Microtensile Bond Strength of a Resin Cement to Silica-Coated and Silanized In-Ceram Zirconia Before and After Aging

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Purpose: This study compared the microtensile bond strength of resin-based cement (Panavia F) to silica-coated, silanized, glass-infiltrated high-alumina zirconia (In-Ceram Zirconia) ceramic in dry conditions and after various aging regimens. ***Materials and Methods:*** The specimens were placed in 1 of 4 groups: group 1: dry conditions (immediate testing without aging); group 2: water storage at 37°C for 150 days; group 3: 150 days of water storage followed by thermocycling (× 12,000, 5°C to 55°C); group 4: water storage for 300 days; group 5: water storage for 300 days followed by thermocycling. ***Results:*** Group 1 showed a significantly higher microtensile bond strength value (26.2 ± 1 MPa) than the other aging regimens (6.5 ± 1, 6.2 ± 2, 4.5 ± 1, 4.3 ± 1 MPa for groups 2, 3, 4, and 5, respectively) ($P < .01$). ***Conclusion:*** Satisfactory results were seen in dry conditions, but water storage and thermocycling resulted in significantly weaker bonds between the resin cement and the zirconia.
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The mechanical properties of feldspar ceramics improve significantly with an increase in the amount of alumina (Al_2O_3) in their composition, resulting in more predictable metal-free restorations.[1] An example of a glass-infiltrated high-alumina zirconia, In-Ceram Zirconia (Vita), has a high crystalline content (nearly 80%: 67% aluminum oxide and 13% tetragonal zirconia crystals). This ceramic contains only 20% lanthanum aluminosilicate glass, which makes it an acid-resistant ceramic substrate. To achieve better adhesion of resin-based luting cements to restorations made of such reinforced ceramics, conditioning of the inner surfaces of these ceramics with either airborne particle abrasion using Al_2O_3 particles[2] or via laboratory[3,4] or chairside tribochemical silica coating and silanization has been recommended.[5,6] The chairside surface conditioning option eliminates possible contamination during delivery of the restoration from the laboratory to chairside, and the smaller particle size results in less damage at the restoration margins.[5]

The durability of bonding between resin cements and reinforced ceramics is good in dry conditions, but the bond tends to weaken after water storage and/or aging conditions such as thermocycling.[7,8] Both water storage and thermocycling aging regimens have not been followed in a standard manner in dental research, and the latter also requires the employment of an expensive apparatus in the laboratory.[9] The objective of this study was to evaluate the effect of long-term water storage, aging via thermocycling, and combinations of both on the bond strength between a phosphate monomer–based resin cement and In-Ceram Zirconia after conditioning with chairside silica coating and silanization.

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Materials and Methods

Fifteen blocks ($6 \times 6 \times 4$ mm) of a glass-infiltrated alumina-zirconia ceramic (In-Ceram Zirconia) were fabricated according to the manufacturer's instructions. The surfaces were finished with a 1,200-grit silicone carbide abrasive. The blocks were duplicated in hybrid composite (W3D Master, Wilcos) and ultrasonically cleaned in distilled water for 3 minutes. Then one surface (6×6 mm) of each block was conditioned via chairside tribochemical silica coating with 30-µm SiO_x (Micro-Etcher, Danville Inc, perpendicular to the surface at a distance of 10 mm for 20 s at a pressure of 2.8 bar; CoJet-Sand, 3M/ESPE). Five minutes were allowed to elapse for silane (ESPE-Sil, 3M/ESPE) reaction.

Each ceramic block was luted to the corresponding resin composite block with the resin cement (Panavia F, Kuraray) according to the manufacturer's recommendation under vertical load (750 g) for 10 min. During this period, excess material was removed, and each surface was light polymerized for 40 s (XL 3000, 3M/ESPE; light output: 500 mW/cm^2). An oxygen-blocking agent (OXYGUARD, Kuraray) was applied to all cementation surfaces. The blocks were washed and rinsed with water and then stored in distilled water at 37°C for 24 hours.

Production of Beam Specimens

The blocks were bonded with cyanoacrylate glue (Super Bonder Gel, Loctite Ltd) to a metal base that was coupled to a cutting machine. The blocks were positioned perpendicular to the diamond disk. Slices were obtained using a low-speed diamond disk (no. 34570, Microdont) under water cooling. The peripheral slices (0.5 mm) were eliminated so that the results would not be influenced by excess cement, an insufficient amount of cement, or irregularities at the interface.

Thereafter, 4 sections (thickness: 0.8 ± 0.1 mm) of each specimen were created. Each section then was rotated by 90 degrees and fixed again to the metallic base. Twenty-five rectangular specimens with an adhesive area of about 0.6 mm^2 and length of about 8 mm were thus obtained from each block. The sticks from each luted block were randomly divided into 5 groups based on type and duration of aging regimen (Table 1).

Table 1 Experimental groups

Group	Storage regimen
1	Dry condition (immediate testing after production)
2	Storage in distilled water at 37°C for 150 d
3	150 d + thermocycling (× 12,000, 5°C to 55°C)*
4	300 d
5	300 d + thermocycling*

*Dwelling time: 30 s; transfer time from one bath to the other: 2 s.

Microtensile Bond Strength Test

The ends of each specimen were fixed with cyanoacrylate adhesive in an adapted device. Microtensile bond strength testing was then performed in a universal testing machine (DL-1000, EMIC) (1 mm/min^{-1}). The bond strength σ (MPa) was calculated according to the formula $\sigma = L/A$, where L is the load (in Newtons) required to rupture the specimen and A is the interfacial area (mm^2) (measured with a digital caliper before testing). The mean bond strength values from the specimens of each block were analyzed by 1-way analysis of variance ($\alpha = .05$) and the Tukey test.

Microscopic Analysis

The tested specimens were analyzed in an optical microscope (MP 320, Carl Zeiss) (×100) for failure analysis (adhesive, cohesive, or mixed). Some specimens were further evaluated in a scanning electron microscope (Jeol JSM 5400, Jeol Ltd) (×75 to ×2,000 magnification).

Results

Before microtensile bond strength testing, 4 failures in group 2, 9 in group 3, 8 in group 4, and 5 in group 5 occurred. No specimens in group 1 failed before testing. Pretest failure values were considered 0 MPa.

Significant differences were observed between the 5 experimental groups (P=.0001). Dry conditions (group 1) resulted in a significantly higher mean bond strength value (26.2 ± 1 MPa) versus the other aging regimens (6.5 ± 1 MPa for group 2, 6.2 ± 2 MPa for group 3, 4.5 ± 1 MPa for group 4, and 4.3 ± 1 MPa for group 5) ($P <$ .01; Tukey test) (Fig 1). No significant difference in the resin-ceramic bond was found between the long-term water storage (groups 2 and 4) and the water storage/thermocycling aging regimens ($P >$.05).

Microscopic analysis of the failed specimens demonstrated exclusively mixed failures in all experimental groups. There were adhesive failures at the ceramic-cement junction, combined with some small areas of cohesive failures in the cement at the corner of the interface (Fig 2).

Discussion

In vitro tests are useful tools for predicting the durability of cement-ceramic adhesion in dental restorations. Adhesive joints are often prone to water absorption, degradation, and shrinkage because of thermal changes. Therefore, extended aging protocols would deliver more information that could be extrapolated to the worst-case clinical situations. The shear test is routinely used and is

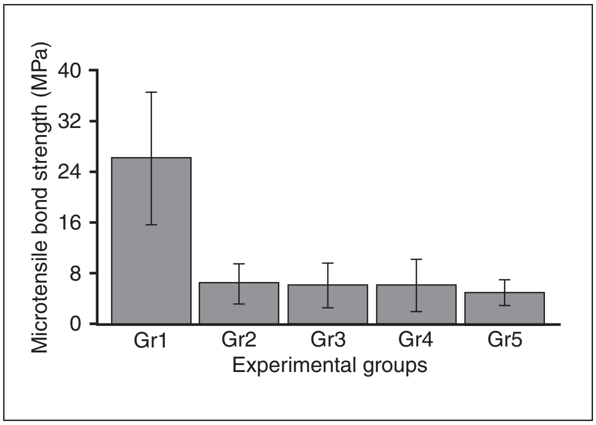

Fig 1 Microtensile bond strength results (MPa) of Panavia F to In-Ceram Zirconia in dry conditions and after various aging treatments.

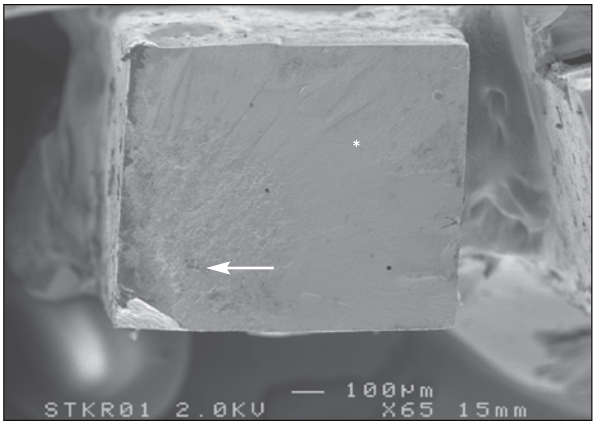

Fig 2 Representative scanning electron microscopic image of a typical mixed failure. The arrow indicates a cohesive failure in the cement. The asterisk indicates the adhesive failed region between the cement and the ceramic surface.

considered an adequate method to screen the performance of bonds between resin-based materials and either dentin or restorative materials. However, it has been previously reported that the shear test may create uneven stress distribution at the adhesive joint, leading to substrate failures.[10] For this reason, in this study, the microtensile test was employed to test the bond strength of a frequently used phosphate monomer–containing cement, Panavia F, which delivers higher bond strength compared to bisphenol glycidyl methacrylate–based cements,[7,8] to a glass-infiltrated, alumina-zirconia ceramic.

The results of this study seen in dry conditions are slightly better than those of a recent study,[8] in which Panavia was bonded to 50-µm alumina-treated Procera AllZirkon ceramic and tested after 3 days of water storage. However, the mean bond strength values obtained in this study after long-term water storage with or without thermocycling were significantly lower (4.3 to 6.5 MPa) than in the earlier study, when Panavia F was bonded with and without bonding agent and tested after 180 days of water storage followed by 12,000 thermal cycles (9.45 to 16.85 MPa). Although the ceramics used were not identical, the microtensile testing method seems to deliver poorer results than the shear test when the bond strength of resin cements is tested on reinforced ceramics. This could be attributed to the specimen preparation technique for microtensile testing, which differs from that of the shear test.

Conclusions

1. Microtensile bond strength tests of Panavia F to In-Ceram Zirconia demonstrated satisfactory results in dry conditions, but aging the specimens reduced the bond strength dramatically ($P = .0001$).
2. Long-term water storage of samples of the materials for either 150 or 300 days in distilled water at 37°C alone resulted in a similar aging effect versus long-term water storage followed by thermocycling for 12,000 cycles with respect to adhesion of the materials tested.

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