

# Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only

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**Abstract** The objectives of this study were (1) to evaluate the bond strength of four resin materials with various chemical compositions following the manufacturers' instructions only and (2) to test their durability in dry and thermal aged conditions when they were bonded to zirconia ceramic. Four types of resin materials namely, Panavia F 2.0, Multilink, SuperBond and Quadrant Posterior Dense, were attached to the disc-shaped zirconia ceramics (LAVA, 3M ESPE) using polyethylene molds and polymerized accordingly after the ceramics were wet ground finished and ultrasonically cleaned. The specimens were randomly divided into two groups for ageing conditions. While the dry groups were tested immediately after attachment of the resin cement, the other specimens were subjected to thermocycling ( $\times 6,000$ ,  $5-55^{\circ}\text{C}$ ). Bond strength results were significantly affected by the storage condition ( $p < 0.001$ ) and type of resin ( $p < 0.001$ ; analysis of variance). Panavia F 2.0 showed the highest bond strength results under dry conditions ( $9.6 \pm 4.1$  MPa). When manufacturers' instructions of the resin cements were followed, no adhesion (0 MPa) was achieved on the zirconia after 6,000 thermal cyclings including Panavia F 2.0.

**Keywords** Bond strength · Resin cement · Shear test · Y-TZP ceramic

## Introduction

New ceramic systems used in dentistry involve reinforced ceramic cores through dispersion with leucite, glass infiltration into sintered alumina and the use of high-purity alumina or yttrium-stabilized tetragonal zirconium dioxide (Y-TZP). These high-strength ceramics offer a wide variety of clinical applications such as fixed-partial dentures (FPDs), resin-bonded FPDs, posts or implant abutments in prosthetic dentistry. Among these ceramics, zirconia has the most favourable properties with flexural strength of 900 to 1,200 MPa, fracture resistance of more than 2,000 N and fracture toughness of 9–10 MPa/mm<sup>2</sup>, which is almost twice the value obtained for alumina-based materials and almost three times the value demonstrated by lithium disilicate-based ceramics [9]. The only problem related to their performance is that adhesion of the resin cements to such ceramics is inferior. There have been efforts made by some manufacturers and researchers to modify the surface properties of the zirconia by means of various air-abrasion methods followed by silanization [2, 6, 8, 10]. This approach has been recently criticized for possible sub-critical crack growth within zirconia [13].

While there is still no consensus in the dental literature that the surface-conditioning method would be more suitable for improved adhesion of the resin cement to such ceramics, manufacturers of some cements do not suggest additional surface conditioning to all ceramics including zirconia. Potential cements to be used for zirconia are based on bisphenol A diglycidylether methacrylate (Bis-GMA), 4-methacryloxyethyl trimellitate anhydride or methacryloxy

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decyl phosphoric acid (MDP) monomers. The dilemma for the clinicians remains whether to follow the manufacturers' instructions of the cements during cementing the FPDs made of zirconia or modify the instructions and follow the instructions of some manufacturers that suggest separate surface-conditioning protocols.

Therefore, the objectives of this study were to evaluate (1) the bond strength of four resin materials with various chemical compositions following the manufacturers' instructions only and (2) to test their durability under dry and thermally aged conditions. The tested hypothesis was that thermocycling would decrease the bond strength of all resins.

## Materials and methods

Disc-shaped (diameter=15 mm, thickness=2 mm) Y-TZP ceramics (LAVA, 3M ESPE, Seefeld, Germany) were embedded in polyethylene molds using polymethylmethacrylate (PMMA; Condular AG, Wager, Switzerland) with one side of the disc exposed for adhering the cement (Fig. 1). They were ground finished up to 1,200-grit silicone carbide abrasive under water cooling and ultrasonically cleaned in distilled water for 3 min.

The discs were randomly divided into eight groups depending on the cement type and the ageing condition to be applied ( $N=80$ ,  $n=10$ /per group; Table 1). Four types of resin materials were chosen: a light-polymerized (Panavia F 2.0, Kuraray, Medical Inc., Osaka, Japan), self-adhering (Multilink Ivoclar Vivadent, Schaan, Liechtenstein) and chemically polymerized cement (SuperBond, Sun Medical, LTD, Tokyo, Japan) where the resin composite (Quadrant Posterior Dense, Cavex, Harlem, The Netherlands) acted as the control material.



**Fig. 1** The Y-TZP disc specimen was embedded in the PMMA with the cementation surface exposed. Cement was applied incrementally, not exceeding 2 mm, to the surface using polyethylene molds (inner diameter=3.6 mm, height=5 mm) and polymerized accordingly

**Table 1** Testing groups considering the types of resin cements and the storage conditions (dry vs thermocycling—TC)

Type of resin cement	Storage condition <sup>a</sup>	Testing groups
Panavia F 2.0 <sup>b</sup>	Dry	Gr1
	TC	Gr2
Multilink <sup>c</sup>	Dry	Gr3
	TC	Gr4
Superbond <sup>d</sup>	Dry	Gr5
	TC	Gr6
Quadrant Posterior Dense <sup>e</sup>	Dry	Gr7
	TC	Gr8

<sup>a</sup> Dry condition—Shear test was performed immediately after cementation; TC condition—6,000 cycles between 5 and 55°C in deionized grade 3 water. The dwelling time at each temperature was 30 s, and the transfer time from one bath to another was 2 s (Willytech, Gräfelfing, Germany).

<sup>b</sup> (1) Conditioning the ceramic using chair-side air-particle abrasion device with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  for 15 s making circular movements from a distance of 10 mm; (2) rinsing the surface for 20 s with water and air drying for 5 s; (3) mixing equal amounts of paste A and B from the Panavia F 2.0 with a cement spatula for 20 s; (4) application of the mixed cement in the polyethylene mould onto the ceramic surface incrementally; (5) light polymerization (light output=500  $\text{mW}/\text{cm}^2$ ) of each increment for 20 s (Astralis<sup>®</sup> 5, Ivoclar Vivadent AG, Schaan, Liechtenstein).

<sup>c</sup> (1) Rinsing the surface for 20 s with water and air drying for 5 s; (2) applying Monobond-S on the ceramic surface with a microbrush, waiting 60 s and air drying; (3) application of the mixed cement in the polyethylene mould onto the ceramic surface incrementally (Ivoclar Vivadent AG, Schaan, Liechtenstein).

<sup>d</sup> (1) Rinsing the surface for 20 s and air drying for 5 s; (2) applying the Porcelain Liner M to the surface with a microbrush; (3) Mixing four drops of Quick Monomer, one drop of Catalyst S and one small scoop of the cement powder with a brush; (4) application of the mixed cement in the polyethylene mould onto the ceramic surface incrementally; (5) waiting for polymerization for 6 min (Sun Medical Co., LTD, Moriyama, Japan).

<sup>e</sup> (1) Rinsing the surface for 20 s and air drying for 5 s; (2) application of the mixed cement in the polyethylene mould onto the ceramic surface incrementally; (3) light polymerization (light output=500  $\text{mW}/\text{cm}^2$ ) of each increment for 40 s (Astralis<sup>®</sup> 5, Ivoclar Vivadent AG, Schaan, Liechtenstein; Cavex, Haarlem, The Netherlands).

## Shear bond test

The specimens were placed in the jig of the universal testing machine (Zwick ROELL Z2.5 MA 18-1-3/7 Ulm, Germany), and load was applied to the adhesive interface until failure occurred (crosshead speed=1.0 mm/min). The maximum force to produce fracture was recorded ( $\text{N}/\text{mm}^2=\text{MPa}$ ) using the corresponding software.

## Statistical analysis

Two-way analysis of variance (ANOVA) and post-hoc multiple comparisons were used to analyze the data (SPSS for Windows Version 10.05; SPSS, Chicago, IL) with the shear bond strength as the dependent variable.  $p$  values less

than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made using Tukey's adjustment test.

## Results

Bond strength results were significantly affected by the storage condition ( $p < 0.001$ ) and type of resin cement ( $p < 0.001$ ; ANOVA). Panavia F 2.0 showed the highest bond strength results under dry conditions ( $9.6 \pm 4.1$  MPa), but after  $\times 6,000$  thermocycling, all resin materials were debonded from the zirconia surfaces and considered as 0 MPa (Fig. 2).

## Discussion

Several studies have demonstrated that the bond strength of resin based materials to acid-resistant ceramics, especially for the Y-TZP ceramic, is neither durable nor stable [4, 6, 11, 12]. The most favourable results with conventional Bis-GMA resin cement were obtained when Y-TZP ceramic was tribochemically silica coated and silanized, but even after this conditioning method, bond strength of resin cements were not durable after thermal ageing [8]. According to the results of the present study, bond strength of the MDP-containing resin cements was also not stable after ageing conditions, and moreover, the bond strength results decreased dramatically resulting in 0 MPa.

Several studies have evaluated the adhesion durability between resin cements with and without MDP monomer in their composition to a Y-TZP ceramic [4, 6, 11, 12]. In these

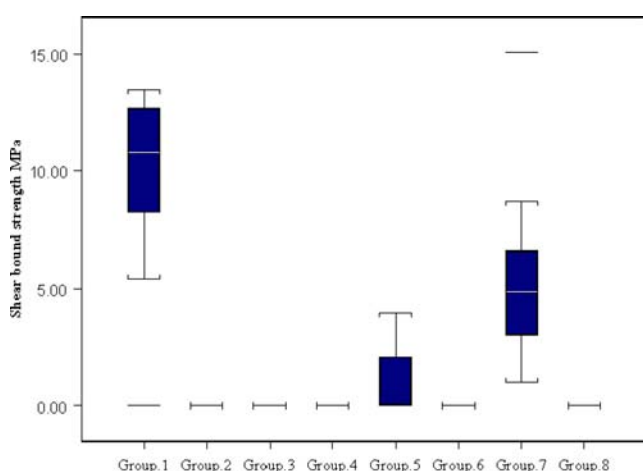
studies, the specimens were stored at  $37^\circ\text{C}$  for periods of 150 days, 2 years and 150 days followed by long-term thermocycling ( $\times 37,000$ ). It was demonstrated that only the MDP-based resin cement developed durable bond values to Y-TZP ceramic conditioned by airborne particle abrasion with  $\text{Al}_2\text{O}_3$  also according to the manufacturer's recommendations. Our results disagree with those obtained in these previous studies, as bond strength of both the MDP-based and Bis-GMA-based resin cements tested in this study showed dramatic decrease after ageing conditions. Similarly, Abo-Hamar et al. [1] also observed a significant decrease in bond strength after thermal ageing where adhesion of resin-based cements was tested on the enamel and dentin. That study, however, did not disclose the restorative materials such as ceramics. On the other hand, several studies stated that the adhesion results of MDP-based resin cements to the silica-coated and silanized glass-infiltrated zirconia ceramic was high and stable after long-term water storage and thermocycling [2, 3, 5, 8, 10]. Thus, despite utilization of acid-resistant ceramics in these studies, possibly, the presence of a glass phase in acid-resistant ceramics may have a contribution to the adhesion and may have a determining factor on the stability of durable resin adhesion. The infiltrated glass phase could optimize the silica impregnation; however, in Y-TZP ceramics, this seems not to be the case.

Another important issue that may explain the unstable adhesive durability between resin cements and Y-TZP ceramics is the poor chemical reaction between the silane-coupling agents (methylmethacryloxypropyltrimethoxysilane) and zirconium oxides present in the Y-TZP ceramic ( $96\% \text{ZrO}_2$ ) [7, 8]. It has been hypothesized that the chemical adhesion between aluminum oxides and silanes present a higher potential for hydrolytic degradation than the chemical adhesion between silicon oxide and silanes [7, 8]. It is evident that more studies need to be conducted to develop silane-coupling agents with better chemical affinity to zirconium oxides that are hydrolytically more stable.

## Conclusions

The conclusions of this study are as follows:

1. When instructions of the manufacturers were followed for the resin cements tested, no adhesion was achieved on the zirconia after 6,000 times thermal cycling.
2. Under dry conditions, Panavia F 2.0 revealed the highest mean bond strength; however, after thermocycling, it also resulted in 0 MPa as all other resin materials tested. Therefore, the hypothesis was accepted.
3. The use of zirconia cannot be recommended for FPDs where adhesive retention is required.



**Fig. 2** Means (MPa) and standard deviation of the shear bond strength data of Gr1 (Panavia F 2.0+Dry), Gr2 (Panavia F 2.0+TC) Gr3 (Multilink+Dry), Gr4 (Multilink+TC), Gr5 (Superbond+Dry), Gr6 (Superbond+TC), Gr7 (Quadrant Posterior Dense+Dry) and Gr8 (Quadrant Posterior Dense+TC)

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