brackets: 14.2 ± 1.7 MPa, $P < .01$; ceramic brackets: 25.9 ± 4.4 MPa, $P < .0001$) than the control groups (metal brackets: 11.9 ± 1.3 MPa; ceramic brackets: 15.6 ± 4.2 MPa). Treated specimens of metal and ceramic exhibited cohesive failures in resin and adhesive failures at the enamel-adhesive interface, whereas control specimens showed mixed types of failures.

Conclusions: Silica coating with aluminum trioxide particles coated with silica followed by silanization gave higher bond strengths in both metal and ceramic brackets than in the control group.

KEY WORDS: Silica coating; Metal and ceramic brackets; Bond strength

INTRODUCTION

The development of adhesives and adhesive techniques has been greatly influenced by the research work directed toward improving adhesive properties of materials used in conservative dentistry.[1,2] Because bonding in orthodontics is semipermanent in nature, bond strength should not only be high enough to resist debonding but also low enough so that damage to the existing tooth or restoration would not occur during debonding.[3]

A number of techniques used on bracket surfaces have been reported to increase bond strength between brackets and enamel.[3–8] Micromechanical bonding systems, such as sandblasting with aluminum oxide particles that create a very fine roughness, increase the surface area, thereby enhancing mechanical and chemical bonding.[9] However, the use of only sandblasting might be insufficient to obtain reliable bond strength, especially after thermal cycles.[10,11] Advances in silane coupling agents contribute to high bond strength by promoting a chemical bond between resin composite, ceramic (silica based) and metal (base metal).[12,13] Many studies have shown that appli-

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cation of a silane agent to base metal and silica-based ceramic surfaces after sandblasting with aluminum oxide particles produces higher bond strengths.[3,13,14]

Another recommended technique for surface roughening to obtain a reliable bond is an intraoral sandblaster.[15,16] In addition, the air abrasion technique based on tribochemical silica coating provides ultrafine mechanical retention and also is used with a silane coupling agent. Moreover the surface, when treated with silica-coating system, is not only "abraded," but will become embedded with a silica coating that is derived from the silica-coated alumina trioxide particles that have been used. Metal or ceramic surfaces are abraded with 30-μm grain size aluminum oxide (Al_2O_3) chemically modified with silica, called CoJet-Sand (3M ESPE AG, Seefeld, Germany), in an intraoral sandblaster.

Silica coatings are used in many dental practices.[17–19] Some studies have also shown that silica coating increased the bond strength of aluminum oxide ceramics to resin composite.[13,19] However, a review of the literature indicated that this system has not been investigated for orthodontic use on metal and ceramic brackets.

This study evaluated the effect of tribochemical silica coating and silane surface conditioning on the bond strength of metal and ceramic brackets bonded to enamel surfaces with light-cured composite resin.

MATERIALS AND METHODS

Twenty human maxillary premolars free of caries and restorations were extracted for orthodontic reasons and used in this study. The teeth were stored in 0.1% thymol solutions at room temperature immediately after extraction and used within 4 weeks.[20,21] After they were cleaned of residual organic material, the labial and lingual surfaces were treated with flour of pumice and a rubber cup, rinsed with tap water, and dried with an air-syringe.

Metal ($n = 20$) (Generus-Roth, stainless steel, GAC International, Bohemia, NY) and ceramic ($n = 20$) (Allure, 99.9% pure alumina, GAC International) brackets designed for maxillary premolars were used consecutively for testing a surface conditioning method and control.

The specimens were randomly assigned to one of the following treatment conditions of the metal and ceramic brackets' surface: (1) airborne particle abrasion with 30-μm aluminum trioxide particles modified by silica (CoJet System, 3M ESPE AG) and (2) no treatment (control). For the silica-coating process, the sandblasting device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark) was used and filled with 30 μm silicon dioxide (CoJet Sand, 3M ESPE AG). In accordance with the manufacturer's instructions, the abrasive was applied vertically to the metal ($n = 10$) and ceramic ($n = 10$) bracket surfaces at 10 mm with 0.25 MPa pressure for 15 seconds. Then residual blast-coating agents were removed with a stream of dry, oil-free air. One coat of silane (ESPE-Sil AG, Seefeld, Germany) was applied to conditioned specimens using a clean brush and allowed to air-dry (5 minutes) according to manufacturer's recommendations.

The silane used in ESPE-SIL is distinguished by two different polar ends on the molecule. The alkoxy groups of the silanol unit, (RO) 3Si group, forms a chemical bond with the silicatized surface. The methacrylate groups can then be copolymerized with the monomers of the resin. In the control group, no treatments were applied on the surfaces of the metal and ceramic brackets.

After preparing bracket surfaces, labial and lingual surfaces of teeth were etched for 15 seconds with 35% phosphoric acid etch (3M Dental Products, St Paul, Minn), rinsed for 20 seconds, and air-dried for 5 seconds with oil-free air. Then the bonding agent containing Bis-GMA (Light Bond, Reliance Orthodontic Products, Ithaca, Ill) was applied in a thin layer, excess resin was removed with air, and it was light polymerized (Hilux 250, First Medica, Greensboro, NC, light intensity of 600 mw/cm^2) for 20 seconds according to the manufacturer's instructions. Brackets were bonded to the enamel surfaces with light polymerized Bis-GMA resin composite luting cement (Light Bond, Reliance Orthodontic Products). The bracket was placed onto the ceramic surface by using bracket pliers under manual control. Before light polymerization, excess resin was removed from the bracket periphery, and polymerization of the luting resin was performed for a total of 30 seconds; 10 seconds over the brackets faces and 20 seconds interproximally (10 seconds mesial, 10 seconds distal). The bonding procedures were carried out by the same operator.

Test specimens were stored in distilled water at 37°C for 1 week. Specimens were then subjected to 5000 thermocycles (custom-made device by Ankara University, Ankara, Turkey) between 5°C and 55°C, with a transfer time of 30 seconds and a dwell time of 30 seconds.[8] After thermocycling, they were embedded in autopolymerizing clear acrylic resin material (Orthocryl EQ; Dentaurum, Ispringen, Germany, #030424), and the specimens were mounted in a universal testing (Lloyd-LRX; Lloyd Instruments, Fareham, UK) machine with a custom shear test apparatus. Shear bond test was applied at a crosshead speed of 0.5 mm/min until failure. The bond strength was expressed in megapascals, as derived from dividing the failure load (N) by the bonding area (mm^2).

Debonded specimen surfaces were examined with a stereomicroscope (Leica MZ 12, Leica Microsys-

Table 1. Results of *t*-test for Metal and Ceramic Brackets

Groups	n	Mean	SD	*t*	df	*P*
Metal brackets						
Tribochemical silica coating + silane	10	14.2	1.7	3.3	18	**
No-treated (Control)	10	11.9	1.3			
Ceramic brackets						
Tribochemical silica coating + silane	10	25.9	4.4	5.4	18	****
No-treated (Control)	10	15.6	4.2			

** $P = .01$; **** $P = .0001$.

Table 2. Modes of Failure of Groups According to ARI[a,b]

Surface Treatment	ARI Index (%)			
	0	1	2	3
Metal brackets				
Tribochemical silica coating + silane	20 (2)	60 (6)	20 (2)	0 (0)
No-treated (Control)	10 (1)	30 (3)	40 (4)	20 (2)
Ceramic brackets				
Tribochemical silica coating + silane	30 (3)	60 (6)	10 (1)	0 (0)
No-treated (Control)	20 (2)	40 (4)	30 (3)	10 (1)

[a] ARI indicates adhesive remnant index.

[b] ARI: 0, no composite left on tooth; 1, less than half of composite left on tooth; 2, more than half of composite left on tooth; 3, all the composite on tooth. Values in the brackets show number of groups.

tems, Bensheim, Germany) at original magnification 80× to assess the mode of failure. Afterward, each tooth was assigned an Adhesive Remnant Index (ARI) value according to the following criteria:[22] 0, no adhesive left on the tooth; 1, less than half of the adhesive left on the tooth; 2, more than half of the adhesive left on the tooth; and 3, all adhesive left on the tooth.

Statistical analysis was performed with statistical software (SPSS 12.0; SPSS Inc, Chicago, Ill). Student's *t*-tests were used to compare the mean shear bond strength of groups ($\alpha = 0.05$).

RESULTS

t-test Results

Mean bond strengths for each group, with standard deviations, are shown in Table 1. The *t*-test result indicates that there were significant differences in the shear bond strength values between the groups treated with tribochemical silica coating followed by silanization and the controls in metal and ceramic brackets (Table 1). The higher bond strength of 14.2 ± 1.7 MPa was obtained with tribochemical silica coating followed by silanization in metal brackets compared with the nontreated control group (11.9 ± 1.3 MPa) ($P < .01$). Similarly, ceramic brackets (25.9 ± 4.4 MPa) treated with silica coating and silanization showed higher bond strength compared with nontreated ceramic brackets (15.6 ± 4.2 MPa) ($P < .0001$).

ARI Index

Table 2 shows the modes of failure according to ARI for brackets and is expressed in percentages. Nontreated metal brackets showed 10% ARI-0, 30% ARI-1, 40% ARI-2, and 20% ARI-3. The silica-coated + silanization group of metal brackets showed 20% ARI-0, 60% ARI-1, 20% ARI-2, and 0% ARI-3. The result indicated that the debonded specimens failed in the resin and at the enamel-adhesive interface (ARI-3 = 0%) (Figure 1).

Nontreated ceramic brackets showed 20% ARI-0, 40% ARI-1, 30% ARI-2, and 10% ARI-3. On the contrary, in the silica-coated + silanized group of ceramic brackets the specimens did not debond at the bracket and adhesive interface (ARI-3 = 0%). It showed 30% ARI-0, 60% ARI-1, 10% ARI-2, and 0% ARI-3 (Figure 2). No enamel fracture was observed in any group tested.

DISCUSSION

The results of this study confirmed that tribochemical silica-coating system increased the bond strength of metal and ceramic brackets to enamel. The effect

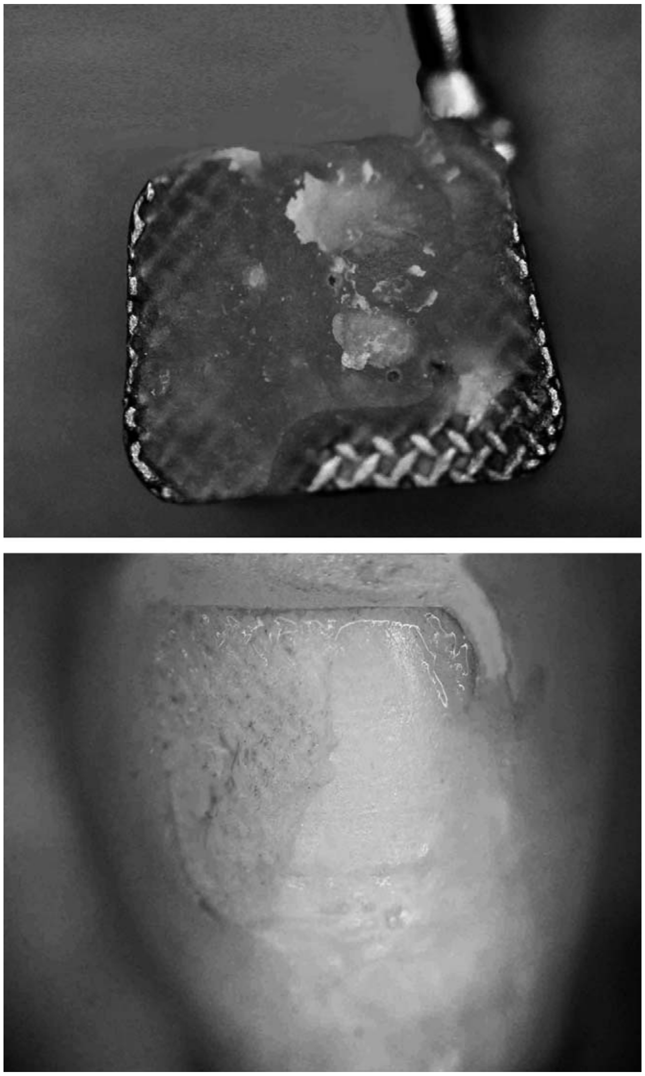

Figure 1. Appearance of bracket base and tooth surface in the silica-coated + silanized group of metal brackets after debonding.

Figure 2. Appearance of bracket base and tooth surface in the silica-coated + silanized group of ceramic brackets after debonding.

of tribochemical silica-coating system on bond strength can be explained by two mechanisms: the creation of a topographic pattern allowing for micromechanical bonding among bracket, resin luting agent, and enamel; and the chemical bond formed between the silica-coated metal and ceramic surface and resin material.[23] In principle, the silica-coating systems are used to obtain higher bond strength between the luting agent and the metal or ceramic surface (or both) with aid of the high-speed surface impact of the alumina particles modified by silica and followed by silanization. It has been reported that the airborne particles can penetrate up to 15 μm into the ceramic (silica based) and metal (gold alloy) substrates.[24]

In general, silanes used to promote adhesion between dissimilar materials are trialkoxy silanes, of the general formula R-Y-SiX3, where R is a nonhydrolyzable organic group; Y, a linker; and X, the hydrolyzable groups. Silanes have a dual reactivity and they have to be activated. The nonhydrolyzable functional group, (eg, methacrylate) can polymerize with the resin composite monomers containing C=C double bonds. The hydrolyzable alkoxy groups can react with a hydroxyl group–rich inorganic substrate, such as a silica surface, forming chemical bonds.[25]

Furthermore, the silane agent also contributed to the improved surface wettability to resin.[12,14,26] Silica coating and silanization of surfaces can be achieved by different methods. The silica-coating methods are different from each other regarding their chemistry or techniques. Rocatec is analogical with CoJet system: both use the tribochemical concept. However, Silicoater® Classical (Heraeus-Kulzer, Wehrheim, Germany) is based on thermal silica-coating systems, and they have been used in dental laboratories for about 20 years, but currently not so intensively.[27,28] The CoJet system is a method introduced in the dental market to create silica-coating surface for clinical procedures, such as the intraoral repair of fractured metal ceramic and all-ceramic restoration.[17–19]

Previous in vitro investigations revealed that the bond strength of stainless steel metal mesh brackets can be increased by sandblasting, sandblasting plus silane treatment, Rocatec (silane treatment and opaquer), and Silicoater® Classical.[29,30] Newman et al[30] found that bond strength between metal brackets and enamel improved through the following techniques: sandblasting (10.8 MPa), sandblasting and silanating (11.9 MPa), Rocatec system (10.8 MPa), Silicoater® Classical (13.2 MPa), and control (9.0 MPa).

In this study, results showed that the stainless steel orthodontic brackets treated with silica coating and silanization (14.2 MPa) had higher bond strength compared with the no-treatment control group (11.9 MPa), which is in agreement with previous studies.[8,30] A review of the relevant literature indicates that CoJet system used on the silica-based ceramic and metal restorations (noble and base metal alloys) increased the bond strength of different brackets such as polycarbonate and stainless steel metal.[31–33] Although this method previously was used on restoration surfaces to obtain higher bond between brackets and restorations, it has not been used on bracket surfaces for bracket bonding to enamel.

It has been suggested that clinically adequate bond strength for a metal orthodontic bracket to enamel should be 6 to 8 MPa.[34–36] The mean shear bond strengths of metal and ceramic brackets to enamel achieved in this study fell within this range or exceeded these limits and therefore could be considered sufficient for clinical applications.

Tribochemical silica coating followed by silanization evidently enhanced the bond between the metal and ceramic brackets and the resin composite. In general, increased bond strength resulted in failures within the resin and the enamel-adhesive interface.[8,31] In this study, treated metal and ceramic specimens exhibited cohesive failures in resin and adhesive failures at the enamel–adhesive interface without enamel fracture, which is similar to other studies.[8,31,37]

An important requirement in bracket bonding is that there should be no or minimal risk of iatrogenic damage to the enamel surface during debonding.[8] Metal brackets ''flex,'' and distort on loading, decreasing the shear forces exerted on tooth enamel during debonding.[38] Ceramic brackets, however, are rigid and unyielding. It can be seen that the shear bond strengths between enamel and bracket exceeding 23 kg (50.7 pounds or 225 N) are to be avoided.[8,39] In this study, although mean bond strength of ceramic brackets treated with tribochemical silica coating followed by silanization showed higher bond strength than these limits (~29 kg or 67 pounds or 300 N), no enamel fracture was observed during debonding in this groups. Meanwhile, the shear bond strength values of 15.6 MPa for nontreated ceramic brackets in our study were similar to mean values of 14.4 MPa in another study obtained with light-cured resin composite and the same type of ceramic brackets.[37]

In this study, modes of failure of groups are recorded according to the ARI. The results show that the most common failures in the ceramic specimens treated with silica coating followed by silanization are ARI-1. Similarly, treated metal specimens showed ARI-1 type of failures. This supports the high bond strengths in the treated groups when compared with the nontreated groups. In general, increased bond strength resulted in failures within the resin so that some resin was left on both the bracket and the ceramic surfaces. Bond strengths are also influenced by the type of luting cement. In this study, light-polymerized Bis-GMA resin composite with a silane-treated quartz and glass filler, Light Bond, was used as a luting resin. It was found that the mean bond strength of Light Bond is clinically acceptable for use in stainless steel orthodontic brackets.[40] Further investigations could also be performed with other resins.

Five-thousand thermocycling was released before the bond test in this study. When no or limited thermocycling is performed, high bond strengths can be found that do not correspond to in vivo conditions.[34,36] Therefore, the clinical relevance of some previous studies appears limited, and this must be taken into consideration when comparing the results of the studies. Limited numbers of specimens and the lack of comparison with silica-coating techniques and other surface conditioning methods such as sandblasting, silanization with silane coupling agent, and hydrofluoric acid (for ceramic brackets) may be seen as a limitation of this study. Further investigations could be performed with the CoJet system, which provides a silica coating and silanization on debonding brackets with different resin composite and compare with other surface conditioning methods.

CONCLUSIONS

- Chairside tribochemical silica-coating systems (CoJet) significantly increased the mean bond strength values of the metal (stainless steel) and the ceramic (alumina) brackets to enamel.
- The most common failures in the ceramic and metal brackets treated with silica coating followed by silanization are ARI-1. This supports the high bond strengths in treated groups when compared with the no-treated groups.

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