



## Effect of Surface Treatment Protocols on Bonding of Resin Luting Agents to Zirconia Based Ceramics

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### Abstract

**Purpose:** Establishing a reliable adhesive bond to zirconia-based materials is always a challenge. The purpose of this study was to evaluate the micro-shear bond strength of conventional and self-adhesive resin cements to zirconium oxide ceramic after different surface treatments.

**Material and Methods:** Yttrium stabilized zirconia ceramic plates of dimensions 10 mm width × 10 mm length × 1 mm thickness were fabricated by a CAD/CAM process. The plates were divided into three groups according to surface treatments performed: (1) no treatment (NT); (2) airborne-particle abrasion with 110- $\mu$ m alumina particles (SB); (3) silica coating with Cojet system (CT) (3M/ESPE, USA). Each group was then divided into two subgroups according to type of resin cement; Panavia F 2.0 (Kuraray, Japan) and RelyXUnicem (3M/ESPE, USA). Ten composite resin cylinders (0.75 - mm diameter × 0.5 - mm height) were bonded to each ceramic plate (N = 10), and each specimen was subjected to a shear load at a crosshead speed of 0.5 mm/min until fracture occurred. The fracture sites were examined with scanning electron microscopy (SEM) to detect the mode of failure. Data were statistically analyzed using two-way ANOVA and multiple comparisons were made using Fisher's test at  $p < 0.05$ .

**Results:** Micro-shear bond strength was significantly affected by the surface treatment and by the type of resin cement. Panavia F 2.0 showed higher significant results in comparison to RelyXUnicem. Surface treatment with CT was highly significant with both cements, followed by SB and then by NT. SEM examination revealed predominantly cohesive failures within the resin cements for CT group, mixed failures within SB group and predominantly adhesive failure at the interfacial area within NT group.

**Conclusions:** The micro-shear bond strength of resin cement to partially stabilized zirconia ceramics varied significantly depending on the type of resin luting agent and surface treatment method. The tribochemical silica coating of zirconia surfaces in combination with MDP-containing resin cement (Panavia F 2.0) showed a superior performance.

**Keywords:** Protocols; Resin Luting; Zirconia; Ceramics

### Introduction

The use of high-strength zirconium oxide ceramics for oral rehabilitation has grown in recent years. It has become a material of choice for esthetic restorations, because of its unique properties and biocompatibility. CAD/CAM technology has simplified the fabrication of zirconia restorations. Additionally, adhesively bonded zirconia restorations, have recently surfaced as a conservative treatment option for minimally invasive approaches. They depend entirely on resin adhesive cementation for retention, marginal adaptation and resistance against masticatory loads [1-7].

Surface treatment is essential for bonding to ceramics. However, zirconia is resistant to hydrofluoric acid etching because of its crystallinity and the limited glassy phase (below 1%) [7-11]. As a consequence, other conditioning methods have been suggested. Airborne abrasion was reported by many studies to be an effective way of increasing the surface area and producing a degree of roughness that can lead to an acceptable resin/ceramic micromechanical interlocking [12,13].

One of the modern introductions of surface conditioning methods is silica coating by the Cojet system (3M/ESPE). In this tech-

nique, the surfaces are blasted with aluminium trioxide particles modified with silica. The impact pressure causes silica coated alumina particles to adhere on the ceramic surface, rendering it chemically more reactive to silane coupling agents. Less information is available regarding the effect of tribochemical silica coating in zirconia–resin bonds [14–18].

Cement selection is crucial for ensuring effective bond strength to zirconia. Luting agents containing phosphate monomers such as 10-MDP (Panavia, Kurary), and self-adhesive cements, have been proposed for bonding zirconia restorations [11,12,19,20]. These systems contain multifunctional acid methacrylates that could react with the substrate to achieve effective adhesion [21].

There are a variety of bond strength testing methods in the literature, and shear testing is one of the most popular ones. Shear stresses are believed to be the major factor involved in clinical failures of adhesive restorative interfaces [22–24]. In this study, bond strengths were assessed by a micro-shear bond which rely on testing a very small bonding area [25].

The aim of this study was to determine the bond strength of two families of resin cements; adhesive and self-adhesive dual-cure cements, to zirconia ceramic after different surface treatments protocols, namely airborne-particle abrasion and tribochemical silica coating.

## Materials and Methods

### Construction of ceramic specimens

Machinable yttrium partially stabilized zirconia core ceramic plates (In Coris ZI, Sirona, Germany) of dimensions 10 mm width × 10 mm length × 1 mm thickness, were fabricated following a CAD/CAM procedure using the CEREC InLab machine (Sirona, Germany). A mould was first made with the specified dimensions and then scanned. The design of the zirconia plates was then performed on the InLab 3D software and they were milled in the InLab machine from pre-sintered YZ blocks (In Coris ZI).

After milling, the zirconia plates were cleaned ultrasonically in distilled water and then sintered in a ceramic sintering furnace (InFire HTC, Sirona), for 7 hours at 1550°C. Fully sintered plates were inspected and measured to verify the dimensions. If necessary, they were adjusted with diamond stones at high speed and

water coolant, and the surfaces were smoothed with 1200 grit silicon carbide abrasives. All specimens were then placed in a ceramic furnace at 1000°C for a process of stress relief.

### Conditioning of ceramic specimens

The plates were then assigned to three groups according to the type of surface treatment:

- **Group NT:** No surface treatment applied.
- **Group SB:** Airborne particle abrasion with 110-µm aluminium oxide particles at 35 psi from a distance of approximately 10 mm for 15 seconds and cleaned with compressed oil-free air for 30 seconds.
- **Group CT:** CoJet system (3M/ESPE); Tribochemical silica coating: Initially, the surface was treated as in group SB and then subjected to airborne abrasion with 30-µm aluminum oxide particles modified with salicylic acid (CoJet-Sand, 3M/ESPE) for 10 seconds at right angle to the surface. Silane coupling agent (RelyX Ceramic Primer, 3M/ESPE) was then applied and left to dry for one minute.

### Preparation of resin specimens and bonding procedures

The materials used in the bonding procedures are described in table 1. For each group, the zirconia plates were further divided into two subgroups according to the type of resin cement used. Half of the specimens (N=10) were bonded to RelyX Unicem dual cure resin cement (3M/ESPE, USA), and the other half (N=10) to Panavia F 2.0 (Kuraray, Japan) dual cure resin cement.

The methodology developed by Shimada, *et al.* [25] was used to prepare specimens for the micro shear test. Surfaces of ceramic specimens were treated with bonding resin. Prior to light-curing of bonding resin, cylindrical plastic translucent molds with an internal diameter and a height of approximately 0.75 and 0.5 mm, respectively, were positioned over the treated surface of each ceramic plate. Bonding resin was then cured for 10 seconds. Following which, freshly mixed resin cement (Panavia F or RelyX Unicem according to grouping) was applied into the molds to fill their internal volume using a C-R syringe (Centrix Dental, Shelton, CT, USA). Light curing was performed for 40 seconds for each specimen. In this manner, very small cylinders of resin, approximately 0.75 mm in diameter and 0.5 mm in height were bonded to the ceramic surface at 3 to 4 locations.

Material	Type	Composition	Manufacturer
Panavia F	Dual polymerizing resin luting agent	Paste A: Silanated silica, microfiller, MDP (10-methacryloxydecyl dihydrogen phosphate, dimethacrylates, photo/chemical initiator  Paste B: Silanated barium glass, surface-treated NaF, dimethacrylates, chemical initiator	Kurary Medical, Inc Okayama, Japan
RelyX Unicem	Dual polymerizing resin luting agent	Base/catalyst Methacrylated phosphoric ester, dimethacrylate, inorganic fillers, fumed silica, chemical and photoinitiators	3M ESPE, USA

**Table 1:** List of the resin cements used in the study showing their composition.

The specimens were stored at room temperature (23°C) for 1 h prior to removal of the plastic tubing, then stored in water for 24h. Ten resin specimens were constructed for each group combination (N = 10). Before testing, all specimens were examined under an optical microscope at 25x magnification. Specimens showing any gaps, or any other defects were discarded.

#### Micro-shear bond strength evaluation

Each zirconia specimen was attached onto a testing device mounted in a universal testing machine (Lloyd, UK) for the micro-shear bond strength test. An orthodontic wire (0.2 mm in diameter) was placed around the resin cylinder, touching half the circumference and held flushed against the resin-zirconia interface. All the assembly of the bonding interface, the wire loop, and the center of the load cell were aligned in a straight line as possible to ensure the correct orientation of the shear force. Each cylinder was then subjected to a shear force at a crosshead speed of 0.5 mm/min until failure occurred. Interfacial shear strength was calculated by dividing the maximum load recorded on failure by the circular bonding area in square millimeters and expressed in MPa.

Data were statistically analyzed using two-way ANOVA and multiple comparisons were made using Fisher's test at  $p < 0.05$ . SPSS statistical software for windows version 20 (Chicago, IL, USA) was used for data analysis.

#### Morphological study using scanning electron microscope (SEM)

Following shear testing, all fractured interfaces were examined under SEM examination, to determine the mode of failure and observe the topographic changes.

They were recorded as:

- **Mode 1:** adhesive (between resin and zirconia),
- **Mode 2:** cohesive in adhesive layer,
- **Mode 3:** cohesive in resin or cohesive in zirconia,
- **Mode 4:** mixed failures (comprising two types).

#### Results

The mean shear bond strength and standard deviation values are shown in table 2 and figure 1. Two-way ANOVA indicated that the micro-shear bond strength was significantly affected by the surface treatment and by the type of resin cement evaluated at  $p < 0.05$ . Tukey's test showed that Panavia F 2.0 cement bonded to zirconia treated with the Cojet system showed the highest significant mean micro-shear bond strength value, and the lowest value was with RelyX Unicem with no surface treatment. Surface treatment with CT was highly significant with both cements, followed by SB and then by NT. With Panavia F 2.0 there was a high significant difference in micro-shear bond strength values between CT and both SB and NT groups, while there was a very small significant difference between SB and NT groups. With RelyX Unicem, there was a high significant difference between the 3 surface treatment groups. Tukey's test also showed that Panavia F 2.0 showed a high statistically significant difference in comparison with Rely X with all types of surface treatments.

SEM examination of the fractured interfaces showed variations among groups. Mode of failure analysis revealed predominantly cohesive failures (mode 2 and 3) within the resin cements within CT group, mixed failures (mode 4) within SB group and predominantly adhesive failure at the interfacial area (mode 1) within NT group.



**Figure 1:** Column chart showing the mean micro-shear bond strengths in MPa of the different treatment protocols with the two types of resin cements. (NT: no treatment. SB: sandblasting. CT: cojet treatment. P: Panavia. R: Rely X).

Resin Cement	NT Group			SB Group			CT Group		
	Mean	Sd	Sig	Mean	Sd	Sig	Mean	Sd	Sig
Panavia F 2.0	14.39	2.37	C	15.92	2.88	B	21.59	1.66	A
RelyXUnicem	8.81	1.14	E	11.67	1.17	D	14.58	1.28	C

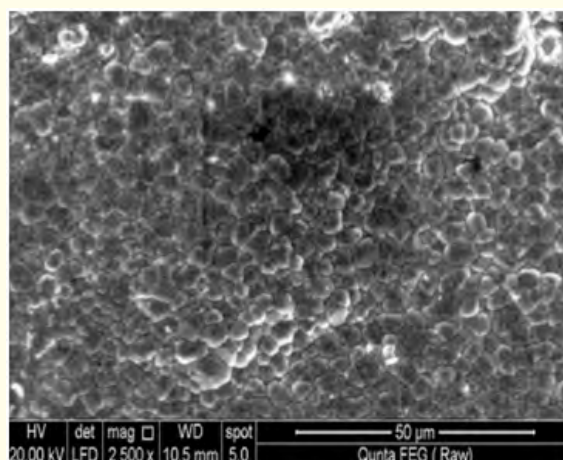
**Table 2:** Mean micro-shear bond strength in MPa of different surface treatment groups for tested resin cements, showing standard deviations and statistical significance.

Sig: Statistical significance. Values with different letters indicate significant difference.

SEM images of zirconia specimens are reported in figures 2-5. Specimens subjected to air abrasion showed a modified surface texture with prevalence of micro-retentive grooves. Zirconia surfaces treated with tribochemical silica coating featured only minor modifications in texture. Cement residuals are detectable on the zirconia surface in the CT and SB groups, while complete detachment from the ceramic surface occurred with the NT group

**Discussion**

Zirconium-oxide ceramics are famous in their ability to withstand high fracture loads [26], but this depends also on a reliable adhesive bond. Concerns still remain regarding the selection of the optimum luting method. The purpose of this investigation was to evaluate the influence of surface treatment methods and cement type on bonding to zirconia.



**Figure 2:** SEM of NT as-sintered zirconia surface.



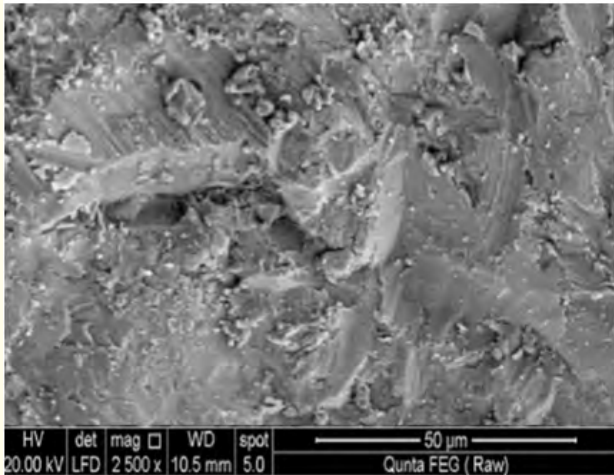


Figure 3: SEM of SB sandblasted zirconia surface.

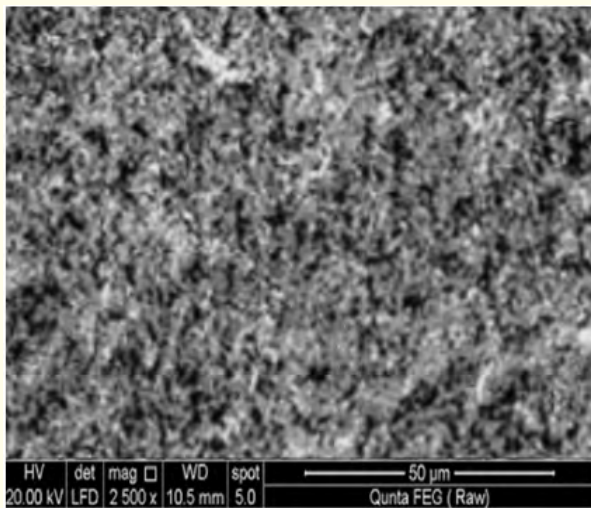


Figure 4: SEM of CT silica-coated zirconia surface.

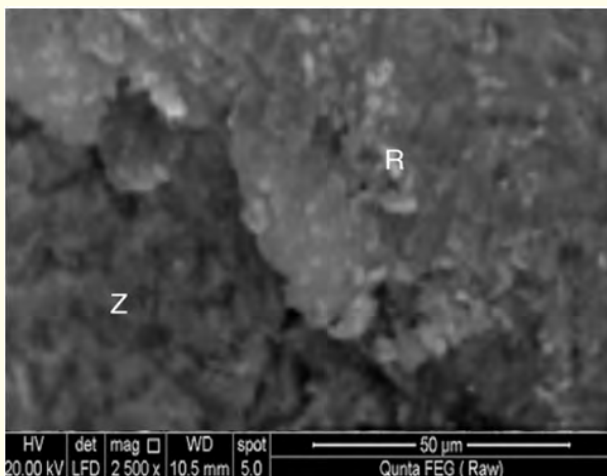


Figure 5: SEM of CT zirconia(Z) showing resin cement(R) covering the surface.

Machined zirconia surfaces are smooth and evenly flat with a regular crystal grain pattern. Untreated zirconium oxide ceramic is a relatively inert substrate with low surface energy and wettability [27,28], which explains the low micro-shear strength values associated with NT group. SEM analysis revealed a substantial increase in surface roughness after sandblasting. Airborne-particle abrasion with  $Al_2O_3$  is the preferred surface treatment method for high-strength ceramic materials [11,29-32]. Surface roughening methods increase surface energy and, therefore, wettability [29]. However, despite the increase in bond strength, application of airborne particle abrasion on such ceramics is controversial, due to the possible introduction of flaws and microcracks. The porosities created by that surface-treatment method may initiate cracks, which might affect the strength of ceramic materials. However, it has been shown that resin luting agents, have the ability to “heal” minor surface flaws created by sandblasting, therefore reinforcing ceramics [7,26]. Actually, this is more relevant with glass ceramics. With Y-ZTP ceramics, there is a particular concern about the effect of sandblasting on crystalline transformation of the tetragonal zirconia to the monoclinic form, producing transformation toughening at such an early stage. This might require a phase reversal procedure through heat treatment of zirconia to return to the tetragonal phase before clinical fixation. Nevertheless, as expected, in the present study, the application of airborne-particle abrasion resulted in a significant increase in micro-shear bond strength [33].

The Cojet system is composed of silica-modified  $Al_2O_3$  particles, which can create some surface roughness upon blasting. This tribochemical reaction produces a high temperature contact area and results in a silica coat which can later react with silane agents to promote resin bonding [34,35]. Microscopic analysis of the treated surface shows a thin microretentive layer [17,36], which enhances the bond strength to resin chemically and mechanically [29]. The silane coupling agent also has the ability of promoting a chemical bond to resins through cross-linking with methacrylate groups, and also increases the surface energy thus improving the wettability to resins [18,29,37]. This adhesive mechanism accounts for the high bond strength values observed for the CT-treated group, making for an interesting choice for conditioning poly-crystalline ceramics [38,39]. In this tribochemical procedure, the effect of particle size and application time need be further evaluated, as probably more aggressive treatments may increase micro-cracks formation, thus affecting the adhesive bond quality [32].

Comparing the effect of sandblasting of zirconia surface on the improvement of bond strength for both cements, it is obvious that it was more significant with RelyXUnicem. This is probably due to the fact that the molecular size of the MDP-containing cement (Panavia) could be larger than the micro-roughness produced by air abrasion of zirconia, in comparison to the low molecular size of Bis-GMA present in RelyXUnicem.

This study showed that the groups treated with Cojet system combined with the monomer-phosphate-based resin cement presented higher bond strength compared to specimens blasted with  $\text{Al}_2\text{O}_3$  particles. The results from the present study agree with those of Bottino, *et al.* [9] and Petrauskas, *et al.* [40] who found that the tribochemical method improved the bond strength of zirconia to an MDP-containing resin cement, compared to the use of airborne-particle abrasion alone. In contrast, another study found that airborne-particle abrasion of the ceramic combined with the use of a phosphate monomer resin cement, produced a durable bond, more favourable than those treated with silica coating and bonded with a Bis-GMA luting resin [11]. Therefore, it is safe to suggest that using a tribochemical system combined with monomer-phosphate-based resin cements is the best alternative for the cementation of zirconia ceramics [41].

Selection of the luting cement is a crucial factor for adhesive zirconia restorations. The ability of the acidic functional monomers, found in phosphate monomer-containing cements, of reacting with the substrate explains the high bond strength obtained with them. The adhesive potential of MDP to zirconia may depend on chemical reactions at the interfacial level involving the hydroxyl groups and the phosphate ester monomers [42,43]. Moreover, the presence of a long carbonyl chain accounts for the relative hydrolytic stability of the functional monomer, ensuring a relatively strong poly-molecular layer at the bonding interface [44]. However, the longevity of these adhesive interfaces needs to be further evaluated [3,45].

RelyX Unicem showed the capability of bonding the substrate, regardless of the ceramic surface treatment and without additional coupling agent application, despite the lower bond strengths values obtained compared to Panavia F cement. Bonding mechanism of RelyX Unicem is reminiscent of its self-adhesiveness and a possible improvement in bond strength may occur after cement maturation overtime [46].

Measurement of bond strength, regardless of the technique chosen, is a controversial topic in dental adhesion [47]. Conventional shear and tensile bond tests have generally been used to evaluate resin to ceramic bonding; however, the most commonly used shear bond test often produces fracture away from the adhesion zone. [48-52] Such failures of the substrate prevent measurement of interfacial bond strength and limit further improvements in bonding systems.

Several studies have identified nonuniform stress distributions along bonded interfaces [48-53]. The nonuniform interfacial stress distribution generated for conventional tensile and shear bond strength tests initiates fractures from flaws at the interface or in the substrate in areas of high stress concentration. Recently researchers have shown a preference in using the micro tensile method and fracture mechanics to understand the properties of the adhesive interface [54]. Unfortunately, the micro tensile bond test, although

an effective method in terms of testing a small area, is difficult to conduct and time-consuming for specimen preparation, especially in the case of ceramic specimens.

In this study, the micro-shear bond test technique was utilized. It is more practical to perform than the 'micro-tensile bond test', because trimming of the specimen after the bonding procedure is not necessary, and hence the bonding surface remains intact. In addition, preparing multiple specimens for this test, even using brittle materials, can easily be made. In the micro-shear test method, stress distribution is uniform because an ultra-small area of bonding interface is tested.

Many debates have focused on the clinical relevance of shear bond tests, because of the large variations of the results and the claimed uneven stress distribution in the tested interface [53,55]. Clinical conditions are usually difficult to duplicate in in-vitro testing. A wide variety of protocols are used for shear force application including wire loops, points and knife edges [22,24,25,56,57]. The use of a wire loop in particular, was reported to reduce the magnitude of stress concentration away from the interface [55]. On the other hand, in tensile bond testing, changes in elastic moduli of the tested materials result in interfacial stresses that are not uniformly tensile [58]. Shear bond testing was suggested by several investigators as a suitable screening mechanism for predicting clinical performance. Clearly different load application methods may lead to various stress distribution patterns. Thus, cautious interpretation of bond strengths values should always be considered, because of the possibility of uneven stress distributions [55].

## Conclusions

Within the limitations of this study, the following conclusions were drawn:

1. Micro-shear bond strength for zirconia ceramic differed significantly according to the type luting agent and surface treatment protocol.
2. The phosphate monomer-containing luting system (Panavia F 2.0) showed superior results making it the most suitable for use with adhesive zirconia restorations compared to the self-adhesive resin cement (RelyXUnicem). However, the durability of these adhesive bonding interfaces should be further evaluated.
3. Regarding the different surface conditioning methods, chairside tribochemical silica coating followed by silanization improved the bonding of zirconia ceramic significantly compared to aluminum oxide abrasion.

## Authors contribution

Prof. Salah contributed with the study design, experimental studies, and data acquisition. Dr. Nossair contributed with the liter-

ature search, and data analysis. Dr. Alaradi did the statistical analysis and manuscript preparation. All authors contributed in editing and reviewing the manuscript.

### Conflict of interest

The authors declare no conflict of interest.

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