The Effect of the Surface Treatment on the Adhesion of Resin Cements to Y-TZP

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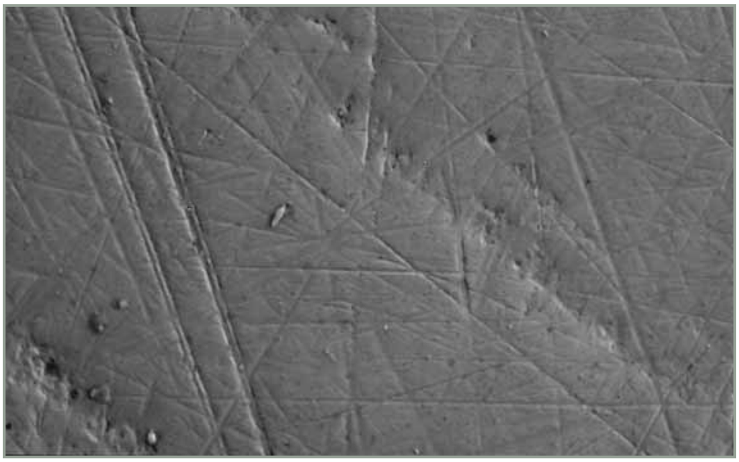

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Abstract

The purpose of the study was to evaluate the shear bond strengths (SBS) of two resin cements to intaglio surfaces of zirconia from two manufacturers after three surface treatment methods. Forty zirconia specimens from one manufacturer (Lava, 3M ESPE) and 40 zirconia specimens from another manufacturer (Cercon, Dentsply), each with system-specific intaglio surfaces, were randomly divided into four treatment groups (20 samples per group, 10 for each manufacturer): no treatment (No_T), sandblasting with 50 μm Al2O3 (S_50), sandblasting with 110 μm Al_2O_3 (S_110), and Rocatec silica coating and silanization (ESPE-Sil, 3M ESPE) (Roc). A 5 mm metal ring was fixed upon the zirconia and was filled with one of two different resin composite cements (RelyX Unicem, 3M ESPE and Panavia F 2.0, Kuraray). Samples were stored in distilled water at 37°C for 7 days; SBS was evaluated using a universal testing machine. Values were calculated in MPa and the data were analyzed by ANOVA and Tukey HSD test ($P < .05$). In the Lava specimens, the three surface treatment methods tested did not increase the bond strength of Panavia compared to the control group ($P = .195$); using RelyX Unicem, S_50 and Roc, mean SBS values were significantly higher than values for No_T (S_50 $P = .0048$; Roc $P < .001$). For Lava surfaces treated with Rocatec, there was a statistically significant difference between the two luting cements used (Unicem 11.39 ± 2.19; Panavia 8.56 ± 1.17; $P = .002$). Regarding the Cercon specimens, there was a significant difference between the Unicem and the Panavia control groups (No_T Unicem 1.48 ± 1.19, No_T Panavia 4.60 ± 2.75, $P = .004$). In conclusion, all surface treatments increased the bond strength of RelyX Unicem resin cement to both zirconia substrates. No statistically significant changes were found using Panavia on Lava. Sandblasting with 110 μm Al_2O_3 provided the highest bond strength for Panavia on Cercon.

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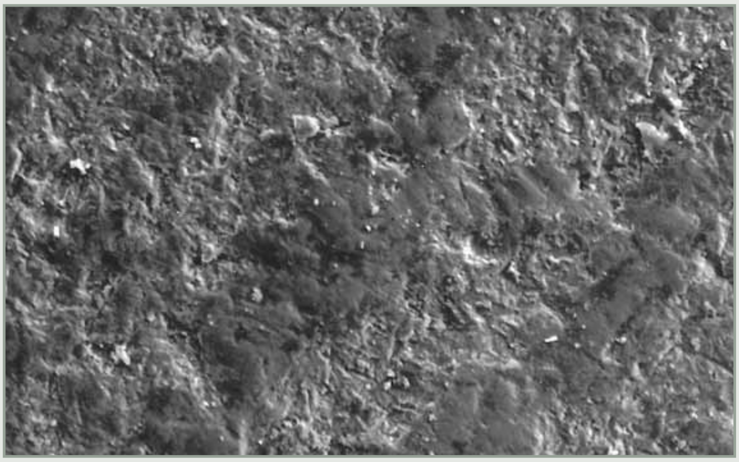

The increased desire for optimum esthetics in restorative treatments has made all-ceramic crowns a frequently used alternative for both anterior and posterior regions.[1] The use of all-ceramic restorations increased after the introduction of computer-aided design/computer-aided manufacture (CAD/CAM) systems. This technology enabled the dental laboratory to control the fabrication process and to produce reconstructions of high quality in a known production schedule. Moreover, automation of the fabrication steps and computer control of the milling system increased efficiency and reduced the technician's working time.[2]

Compared to conventional ceramics like glass-ceramic, zirconia exhibits higher material stability as a core material. It enables all-ceramic restorations to be performed using CAD/CAM technology due to its favorable optical properties and its high flexural strength of over 1,000 MPa.[3] In particular, research efforts appear to be more focused on zirconia-yttria ceramics, characterized by fine grained microstructures known as tetragonal zirconia polycrystals (TZP). Different studies support the clinical application of zirconia frameworks due to the acceptable marginal fit of these kinds of restorations.[4,5] However, many doubts remain about the cementation process.

Adherence to glass-ceramics normally is obtained by etching the ceramic with hydrofluoric acid. By dissolution of the glassy phase, the etching of the ceramic creates a rough surface, which favors adherence that relies on mechanical interlocking.[6] The zirconia surface, due to its chemical properties, can not be modified by hydrofluoric acid etching.[7] The use of silane coupling agents in enhancing the bond of resin composites to silica-based ceramics is well accepted in the dental literature.[8] Due to the silica-free chemical structure of zirconia, this kind of treatment does not affect adhesion to zirconia.[9] Therefore, zirconia frameworks require alternative surface conditioning methods to create a rough surface and to increase micro-mechanical retention and adhesion of resin bonding systems. A recent study showed that pre-treatment of zirconia ceramic surfaces with plasma spraying or sparsely fused glass pearls could increase the bond strength to composite resin cements.[10]

The aim of this study was to test the hypothesis that the bond strength of two resin luting agents, containing phosphate monomers, was modified by different zirconia surface conditioning methods.

Method and materials

Densely sintered high-purity zirconia ceramic (Y-TZP) samples from two different manufacturers (Lava, 3M Espe, St. Paul, MN, USA and Cercon, Dentsply International, York, PA, USA) were included in this study. Forty discs (15 mm in diameter and 2.5 mm in thickness) with intaglio surfaces were made out of zirconia blanks as provided by the two manufacturers. All samples were embedded in acrylic resin leaving one surface exposed. The specimens were randomly divided into four groups (20 discs per group, 10 per each manufacturer) and were subjected to four different surface treatments as follows:

- Group 1 (No_T): No treatment.
- Group 2 (S_50): Specimens were airborne-particle abraded with 50 μm Al_2O_3 particles (at 2.5 bar for 10 seconds at a distance of 10 mm), followed by ultrasonic cleaning in 96% isopropyl alcohol for 3 minutes.

Table 1 Chemical composition of the materials employed in this study.

LAVA 3M ESPE, St. Paul, MN, USA	Zirconium oxide 97%* Yttrium oxide 3%*
CERCON Dentsply International, York, PA, USA	Zirconium oxide 92%* Yttrium oxide 5%* Hafnium oxide 2%* Alumina and silica <1%*
Rely X Unicem 3M ESPE, St. Paul, MN, USA (Batch no. 200315)	Glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound, pigment, methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator
PANAVIA F 2.0 Kuraray Medical Inc, Okayama, Japan (Batch no. 41137)	A paste: MDP, Bis-phenol A, hydrophobic dimethacrylate, silanated silica, benzoyl peroxide, photo initiator B paste: Bis-phenol A, hydrophobic dimethacrylate, barium glass, titanium dioxide, treated sodium fluorite, sodium aromatic sulfinate, aromatic tert-amine

* Percent volume

- Group 3 (S_110): Specimens were airborne-particle abraded with 110 µm Al_2O_3 particles (at 2.5 bar for 10 seconds at a distance of 10 mm), followed by ultrasonic cleaning in 96% isopropyl alcohol for 3 minutes.
- Group 4 (Roc): Specimens were tribochemically silica coated with the Rocatec system (3M Espe, St. Paul, MN, USA). For this, they were first cleaned by sandblasting with 110 µm Al_2O_3 (Rocatec-Pre powder) at 2.5 bar pressure for 13 seconds at a distance of 10 mm. Subsequently, a silica layer was produced by sandblasting the samples with a special silica particle containing 110 µm Al_2O_3 (Rocatec-Plus) powder. The samples were ultrasonically cleaned in 96% isopropyl alcohol for 3 minutes after the silica sandblasting treatment. Then the silica-coated zirconia surfaces were silanated (Espe-Sil, 3M Espe, St. Paul, MN, USA) for 5 minutes.

Two different cements were used for bond strength testing: RelyX Unicem (3M Espe, St. Paul, MN, USA) [UNI] and Panavia F 2.0 (Kuraray Medical Inc, Okayama, Japan) [PAN]. The chemical formulations of the materials used in this study are listed in Table 1. A bonding area with a diameter of 5 mm was defined on the surface of each sample using a plastic disk. Metal rods (5 mm internal diameter, 2 mm thickness) were used to set the resin cement. The cements were applied following the manufacturers' instructions without the use of any adhesive system or primer solution. Excess material was removed using pellets. Both cements were light-cured for 40 s using a LED lamp (Freelight II, 3M Espe, St. Paul, MN, USA). The bonded specimens were stored in distilled water (37°C) for 1 week. After this storage period a shear bond strength (SBS) test was carried out at a cross-head speed of 1 mm/min in a universal testing machine (LR30K PLUS, LLOYD Instruments).

Furthermore, 8 additional disks (4 per manufacturer) were treated with the 4 treating methods described (1 sample per method) and examined in a scanning electron microscope (SEM) to determine the surface morphology.

Table 2 Detailed shear bond strength mean values and SD.

LAVA				
	No_T	**S_50**	**S_110**	**Roc**
Unicem	5.23 (1.91)	8.39 (2.42)	7.15 (2.97)	11.39 (2.19)
Panavia	6.92 (2.92)	7.07 (1.54)	8.28 (2.14)	8.56 (1.17)
Cercon				
	No_T	**S_50**	**S_110**	**Roc**
Unicem	1.48 (1.19)	4.68 (1.92)	6.49 (1.85)	6.96 (1.44)
Panavia	4.60 (2.75)	5.42 (1.57)	8.05 (3.00)	7.34 (2.97)

Mean values are expressed in MPa (Standard deviation).

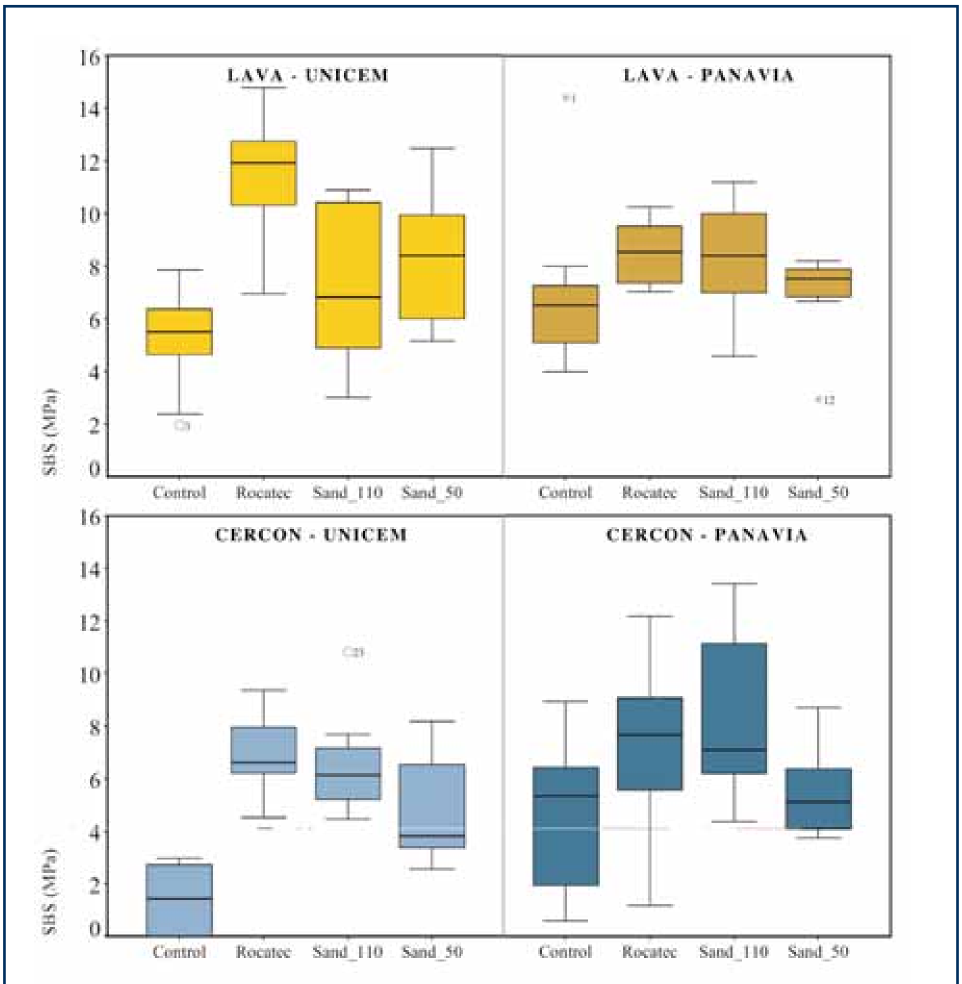

Fig 1 BoxPlot of the data. Every surface treatment increases bond strength values. Panavia F 2.0 obtains more uniform results than RelyX Unicem.

Table 3 Tukey-Kramer test to compare the different surface treatment.

LAVA			
Unicem		Panavia	
Treatment	Mean	Treatment	Mean
No_T	5.23 a	No_T	6.92 a
S_50	8.39 b	S_50	7.07 a
S_110	7.15 a,b	S_110	8.28 a
Roc	11.39 c	Roc	8.56 a
Cercon			
Unicem		Panavia	
Treatment	Mean	Treatment	Mean
No_T	1.48 a	No_T	4.60 a
S_50	4.68 b	S_50	5.42 a,b
S_110	6.46 b,c	S_110	8.05 b
Roc	6.96 c	Roc	7.34 a,b

Data connected by the same letter are not statistically significantly different.

Values were calculated in MPa and statistically analyzed by means of ANOVA and Tukey-Kramer HSD test ($P < .05$).

Results

Detailed shear bond strength values and standard deviations are listed in Table 2. The adhesion of the two cements (RelyX Unicem, Panavia F 2.0) on the two zirconia surfaces (Lava, Cercon) is demonstrated in Fig 1.

First, the effect of surface treatment on the bond strength of the two cements was analyzed (Table 3). Concerning Panavia F 2.0 for Cercon-zirconia, a statistically significant difference was found between S_110 surfaces and the control group (S_110 8.05 MPa, No_T 4.60 MPa, $P = .015$), while for Lava-zirconia Panavia F 2.0 obtained uniform results. Every surface treatment was found to significantly increase the bond strength to Cercon-zirconia surfaces when using RelyX Unicem (S_50 4.68 MPa, No_T 1.48 MPa, $P < .0001$; S_110 6.46 MPa, No_T 1.48 MPa, $P = .0003$; Roc 6.96 MPa, No_T 1.48 MPa, $P < .0001$), while on Lava-zirconia only S_50 and Roc surface treatments significantly increased bond strength values (S_50 8.39 MPa, No_T 5.23 MPa, $P = .0048$; Roc 11.39 MPa No_T 5.23 MPa $P < .0001$) (Table 3). Overall, Roc was the best surface treatment for RelyX Unicem, regardless of the zirconia material used.

Furthermore, the bond strength values of the cements on the two zirconia materials

Table 4 Tukey-Kramer test to compare the two cements used for each treatment.

LAVA			
No_T		**S_50**	
Unicem	5.23 a	Unicem	8.39 a
Panavia	6.92 a	Panavia	7.07 a
S_110		**Roc**	
Unicem	7.15 a	Unicem	11.39 a
Panavia	8.28 a	Panavia	8.56 b
Cercon			
No_T		**S_50**	
Unicem	1.48 a	Unicem	4.68 a
Panavia	4.60 b	Panavia	5.42 a
S_110		**Roc**	
Unicem	6.46 a	Unicem	6.96 a
Panavia	8.06 a	Panavia	7.34 a

Data connected by the same letter are not statistically significantly different.

were compared for each surface treatment (Table 4). Panavia F 2.0 exhibited higher bond strengths than RelyX Unicem on the control Cercon-zirconia specimens (Panavia 4.60 MPa, Unicem 1.48 MPa, $P=.004$). However, every surface treatment method increased bond strength values of the cements and led to similar bonding strength, when the two cements were compared. On Lava-zirconia, Roc pre-treatment significantly increased the bond strength of RelyX Unicem (Unicem 11.39 MPa, Panavia 8.56 MPa, $P=.002$). No differences were found between the cements with the other pre-treatment methods.

Finally, SEM comparison of the intaglio zirconia surface (control) and the three pre-treated surfaces demonstrated large differences in the surface morphology (Figs 2–9).

Discussion

The aim of this study was to evaluate the influence of the surface pre-treatment on the bond strength of resin luting agents to two different available zirconia core materials (Table 1).

Unlike conventional dental ceramic, polycrystalline material zirconia ceramics contains almost no glass. However, the composition of densely sintered ceramic blanks and their intaglio-surface configuration are specific for each commercially available material. The microstructure of Y-TZP ceramics depends on the processing conditions, like the sintering temperature and the type of surface finishing.[11,12] Therefore, conclusions drawn for one manufacturer's zirconia may not be applicable to the zirconia from other manufacturer.

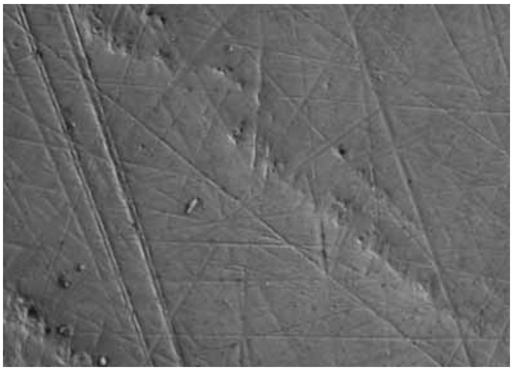

Fig 2 SEM photomicrograph of a Cercon specimen of the control group. The surface without any treatment shows a smooth morphology.

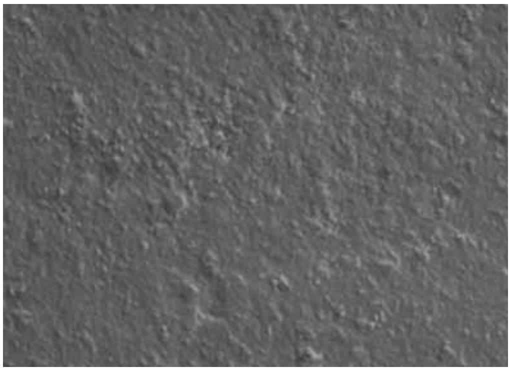

Fig 3 SEM photomicrograph of a Lava specimen of the control group. The intaglio surface of Lava samples shows a morphology less smooth than the Cercon surface.

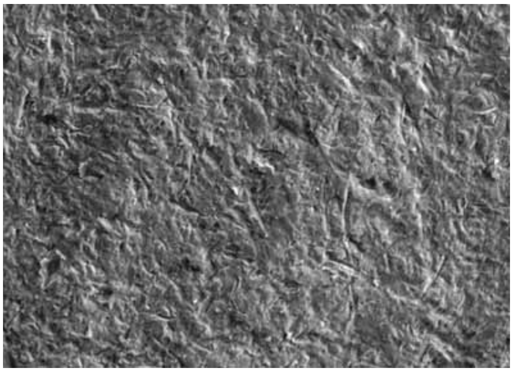

Fig 4 SEM photomicrograph of a Cercon specimen after sandblasting with 50 µm Al_2O_3. The roughness of the surface is suitable for the mechanical interlocking between zirconia and the cement.

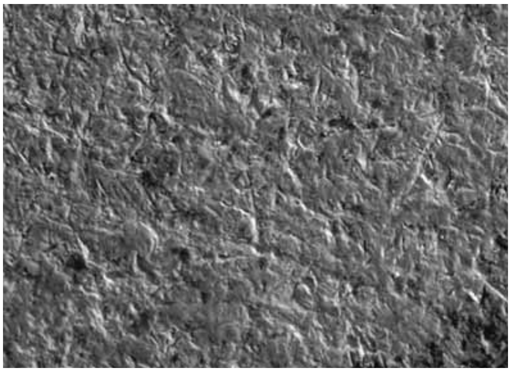

Fig 5 SEM photomicrograph of a Lava specimen after sandblasting with 50 µm Al_2O_3. Sandblasting treatment increases surface energy and, therefore, wettability of the surface.

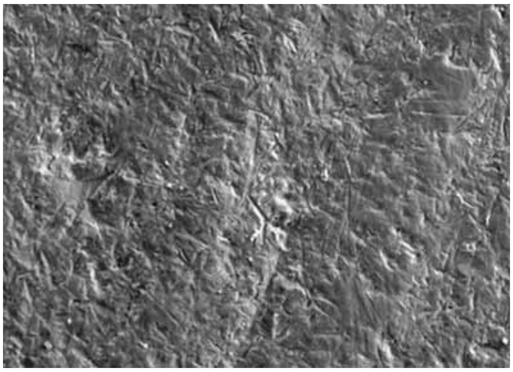

Fig 6 SEM photomicrograph of a Cercon specimen after sandblasting with 110 µm Al_2O_3. The pattern morphology is similar to the S_50 sample.

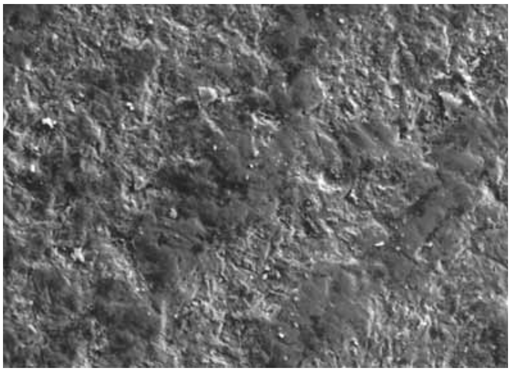

Fig 7 SEM photomicrograph of a Lava specimen after sandblasting with 110 µm Al_2O_3. The surface shows clusters of densely-sintered zirconia particles and deep undercuts.

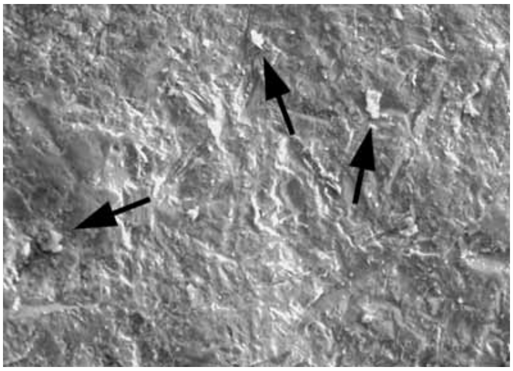

Fig 8 SEM photomicrograph of a Cercon specimen after Rocatec treatment. Some silica particles are incorporated on the surface of the sample.

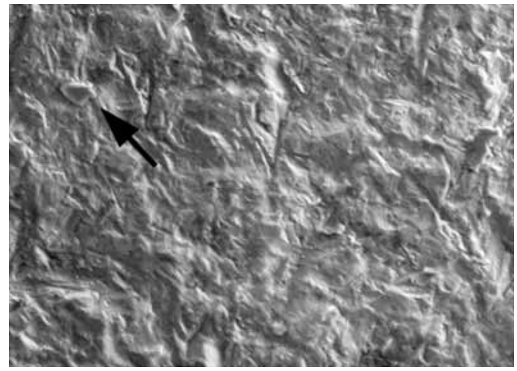

Fig 9 SEM photomicrograph of a Lava specimen after Rocatec treatment. The roughness of the surface is similar to the Cercon sample.

It has been suggested that even if the clinical success of a restoration does not rely on the resin bond to the tooth, successful resin bonding can improve retention,[13,14] marginal adaptation,[15] and fracture resistance of the restoration and the abutment tooth.[16] Based on the published evidence, it can be concluded that adhesive cementation procedures are necessary to support all-ceramic materials.[17]

It has been proposed to use a silane agent to increase the adhesion of resin cements to ceramics.[18] For conventional glass-ceramics like Dicor, silane bonding is mediated through the silica at the ceramic surface.[19–21]

Since Y-TZP does not contain silica, a traditional silane agent does not increase the resin bond strength to zirconia. On this kind of ceramic, a tribochemical silica coating system (Rocatec, 3M ESPE) is needed as coupling to allow fixation of a silane agent.[22]

Differences in composition and microstructure of the materials and the resulting surface morphology after the treatments are important factors in explaining the differences in bond strength of various ceramic materials. As dense highly crystalline ceramics like zirconia do not contain a glassy phase, the conventional acid etching technique does not affect the surfaces.[7] Therefore, mechanical roughening of the surface is required to increase the bond strength to zirconia.[23] It should also be taken into account, however, that air-abrasion may affect the ceramic surface by creating micro-cracks, which may reduce the fracture strength of a ceramic.[24,25] Moreover, the air-abrasion treatment can be an important degrading factor in long-term performance of all ceramic crowns.[26]

The airborne-particle abrasion with Al2O3 was the most appropriate method to increase roughness of high-strength ceramic materials.[27,28] Surface roughening methods increased surface energy and, therefore, its wettability.[9] As illustrated by the SEM analysis of the surfaces produced in this study, pre-treatment methods led to different surface morphologies. The untreated specimens (Figs 2–3) had a smooth surface. The airborne-particle abrasion with 50 and 110 µm (Figs 4–7), however, increased the roughness. Increased mechanical interlocking of the cements to roughened zirconia was expected, leading to higher bond strength values. The present study demonstrated that roughening of the zirconia surface increased the adhesion of resin cements. The bond strength values differed significantly between the control groups and the treated samples. Interestingly, the intaglio surface of the Lava sample was characterized by a rougher surface than that of Cercon. This may be the reason for the varying bond strength results obtained.

In this study, a shear bond test was used. The question remains whether tension tests are appropriate for evaluating the adhesive capabilities of resin agents to ceramic.[29] However, the present study did not set out to find absolute bond strength values, rather it evaluated whether the used pre-treatments showed significant differences in bond strength.

SEM photomicrographs of the samples treated with Rocatec demonstrated silica particles on the rough surface. In the present study, the adhesion of RelyX Unicem was positively influenced by the use of the Rocatec system, while silicatization did not affect the bonding strength of Panavia F 2.0.

The testing method used in this research did not include evaluation of long-term bond durability, as well as other relevant ageing parameters like water storage, thermal cycling, and dynamic loading. Further work is required to explore these issues.[25,30–32]

Two of the primary objectives of in vitro studies are elimination of extraneous parameters and limitation of the variables, but care should be taken whenever in vitro investigations are used to draw clinically relevant conclusions, especially because the use of materials of different composition and different manufacturers cannot be recommended without reservation and without further investigations. Both laboratory CAM procedures and clinical variables such as preparation, design, and three-dimensional geometry of the restoration may influence the clinical outcome of resin-bonded all-ceramic restorations.[33] Therefore, randomized clinical trials are needed to support the findings of in vitro studies.

Based on the present study, it can be concluded that mechanical roughness of the surface increased the bond strength of resin cements to zirconia. In addition, Panavia F 2.0 was less influenced by the surface treatment methods than RelyX Unicem.

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