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Shear Bond Strength of Resin Cement Bonded to Alumina Ceramic after Treatment by Aluminum Oxide Sandblasting or Silica Coating

Sheila Pestana Passos, DDS, MSc, PhD,[1] Luiz Felipe Valandro, DDS, MSc, PhD,[2] Marco Antonio Bottino, DDS, PhD,[1] Maria Jacinta Moraes Coelho Santos, DDS, MSc, PhD,[3] & Gildo Coelho Santos Jr, DDS, MSc, PhD[3]

[1]Department of Dental Materials and Prosthodontics, São Paulo State University, São José dos Campos, Brazil
[2]Department of Restorative Dentistry, Federal University of Santa Maria, Brazil
[3]Department of Restorative Dentistry, University of Western Ontario, London, Canada

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Correspondence
Sheila Pestana Passos, Department of Dental Materials and Prosthodontics, São Paulo State University, Av Engenheiro Francisco José Longo, 555/1306. São Dimas, CEP: 12245000, São José dos Campos, São Paulo, Brazil. E-mail: sheilapestana@hotmail.com

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Abstract

Purpose: To evaluate the shear bond strength and bond durability between a dual-cured resin cement (RC) and a high alumina ceramic (In-Ceram Alumina), subjected to two surface treatments.
Materials and Methods: Forty disc-shaped specimens (sp) (4-mm diameter, 5-mm thick) were fabricated from In-Ceram Alumina and divided into two groups (n = 20) in accordance with surface treatment: (1) sandblasting by aluminum oxide particles (50 μm Al_2O_3) (SB) and (2) silica coating (30 μm SiO_x) using the CoJet system (SC). After the 40 sp were bonded to the dual-cured RC, they were stored in distilled water at 37°C for 24 hours. After this period, the sp from each group were divided into two conditions of storage (n = 10): (a) 24 h—shear bond test 24 hours after cementation; (b) Aging—thermocycling (TC) (12,000 times, 5 to 55°C) and water storage (150 days). The shear test was performed in a universal test machine (1 mm/min).
Results: ANOVA and Tukey (5%) tests noted no statistically significant difference in the bond strength values between the two surface treatments ($p = 0.7897$). The bond strengths (MPa) for both surface treatments reduced significantly after aging (SB-24: 8.2 ± 4.6; SB-Aging: 3.7 ± 2.5; SC-24: 8.6 ± 2.2; SC-Aging: 3.5 ± 3.1).
Conclusion: Surface conditioning using airborne particle abrasion with either 50 μm alumina or 30 μm silica particles exhibited similar bond strength values and decreased after long-term TC and water storage for both methods.

The cementation procedure has a crucial impact on the longevity of the restoration. The adhesive cementation technique requires pretreatment of both dental tissues[1] and restorative material before luting.[2,3] Several studies have shown an additional reinforcement of all-ceramic restorations when cemented with resin-based luting materials. The additional resistance over the ceramic material is due to the effective bond of the resin cement (RC) to the ceramic and to the dentin, which allows the occlusal forces to be equally distributed over the ceramic and dentin.[4,5] In addition, many studies have resulted in the evaluation of bonding between ceramic and RC varying the pre-cementation treatment, especially with laboratory mechanical tests.[6–15]

Ceramic materials with a high glass phase content have shown excellent adhesion to resin cements when submitted to surface treatments.[16–18] Sandblasting (SB) and etching with hydrofluoric acid, followed by application of the silane agent have resulted in high bond strengths.[16,18,19] However, ceramic systems with high alumina content (In-Ceram Alumina, VITA Zahnfabrik, Bad Säckingen, Germany) have not demonstrated high bond strength values to the RC with commonly used pre-cementation treatments.[20] The scarcity of glass, rich in silica phase, disallows a solid bond with the silane, resulting in low bond strength values,[21] such as in Nakamura et al's study,[8] which presented shear bond strength values from 17.0 to 38.7 MPa after 24 hours and from 1.9 to 25.2 MPa after aging, when RelyX ARC RC was evaluated, depending on the silane agent studied. Diverse treatments have been suggested[10,12,22,23] with the aim of promoting high bond strengths between resin cements and ceramic materials with high alumina content.

Dental ceramics with low glass phase content may be categorized as acid-resistant ceramics (glass-infiltrated alumina/zirconia; tetragonal zirconia stabilized by yttrium oxide [Y-TZP]; densely sintered alumina). For these acid-resistant ceramic systems, SB has been the treatment of choice to optimize adhesion with RC.[6,7,24–28] Silica and aluminum oxide particles with sizes from 30 to 250 μm have been used for this purpose.[29–33] High bond results have been achieved when glass-infiltrated alumina ceramic was abraded with aluminum oxides and bonded to 10-methacryloyloxydecyl dihydrogen phosphate (MDP) based resin cements or when treated with tribochemical silica and bonded to resin cements without MDP.[6,7,24–28]

Due to the increased use of all-ceramic restorations with high alumina content, it is important to evaluate the effect of different surface treatments on bond strength values between ceramic materials and resin cements. RelyX ARC resin composite cement is a reliable product frequently used in daily clinical practice and represents one of the most thoroughly investigated resin-based cements in the literature.[34–36]

Thus, the aim of this study was to verify the shear bond strength of a successful RC and a high content alumina ceramic subjected to different surface treatments to test the bond durability. The null hypotheses to be tested were: (1) alumina ceramic conditioning treatments do not influence bond strength at cement/ceramic interfaces and (2) 150 days of water storage and TC do not affect adhesion to alumina ceramic when sandblasted by aluminum oxide particles or silica coating (SC).

Materials and methods

One type of ceramic and RC was selected for this experiment, and the ceramic surface treatment was performed using SC or SB. The materials used in the present study are listed in Table 1. Forty In-Ceram Alumina (Vita Zahnfabrik, Bad Säckingen, Germany) disc-shaped specimens (4-mm diameter, 5-mm thick) were fabricated, and then a glass-infiltration process was done, according to the manufacturer's instructions.

A silicone impression was made (Elite HD, Zhermack, Badia Polesine, Italy, LOT: 18443) to fabricate standard rectangular boxes (1.0-cm high, 2.0-cm wide, 3.5-cm long), in which the ceramic cylinders were placed in the center, and included in acrylic resin. After this, the exposed ceramic surface (3-mm diameter) was polished in a machine using silicone carbide papers (3M ESPE, St. Paul, MN) in sequence (600, 800, 1200 grit) under water cooling. They were then cleaned in an ultrasonic bath (Vitasonic, Vita Zanhfabrik) for 5 minutes in distilled water and divided into two groups (n = 20), in accordance with the surface treatment:

(1) SB: chairside airborne particle abrasion with 50 μm Al_2O_3 particles was applied using an intraoral air abrasion device (Microetcher, Danville Inc., San Ramon, CA) from a distance of approximately 10 mm, in circular motions perpendicular to the disk surface at a pressure of 2.8 bars for 15 seconds and air dried.[37]
(2) SC: the silica coating process was done using the same intraoral abrasion device under the same conditions as the SB group, and using 30 μm SiO_x (CoJet-Sand®) particles.

After treatment, the ceramic surfaces were rinsed with air/water spray for 30 seconds (except for the SC group) and air-dried. Silane agent (RelyX Ceramic Primer) was applied, allowing time for evaporation. A matrix (2-mm diameter, 4-mm high) was placed in the center of the surface of each specimen. A dual-cured RC was selected (RelyX ARC). Dual-cured cements were developed to combine the most desirable properties of the light- and chemical-cured systems.[38] RelyX ARC luting agent delivers precise dosages with a click dispenser. The cement was dispensed onto a mixing pad and mixed for 10 seconds and was inserted into the matrix on the treated ceramic surface. Then, with use of a Centrix syringe,[7,30] the specimen was light cured for 40 seconds, through the exposed surface, by a light-cured unit (XL 3000 – 3M ESPE; light output: 500 mW/cm^2). A cylinder of cement was used to obtain just one interface as performed in previous studies.[6,7] The ceramic-resin cement set was removed from the matrix after 10 minutes. The cement was light activated in all the lateral faces for 40 seconds. The specimens were stored in distilled water at 37°C for 24 hours, since the significant polymerization reaction finishes within 24 hours post-mix or post-light activation for RelyX ARC, according to Yan et al.[39]

The specimens were randomly divided into two testing conditions (n = 10). In immediate conditions, specimens were submitted to the shear bond test after 24 hours of storage. In aging conditions, specimens were submitted to TC (12,000 cycles; 5 to 55°C, dwell time: 30 seconds, transfer time: 2 seconds) (Nova Etica, São Paulo, Brazil) and stored in distilled water at

Table 1 Materials used

Material	Application procedure	Batch number	Manufacturer
50 μm Al_2O_3 particles	Applied using an intraoral air abrasion device perpendicular to the surface at a distance of 10 mm for 15 seconds at a pressure of 2.8 bars[34]	#20919	Polidental Ind. e Com. Ltda, Sao Paulo, Brazil
CoJet-Sand®	Same conditions as for specimens in the Al_2O_3 groups	#351794	3M Dental Products, St. Paul, MN
RelyX ARC	Dispensed onto a mixing pad and mixed for 10 seconds; applied on the bonding surface of the block; light-cured for 40 seconds; then light activated in all lateral faces for 40 seconds	#EJFG	3M ESPE, St. Paul, MN
RelyX Ceramic Primer	Applied allowing enough time to evaporate.	#6XH	3M ESPE, St. Paul, MN

Table 2 Mean bond strength values and standard deviation of test groups

Groups	Shear bond strength (MPa)	Failure types			
		ADHES	COHES-Cer	COHES-Cem	COHES-Mixed
SB-24h*	8.2 ± 4.6^a	7	0	2	1
SB-aging**	3.7 ± 2.5^b	9	0	1	0
SC-24h*	8.6 ± 2.2^a	7	0	1	2
SC-aging**	3.5 ± 3.1^b	8	0	2	0
	Total	31	0	6	3

Note: Failure between ceramic and cement (ADHES); cohesive failure of the ceramic (COHES-Cer); cohesive failure of the cement (COHES-Cem); cohesive failure of cement and ceramic (COHES-Mixed).

*24h: shear bond test after 24 hours; **aging: TC 12,000 cycles, 5 to 55°C and water storage in distilled water at 37°C for 150 days. Same superscript letters indicate no significant differences ($p > 0.05$).

37°C for 150 days and then submitted to the shear bond test. Thus, four groups were obtained with the "surface treatment" (2 levels) and "storage condition" (2 levels).

Shear bond strength testing was done following ISO standards 6872 and 11405. The tests were performed in a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil) at a 1 mm/min crosshead speed until fracture at 25°C. The load to failure (F in N) was recorded, and the mean shear bond strength (σ) values (in MPa) were calculated ($\sigma = F/A$) (F = force and A = area). Values were statistically analyzed using two-way ANOVA and Tukey tests ($\alpha = 0.05$). The fractured ceramic surfaces were analyzed by light microscopy.

All debonded surfaces were examined by microscope (MP 320, Carl Zeiss, Jena, Germany) at 50×, and some specimens were selected for analysis under scanning electron microscopy (SEM) (Jeol JSM T330A, Jeol Ltd, Tokyo, Japan) at 500× magnification for observation of failure type. Failures were classified as follows: adhesive between ceramic and cement, cohesive failure of the cement, cohesive failure of ceramic, mixed, and cohesive failure of cement and ceramic.

The shear bond data were analyzed by two-way ANOVA, with shear bond strength as the dependent variable. Surface treatment and storage conditions were the independent factors (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL). In all tests, $p \leq 0.05$ was considered statistically significant.

Results

The mean shear bond strength values and standard deviations are shown in Table 2. The mean bond strength values of groups treated with aluminum oxide (SB) and CoJet System (SC) were not significantly different before and after aging ($p = 0.7897$), and no interaction effects were observed; however, bond strengths for both conditions were damaged by aging, showing that bond durability was impaired by the storage and TC condition.

SEM photographs of specimens tested are presented in Figure 1. The most common failure mode observed for all groups was adhesive failure after aging (Table 2). Some cohesive failures in the cement were observed.

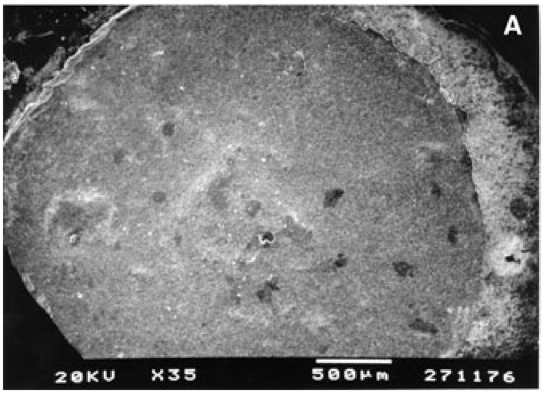
Discussion

Mechanical tests such as pull-out,[12] shear bond strength,[3,6–8,24,31] tensile,[14,27] or microtensile tests,[10,13,32,33,37] have been used to evaluate bond strength. Therefore, mechanical laboratory tests are an important factor to consider. In this study, shear bond strength testing was chosen because it is a commonly used method and has proven to be reliable.[3,31] Shear strength testing has been discussed because cohesive failures are found in the bonding substrate that may cause false interpretation of the resultant bond strength data as a consequence of the nonuniform interfacial stresses.[15,27] However, in the present study, mainly adhesive failures were observed (Fig 1), and cohesive failures in the composite cylinders were rare (Table 2), indicating the validity of the applied testing method. Mixed and cohesive fracture patterns are clinically preferable to totally adhesive failure, since the adhesive type of failure is usually associated with low bond strength values.[1]

It is difficult to define a target value for clinical success, since the retentive effect can help keep the crown on the preparation using nonadhesive cement. The resin bond to the ceramic surface can optimize the retention of the crown. In this study, the bond strength values were very low for all groups and could be considered unsuccessful since 20 MPa is a standard target value for bonding studies of bonding systems to enamel.

In the present study, the particle sizes used (50 μm Al_2O_3, 30 μm SiO_x) presented no difference in bond strength values after 24 hours. These results are in accordance with previous studies that used acid-resistant ceramic.[13,33] Oyagüe et al[13,33] compared both conditioning treatments with different resin-based cements. These studies observed that the microtensile bond strengths of Clearfil Esthetic Cement and RelyX Unicem to zirconia were similar when used with SC and SB; however, other studies presented superior results when bonding glass-infiltrated alumina ceramics coated by silica oxide to the resin-based cements.[6,7]

The bonding interface between the ceramic material and the RC into the oral cavity is susceptible to thermal, chemical, and mechanical stresses. Water storage and TC are frequent methods used to simulate the aging conditions achieved into the oral cavity. Some studies have shown that the aging process affects bonding durability among different



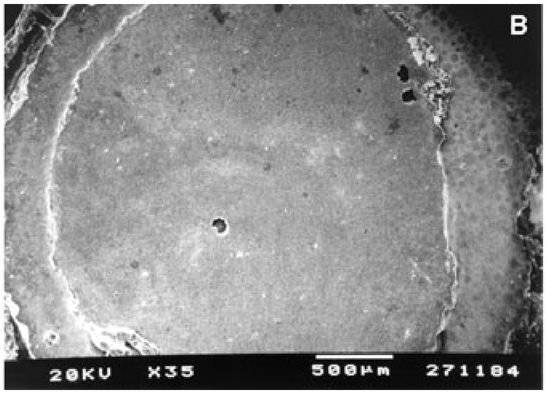


Figure 1 SEM microphotographs of the debonded surface after aging (TC and 150 days): (A) Specimen treated with 50 μm Al_2O_3 particles; (B) Specimen treated with SC. A complete detachment from the substrate occurred in both specimens (adhesive failure).

materials.[27] The present investigation observed that SB of the In-Ceram Alumina specimens with aluminum or silica oxide did not promote stable resin shear bond strength. Water storage and TC allowed the aging effect in the humid environment and thermal stress, respectively, which resulted in decreased bond strength values between the Bis-GMA resin-based cement and the In-Ceram ceramic when compared to the initial values of shear bond strength, after 24 hours, for both groups (Table 2). Nakamura et al[8] observed that no surface treatment between In-Ceram Alumina ceramic and RelyX ARC cement achieved the lowest shear bond strength at 24 hours and after TC (20,000 cycles).

The results of this study are similar to previous studies, which found reduced bond strength values between RC and glass-infiltrated alumina ceramic when sandblasted with aluminum oxide after storage and/or TC.[7,27] It has been proven that water, due to its small molecular size and high molar concentration, can penetrate into nanometer-size free-volume spaces between polymer chains or cluster around functional groups capable of hydrogen bonding, resulting in a decrease in thermal stability and polymer plasticization.[40] Water sorption may have determined the cement hydrothermal degradation during aging (such as the creation of swelling stresses).[20] Additionally, Özcan et al[11] verified no difference in the shear bond strength between these methods after TC (6000 cycles) using 50 μm Al_2O_3 and 110 μm SiO_x, in accordance with the present study.

The findings of this study require confirmation of the first null hypothesis, as the bond strength at the cement/ceramic interfaces was not affected by conditioning treatment; and required rejection of the second null hypothesis, as 150 days of water storage and TC affected the bond strength for both conditions. However, the results of this experiment provided only an indication of the possible performance of resin luting agents to alumina ceramics. These results need to be refined before prospective clinical studies can be performed, because the clinical environment is more complex than in vitro tests.

Conclusions

Within the limits of the current experiment, the following conclusions can be drawn:

(1) Zirconia surface conditioning using airborne particle abrasion with either 50 μm alumina or 30 μm silica particles exhibited similar bond strength values ($p \leq 0.05$).
(2) Water storage and TC played important roles in resin-cement/alumina-ceramic bond degradation ($p \leq 0.05$).

Clinical Relevance

Surface conditioning using airborne particle abrasion with either 50 μm alumina or 30 μm silica particles exhibited similar bond durability.

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