



Can the Application of Multi-Mode Adhesive be a Substitute to Silicized/ Silanized Y-TZP Ceramics?

Arthur Magno Medeiros de Araújo¹, Ana Beatriz do Nascimento Januário¹, Dayanne Monielle Duarte Moura¹, João Paulo Mendes Tribst², Mutlu Özcan³, Rodrigo Othávio Assunção Souza¹

This study evaluated the effectiveness of a multi-mode adhesive (SBU-Scotch Bond Universal/3M) as a substitute for silica coating and silane application on the bonding of zirconia ceramics to resin cement. One-hundred and twenty sintered zirconia ceramic blocks (5 x 5 x 5 mm) were obtained, finished by grinding with silicon carbide paper (#600, #800, #1000 and #1200) and randomly divided into 12 groups (n=10) in accordance with the factors "surface treatment" (ScSi - silicization + silanization; ScSBU - silicization + SBU; SBU - SBU without photoactivation and SBUp - SBU photoactivated) and "ceramic" (Lava / 3M ESPE, Ceramill Zirconia / Amann Girrbach and Zirkozahn / Zirkozahn). Dual resin cement cylinders (RelyX Ultimate/3M ESPE) were subsequently produced in the center of each block using a silicon matrix (Ø=2 mm, h=5 mm) and photoactivated for 40 s (1200 mW/cm²). The samples were stored for 30 days in distilled water (37°C) and submitted to shear bond strength test (1 mm/min, 100 KgF). Data (MPa) were analyzed under ANOVA (2 levels) and Tukey test (5%). Complementary analyzes were also performed. ANOVA revealed that only the factor "surface treatment" was significant (p=0.0001). The ScSi treatment (14.28^A) promoted statistically higher bond strength values than the other ScSBU (9.03^B), SBU (8.47^B) and SBUp (7.82^B), which were similar to each other (Tukey). Failure analysis revealed that 100% of the failures were mixed. The silica coating followed by the silanization promoted higher bond strength values of resin cement and ceramic, regardless of the zirconia ceramic or SBU.

Introduction

Among the ceramic materials currently available for use in metal-free restorations, zirconia-based ceramics presents high flexural strength (1), biocompatibility of dental tissues and long-term chemical stability (2), allowing it to be applied in several clinical situations such as fixed prosthesis infrastructures (3), single crowns and veneers (4). Despite the excellent properties of zirconia ceramics, some studies have reported difficulties in adhesion to resin cements (5) because they have a high crystalline content in their composition, which makes hydrofluoric acid etching ineffective. For this reason, several alternative methods for the surface treatment of these ceramics have been proposed in the literature, such as: air-abrasion with alumina particles, silicization, application of silica-based primers (6), selective infiltration etching (7), laser irradiation (Nd:YAG, Er:YAG) (8), the application of low-melt glass (9), and plasma spray (9), among others.

Air-abrasion with aluminum oxide particles Al₂O₃ (50 µm) is widely used, as it increases surface roughness, thereby increasing mechanical retention and the ceramic/cement contact area. (9) However, some studies have reported that this procedure induces surface damage and weakens the

ceramic by about 25% after thermal fatigue, making it a controversial procedure (10). Regarding silicization, it corresponds to air-abrasion of aluminum oxide particles coated with silica, followed by an application of silane, thus promoting the deposition of silica on the surface of the ceramic which is chemically reactive to the silane, and increasing zirconia adhesion to resin cements (11). This technique has been evaluated by several studies (12) which were related to its effectiveness. Corroborating these studies, Özcan and Vallittu (2003) (12) investigated the effect of three types of surface treatments on ceramics and concluded that tribochemical silica coating considerably increased the bond strength between zirconia ceramics and resin cements when compared with alumina particles.

In addition to the air-abrasion technique, some authors (13) have proposed the use of metal primers containing functional acid monomers such as MDP (10-methacryloyloxydecyl dihydrogen phosphate), which promotes chemical interaction with zirconia, thereby decreasing clinical stages and increasing resin cement adhesion (14). A recent study observed that the association of superficial air-particle abrasion followed by the use of

¹Department of Dentistry, UFRN – Universidade Federal do Rio Grande do Norte, Natal, RN, Brazil

²Department of Dental Materials and Prosthodontics, Institute of Science and Technology, UNESP – Universidade Estadual Paulista, São José dos Campos, SP, Brazil

³Dental Materials Unit, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science University of Zurich, Center for Dental and Oral Medicine, Zurich, Switzerland

Correspondence: Rodrigo Othávio de Assunção e Souza, Avenida Salgado Filho, 1787, Lagoa Nova, 59056-000 Natal, RN. Tel: +55-84-3215-4104. e-mail: rodrigothavio@gmail.com

Key Words: bond strength, zirconia, surface treatment, silica coating, MDP primer.

metal primers was effective in increasing the bond strength between zirconia and resin cement (15). On the other hand, Pereira et al. (16) observed that the application of metal primer after ceramic air-abrasion does not necessarily imply in increasing adhesion between resin cement and zirconia.

MDP has also been incorporated to the universal adhesive systems/Multi-Mode, such as the Scotch Bond Universal (SBU). In addition to the MDP monomer, the SBU has other components such as having an adhesive system and silane in a single vial. A recent study (17) investigated the effect of different zirconia surface treatments such as silicatization and the use of a primer (SBU) on the adhesion to human dentin using conventional and self-adhesive resin cements. The authors concluded that both surface treatments significantly improved the zirconia-dentin interface adhesion. However, there is still limited information on how the combination of these surfaces can influence the adhesion and it is necessary to compare the performance of these universal adhesives as substitutes for other surface treatments.

In this perspective, the present study proposed to compare the effectiveness of a multi-mode adhesive (SBU) as a substitute for silica coating and silane application on bonding zirconia ceramics to resin cement. The tested hypotheses were: 1) the SBU application will promote bond strength values similar to silicatization, and 2) there will be no statically significant differences between the used ceramics.

Material and Methods

Sample Preparation

One hundred twenty (120) blocks of three zirconia-based ceramics [Lava, 3M ESPE, Irvine, CA, USA: (Zircon: 52% to 59%), Amann Girrbach GmbH/Durrenweg Germany: (Y2O3: 4,5-5,4%) and Zirkozahn GmbH/Gais, Italy: (Y2O3: 4-6%)] were cut with a low-speed diamond saw using water cooling into 7x7x7 mm blocks verified with the aid of a digital caliper (Eccofer, Curitiba, PR, Brazil). The block surfaces were ground flat with decreasing granulated SiC paper (#600, #800, #1000 and #1200 - 3M, St. Paul, MN, USA), so that all faces were flat. The blocks were subsequently sintered in a specific furnace of the respective ceramic systems according to the manufacturer's recommendations. Due to the approximation of zirconia particles during sintering, each ceramic sample showed final dimensions of approximately 5x5x5 mm³.

Embedded Blocks

Using a mold produced with the aid of industrial silicon for duplication, the one hundred and twenty blocks obtained after sintering were embedded in self-curing acrylic resin (JET; Artigos Odontológicos Clássico, São

Paulo, SP, Brazil). After resin polymerization, the surface of the ceramic blocks was wet-ground polished with silicon carbide papers of decreasing grit sequence (#600, #800, #1000 and #1200) in a Politriz machine (Labpol 8-12, Extec Corp., Enfield, CT, USA) until the acrylic resin and the zirconia air-blasted surface were at the same level. Next, the samples were randomly distributed into twelve groups according to the factors: "surface treatment (4 levels): 1- Scotch Bond Universal (SBU, 3M ESPE), 2- SBU photoactivated, 3- silicatization + silanization and 4- silicatization + SBU" and factor "ceramic (3 levels): 1- Lava, 3M ESPE, 2- Amann Girrbach and 3- Zirkozahn GmbH). * N= 150 / n=10.

Surface Treatment

Prior to the surface treatment procedures, all ceramic blocks were immersed in 10% isopropyl alcohol and ultrasonically cleaned for 5 minutes (Cristófoli Equipamentos de Biossegurança LTDA, Campo Mourão, PR, Brazil). The blocks were subsequently placed onto gauze, where they remained for 10 minutes to ensure complete alcohol evaporation.

For sandblasted groups (n=6), the silica coating (Cojet; Alumina particles coated with silica, 3M ESPE) was performed with a chairside air-abrasion device (Microjato Standard, Bioart, Brazil) for 20 s (2.5 bar pressure, distance: 10 mm, incidence angle: 90°). A metallic device was used to standardize the distance and incidence angle of the particles between the block surface and the tip of the chairside air-abrasion device. With the aid of a clinical clamp, circular movements on the base of the sample were performed in order to achieve a uniform blasted surface. Next, half of these sandblasted groups received a layer of silane (EspeSil, 3M ESPE) that was applied with a microbrush - ScSi (silicatization + silanization) surface treatment, and the other half received a layer of SBU adhesive that was applied with active friction using a microbrush for 20 s and dried until solvent evaporation - ScSBU (ScSBU - silicatization + SBU) surface treatment.

For non-coated groups submitted to the SBU (SBU without photoactivation) and SBU_p (SBU photoactivated) treatments, a layer of the SBU was applied as previously described. However, in the SBU_p condition, the adhesive was photoactivated for 40 s (1200 mW/cm²; Rádii Cal, SDI, Australia) after its application. All explained conditions were applied to each zirconia based ceramic.

Fabrication of Resin Cement Cylinders

A resin cement cylinder (RelyX Ultimate, 3M ESPE) was built on the cementation surface of the ceramic according to each group, following the methodology of Pereira et al. (16). Cylindrical silicone molds (Ø=3.5 mm, height: 4 mm)

were used to standardize the adhesive area and the height of the resin cement. These silicone molds were obtained from perpendicular sections (height: 4 mm), using a #11 scalpel blade, to the long axis of a tracheal tube (16). The molds were placed in the center of each zirconia block and fixed using dental wax heated. The center of the matrix corresponded with the center of the ceramic's cementation surface so that the entire layer of the cement stayed in contact with the ceramic.

After the matrix adaptation, the base paste and catalyst paste of the resin cement were manipulated with a metal spatula until total homogenization of the cement, and then the cement was inserted into the matrix with the aid of a Centrix syringe and photoactivated for 40s (1200 mW/cm²; Radium Cal). For each ceramic surface, only one resin cement cylinder was built-up. The cement was left for twelve hours to complete its chemical polymerization, and then the cylindrical polyethylene molds were removed with a #12 scalpel blade in a #3 scalpel cable.

Storage and Shear Bond Strength Test

All samples were stored in distilled water (37 °C) in closed containers for a period of 30 days in a bacteriological stove. After this period, they were submitted to a shear strength test (100KgF, 1 mm/min) in a Universal Testing Machine (Shimadzu, model Autograph AG-X/300 KN, Natal, Brazil).

Bond strength was calculated using the formula: $R=F/A$, where R = adhesive strength (MPa); F=force (N); A=interfacial area (mm). The force (F) corresponds to the value required for rupture of the specimen, and it is provided by the Universal Testing Machine software. The adhesive area of each ceramic block was defined by the area of a circle, and calculated by the following formula: $A=\pi r^2$, where $\pi=3.14$ and $r=1.75$ mm, where the radius (r) corresponds to half the diameter of the cylinder. Using this formula, the cross-sectional area was 9.06 mm².

X-ray Diffraction Analysis

In order to characterize the ceramics used in this study, X-ray diffraction analysis was performed in an X-ray diffractometer (Philips, PW 1830) in a sintered ceramic block from each studied brand in order to analyze the percentage of zirconia phases after ceramic sintering. Each sample was placed in a metal device (sample port), with the surface to be analyzed facing upwards, and then it was fixed onto the diffractometer reading compartment. After obtaining the acquisition data, they were sent to a computer unit and the data were analyzed using a computer program (Oring 8.0, Paso Robles, CA, USA). Quantification of the monoclinic phase (FM) volumetric fraction was calculated using the intensity of the monoclinic peaks (-111)M and (111)M and the tetragonal peak (101)T.

SEM/EDS

For SEM and EDS, the ceramic specimens were coated with gold particles (Inspect S50, FEI, Czech Republic) for 15 s to obtain a 90 Å thick layer. The surfaces were then observed at a magnification from 1000 to 10,000 times.

Wettability Test

Four blocks (5x5x5 mm) were used for the wettability evaluation. For this, samples were prepared and treated by air-abrasion Cojet® and SBU as described above. One extra sample of each ceramic was polished to be used as a control. Wettability was analyzed by the sessile drop technique using an optical tensiometer (TL 1000, Theta Lite Attention, Lichfield, Staffordshire, UK). For this, a syringe (#1001 Gastight Syringes – 1 mL, Hamilton, Reno, NV, USA) deposited one drop of distilled water onto the sample surface. After 5 s waiting for the drop to settle, a series of 60 images per second was recorded by the equipment for 20s. Oneattention (Biolin Scientific, Lichfield, Staffordshire, UK) software was used for calculating the mean contact angle values for each sample from the images obtained from 5 different areas of each sample.

Degree of Conversion

Nine specimens were produced for the degree of conversion analysis. For this, RelyX Ultimate cementation system was used in three different ways, as follows: 1) with adhesive system: base and catalyst pastes of resin cements were mixed with a portion of its adhesive system (SBU); 2) without adhesive system: base and catalyst pastes of resin cements were manipulated without the adhesive system; 3) Adhesive system: one drop of adhesive system (SBU).

Degree of conversion resin cement measurements were performed using Fourier Transform Infrared Spectroscopy - FTIR (Spectrum 65® PerkinElmer, São Paulo, SP, Brazil). Regarding specimen preparation, equal parts of the resin cement base and catalyst pastes were manipulated and placed on a glass cover. Thereafter, a polyester strip was superimposed on that portion of resin cement and pressure (750 mmHg) was applied. The cement layer was photoactivated using a LED (1200 mW/cm²) (Radium Cal) for 40 s. The resin cement specimens were stored in water medium for 24 h in black and totally opaque Eppendorf tubes in order to prevent light passage. Next, the specimens were submitted to the degree of conversion analysis. For the photoactivated SBU degree of conversion analysis, one drop of adhesive was placed on the crystal coupled to the spectrometer and photoactivated for 20 s. The degree of conversion was calculated by measuring the maximum absorption height of the peaks at 1638 and 1608 cm⁻¹. These values

were introduced in the equation:

$$GC(\%) = [1 - (R_{polim}/R_{nopolim})] \times 100,$$

where corresponds to the polymerized material and to non-polymerized material.

Analysis of Fractured Surfaces

The fractured specimens were examined under a stereomicroscopy (20×) (Stereo Discovery V20, Zeiss, Göttingen, Germany) and the failure modes were classified with the following scores: A) Adhesive failure at the ceramic/cement interface; B) Cohesive failure in the ceramic; C) Cohesive failure in the cement, and D) Mixed failure (Ceramic/cement interface adhesive failure + Cohesive failure of cement). One representative sample of each group was randomly selected and was used for analysis using a scanning electron microscope (SEM) (Inspect S50, FEI, Czech Republic).

Statistical Analysis

Statistical analysis was performed using 2-way ANOVA and Tukey's test ($\alpha=5\%$) to compare the experimental groups with each other. In order to perform these tests, the obtained data were submitted to statistical analysis through the computer program STATISTIX (Analytical Software Inc., version 8.0, 2003). The degree of conversion and the wettability test results were then qualitatively analyzed. The sample power was calculated using the OpenEpi website (www.openepi.com).

Results

The sample power was calculated based in the OpenEpi website. Considering a 95% two-tailed confidence interval, a sample power of 91.3% was obtained.

Shear Strength and Analysis of Fractured Surfaces

The "surface treatment" ($p=0.0001$) factor presented statistical significance, while the "ceramic" factor ($p=0.7505$) was not statistically significant. Tukey's test ($p=0.05$) showed that when only the "surface treatment" factor was considered, silica coating

followed by silanization (ScSi) - (14.28)^A - promoted statistically higher bond strength values than the other surface treatments, which were similar to each other: ScSBU (9.03)^B, SBU (8.47)^B, SBU_p (7.82)^B. When only the "ceramic" factor was considered, mean bond strength values were: Lava (10.17)^A, Amann (9.60)^A and Zirkonzahn (9.94)^A.

When comparing the experimental groups with each other, it was observed that the coated groups followed by the silane application, regardless of the ceramic type used, showed the highest bond strength values: LavaScSi (14.76)^A, AmannScSi (14.64)^A, ZirkonScSi (13.45)^{AB}. On the other hand, the groups AmannSBU (8.36)^C, AmannSBU_p (7.86)^C, AmannScSBU (7.52)^C and ZirkonSBU_p (6.85)^C had the lowest bond strength values (Tukey test) (Table 1).

X-ray Diffraction

The X-ray diffraction analysis showed an absence of monoclinic phase and 100% tetragonal phase in three of the zirconia ceramic brands studied, which presented similar graphical behavior when considering the existence of tetragonal phase after the sintering process (Figure 1).

EDS Analysis

EDS analysis quantified the chemical component percentages of each tested zirconia ceramic, all of which showed similar chemical composition for the constituent chemical elements, corresponding to oxygen (O₂), zirconium (Zr) and carbon (C): Lava (Zr=74.13%; O₂=21.52% and C=4.35%), Zirkonzahn (Zr=73.84%; O₂=21.07% and C=5.10%) and Amann (Zr=71.55%; O₂=21.81% and C=6.64%).

Wettability

According to the wettability analysis results, it was observed that the ceramic surface treatment with silica coating showed the smallest contact angle (44.05°) between the ceramic surface and distilled water drop (the substance used as measurement), followed by the samples treated with SBU without photoactivation (70.05°), and finally, samples treated with photoactivated SBU showed the smallest average wettability (72.17°). The sample used for control without surface treatment presented average wettability of 35.45°.

Degree of Conversion

The results showed that higher degree of conversion values were obtained when RelyX Ultimate + SBU (82.27% ± 1.7) were associated. The SBU (77.78% ± 1.3) showed an approximated degree of conversion, however, it was still lower. The resin cement without the presence of the adhesive system showed a lower polymer conversion

Table 1. Mean (±SD) shear bond strength values (MPa) of ceramic systems after the treatment surfaces and their statistical differences

Group (n=10)/Treatment	Amann	Lava	Zirkonzahn
S _c +S _{il} (silicatization + silanization)	14.6 (3.57) ^A	14.7 (2.29) ^A	13.4 (2.16) ^{AB}
S _c +SBU (silicatization + SBU)	7.5 (4.00) ^C	8.7 (2.00) ^{BC}	10.8 (3.64) ^{ABC}
SBU (SBU without photoactivation)	8.3 (3.48) ^C	8.4 (4.90) ^{BC}	8.6 (3.26) ^{BC}
SBU _p (SBU photoactivated)	7.8 (2.66) ^C	8.7 (3.59) ^{BC}	6.8 (3.77) ^C

Different letters indicate statistically significant difference.

(61.38% \pm 5.7).

Failure Analysis

The failure analysis showed that there was 100% of mixed failures for all the studied groups, regardless of the ceramic type or surface treatment performed, with these being cement cohesion failures.

Discussion

The surface treatment performed on crystalline ceramics, especially zirconia, has been the subject of several scientific studies, as this procedure is directly related to the clinical success of ceramic restorations (18). Therefore, this study proposed to evaluate different strategies for surface treatment of zirconia ceramics in bond strength to a dual resin cement.

Several tests can be used in order to verify the bond strength of the ceramic-cement interfaces, such as shear, micro-shear, tensile and micro-tensile. Although the micro-tensile test promotes a more uniform distribution of stresses along the interface, the zirconia section procedures for the micro-tensile test makes it difficult to obtain the specimens because of the high strength and hardness of this ceramic material (19). In addition, the sectioning

procedures of these tests can induce premature interface failures, decreasing their reliability (20). In this study, the samples were submitted to shear testing. Several studies have also used this test (20) to evaluate the bond strength between the adhesive ceramic interface of zirconia-resin cement. In addition to the ease of execution and low cost (21) of SBS tests performed with zirconia ceramics, problems related to cohesive failure of the base material can be avoided, since the adhesion of the zirconia/cement interface is not high. Thus, the shear test was selected for use in this study.

Adhesion between ceramic and resin cement is also influenced by the thermal, chemical and mechanical conditions generated by the oral environment (8). Water storage and thermocycling are often used to simulate the aging of the interfaces in vitro (8). Water storage is a valuable method to evaluate the bond strength between the cement-ceramic interface by exposing the adhesive interface to the water, allowing the hydrolytic degradation (14). Immediate aging (24 h) is widely used, however, according to Ozcan and Vallittu (11), resinous materials absorb water to some extent during storage in water, requiring days or weeks to reach maximum absorption. In our study, storage in distilled water at a constant

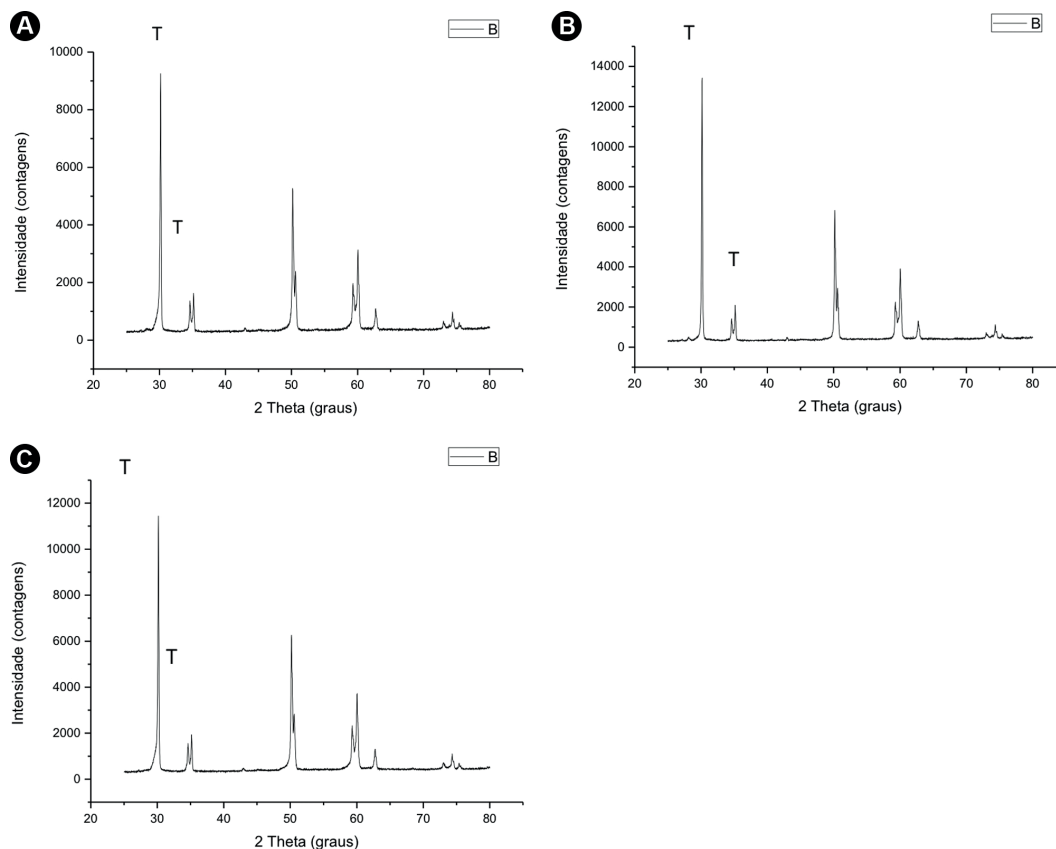


Figure 1. Graphic presentation of the X-ray diffraction performed in a sample showing characteristic peaks of tetragonal zirconia (AMANN) after the sintering processes: A: Amann. B: Zirkonzahn. C: Lava.

temperature of 37 °C for a period of 30 days was used. Several studies that used the same aging protocol (23) observed a significant decrease in bond strength between the ceramic-cement interface, proving that this time is sufficient to promote a degradation of this interface.

According to the results of this study, the hypothesis that the SBU promotes adhesion values similar to silica coating was rejected. The silica coating in this study promoted significantly higher values of bond strength than the other groups for the three types of tested ceramics. Supporting these findings, several studies (11,22) have proven the effectiveness of silica coating, since this procedure promotes silica deposition on the zirconia surface, and chemical bonding of the resin cement to the ceramic is made possible by applying a silane coupling agent. Several authors have reported that air-abrasion procedures may also increase wettability by reducing the surface energy of the ceramic, thereby increasing adhesion to resin cements (18). In our study, greater wettability was found when ceramics were submitted to the silica coating protocol. The impact of silica coated particles results in a layer of silica forming on the zirconia surface, improving silanization and adhesive cementation (16). High wettability can allow greater interaction of adhesive and resinous components (16), which may explain the satisfactory results obtained from the silica coating of the studied groups.

Regarding the use of SBU in our study, lower bond strength values were found when compared to the silica coating followed by silanization regardless of the surface treatment, the photopolymerization or ceramic type used. Xie et al. (22) also compared silicatization using three metal primers (Z-Prime Plus, ScotchBond Universal and Clearfil Universal) and concluded that the silica coating followed by the silane application presented higher shear strength than all the other groups, except for Z-primer Plus. The authors reported that while universal adhesive manufacturers have made progress in combining all components in a single vial, the bond of resin cements to Y-TZP still depends on the action of the acid phosphate monomers within the product. In addition, it has been found that there is less hydrolytic stability between the MDP-zirconia chemical bonds than the silica and silane chemical bonds, thereby reducing adhesion.

Despite the lower performance of the SBU observed in our study, some authors have reported that it promotes satisfactory adhesion of resin cements to zirconia, even when used without additional surface treatments (4,5). In comparing only the SBU groups, it was possible to identify that there were no significant differences between the groups that were silicatized followed by SBU, and those that used only SBU; i.e. the silica coating associated with the primer did not significantly contribute to adhesive

resistance. Similar results were also reported by Pereira et al. (16), in which the authors evaluated the influence of universal primers (including SBU used alone or in combination with air-particle abrasion) on the adhesion of resin cements to zirconia ceramics, and concluded that the use of SBU without additional surface treatments improves adhesion. This fact can be explained by the presence of MDP in the SBU composition. The MDP molecules have chemical interaction with the zirconia surface, thus increasing bond strength between zirconia and resin cements (23,24).

Regarding the effects of SBU photopolymerization prior to adhesive cementation, the groups that were photopolymerized prior to cement obtained the same bond strength results as those that were photopolymerized with the cement. Even though studies on the effect of SBU photoactivation on bond strength between zirconia ceramics and resin cements are not available in the literature and considering the degree of conversion values of these materials, higher degree of conversion values were found in our study when resin cements were mixed with a portion of adhesive system (SBU), suggesting that the photoactivation of the combination (cement + adhesive) is also effective (as recommended by the manufacturer), which eliminates the need for prior SBU photopolymerization in clinical procedures, and supporting the statically similar results between the SBU photoactivated groups and those that were not. However, more studies are needed to support this information.

The second hypothesis that there would be no statically significant differences between the used ceramics was accepted. In the present study, the three systems (Lava/3M ESPE, Ceramill Zirconia/Amann, Grrrbach and Zirkozahn/Zirkozahn) showed similar chemical composition and adhesive behavior in multiple qualitative analyses. According to the X-ray (DRX) diffraction analysis, the tetragonal phase percentage after ceramic sintering was similar for all ceramic systems, as well as the absence of the monoclinic phase. EDS analysis also confirmed similarities in the chemical composition among the studied ceramics, showing equal percentages of chemical elements of zirconia, oxygen and carbon. The failure analysis in a stereomicroscope (20×) showed 100% mixed failures (adhesive + cohesive failures in the cement). Similar results were also reported by Otani et al. (19,25), who observed a major occurrence of mixed failures in their study, where zirconia samples cemented to cylinders of resin cement were submitted to macro-shear strength test after a silica-coating surface treatment followed by silanization. According to the authors, mixed failures are easily detected in samples having zirconia as base material and some type of resin cement as bonding material. According to Seabra et al. (25), the presence of mixed failures (predominantly

cohesive failures in the cement) are associated to satisfactory adhesion values between zirconia ceramic and resin cement. Moreover, these failures may also have been mainly influenced by the non-uniform distribution of the stresses generated from the shear test which are directed to the base material instead of the adhesive interface (19).

In order to validate the outcomes of this study, randomized clinical trials must be performed in order to ascertain long-term bond strength between zirconia ceramic and resin cement according to different zirconia surface treatments. Thus, based in the results, it can be concluded that silicization promoted significantly better bond strength than SBU, regardless of the surface treatment, photopolymerization and ceramic type used. Moreover, light-curing of SBU prior to cement application did not influence the zirconia bond strength to resin cement.

Acknowledgements

The authors give thanks to the 3M ESPE company for supplying materials that made this research feasible, and to the National Institute of Spatial Research (INPE) for their support in the X-ray diffraction test performance.

Resumo

Este estudo avaliou a efetividade de um adesivo "multi-mode" (Single Bond Universal/3M) como um substituto para a silicização e aplicação do silano na resistência de união das cerâmicas de zircônia e um cimento resinoso. Para isso, 120 blocos cerâmicos sinterizados de zircônia nas dimensões de (5 x 5 x 5 mm) foram obtidos, lixados com lixas de granulação decrescente (#600, #800, #1000 e #1200) e divididos aleatoriamente em 12 grupos (n = 10), de acordo com os fatores "tratamento de superfície" (ScSi - silicização + silanização; ScSBU - silicização + Single Bond; SBU - SBU sem fotoativação e SBUp - SBU com fotoativação) e "cerâmica" (Lava/3M ESPE, Ceramill Zircônia/ Amann Girrbach e Zirkozahn/ Zirkozahn). Posteriormente, cilindros de cimento resinoso dual (RelyX Ultimate/3M ESPE) foram confeccionados no centro de cada bloco com auxílio de uma matriz de silicone (Ø=2 mm; h=5 mm) e fotopolimerizados por 40 s (1200 mW/ cm²). Em seguida, as amostras foram armazenadas durante trinta dias em água destilada (37 °C) e submetidas ao ensaio de resistência de união ao cisalhamento (1 mm/min, 100 kgf). Os dados (MPa) foram analisados sob ANOVA (2 fatores) e teste de Tukey (5%). Análises complementares também foram realizadas. ANOVA revelou que apenas o fator "tratamento de superfície" foi significativo (p=0,0001). O tratamento ScSi (14.28^A) promoveu valores de adesão estatisticamente superiores aos demais ScSBU (9.03^B), SBU (8.47^B) e SBUp (7.82^B), os quais foram semelhantes entre si (Tukey). A Análise de falhas revelou que 100% da falhas que ocorreram foram mistas. A silicização seguida da silanização promoveu a melhor resistência de união entre cimento resinoso e a cerâmica, independentemente do tipo da cerâmica ou do SBU.

References

- Tinschert J, Zvez D, Marx R, Anusavice KJ. Structural reliability of alumina-, feldspar, leucite-, mica- and zirconia-based ceramics. *J Dent* 2000; 28:529-535.
- Denry I, Kelly JR. Emerging ceramic-based materials for dentistry. *J Dent Res* 2014; 93:1235-1242.
- Özcan M, Valittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. *Dent Mater* 2003;19:725-731.
- Alghazzawi TF, Lemons J, Liu P-R, Essig ME, Janowski GM. The failure load of CAD/CAM generated zirconia and glass-ceramic laminate veneers with different preparation designs. *J Prosthet Dent* 2012;108:386-393.
- Amaral R, Özcan M, Bottino MA, Valandro LF. Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning. *Dent Mater* 2006;22:283-290.
- Yun J, Ha S, Lee J, Kim S. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. *Dent Mater* 2010;26:650-658.
- Aboushelib MN. Evaluation of zirconia/resin bond strength and interface quality using a new technique. *Journal of Adhesive Dentistry* 2011;13:255-260.
- Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Bond strength of resin cements to a zirconia ceramic with different surface treatments. *Oper Dent* 2009;34:280-287.
- Derand T, Molin M, Kvam K. Bond strength of composite luting cement to zirconia ceramic surfaces. *Dent Mater* 2005;21:1158-1162.
- Özcan M, Kerkdijk S, Valandro LF. Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only. *Clin Oral Investig* 2007;13:279-282.
- Valandro LF, Della Bona A, Bottino MA, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. *J Prosthet Dent* 2005; 93:253-259.
- Özcan M, Valittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. *Dent Mater* 2003;19:725-731.
- Özcan M, Cura C, Valandro LF. Early Bond strength of two resin cements to Y-TZP ceramic using MPS or MPS/4-META silanes. *Odontology* 2011;99:62-67.
- Dantas DC, Ribeiro AI, Lima LH, de Lima MG, Guênes GM, Braz AK, et al. Influence of water storage time on the bond strength of etch-and-rinse and self-etching adhesive systems. *Braz Dent J* 2008;19:219-223.
- Amaral M, Belli R, Cesar PF, Valandro LF, Petschelt A, Lohbauer U. The potential of novel primers and universal adhesives to bond to zirconia. *Am J dent* 2014;42:90-98.
- Pereira LL, Campos F, Dal Piva AMO, Gondim LD, Souza ROA, Özcan M. Can application of universal primers alone be a substitute for airborne-particle abrasion to improve adhesion of resin cement to zirconia? *J Adhes Dent* 2015;17:169-174.
- Alves MLL, Campos F, Bergoli CD, Bottino MA, Özcan M, Souza ROA. Effect of adhesive cementation strategies on the bonding of Y-TZP to Human Dentin. *Oper Dent* 2016;41:276-283
- Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: A systematic review and meta-analysis. *J Adhes Dent* 2015;17:7-26.
- Otani A, Amaral M, May LG, Cesar PF, Valandro LF. A critical evaluation of bond strength tests for the assessment of bonding to Y-TZP. *Dent Mater* 2015; 31:648-56.
- Tanis MÇ, Akçay C. Effects of different surface treatments methods and MDP monomer on resin cementation of zirconia ceramics an in vitro study. *J Lasers Med Sci* 2015; 6:174-81.
- Bottino MA, Bertoli C, Lima EG, Marocho SMS, Souza RO, Valandro LF. Bonding of Y-TZP to dentin: effects of Y-TZP surface conditioning, resin cement type, and aging. *Oper Dent* 2014;39:291-300.
- Xie H, Li Q, Zhang F, Lu Y, Tay FR, Quian M, Chen C. Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better? *Dent Mater* 2015;32:403-411.
- Valandro LF, Ozcan M, Amaral R, Leite FP, Bottino MA. Microtensile bond strength of a resin cement to silica-coated and silanized In-Ceram zirconia before and after aging. *Int J Prosthodont* 2007;20:70-72.
- Cheung GJ, George JK, Botelho MG. Zirconia surface treatments for resin bonding. *J Adhes Dent* 2015;17:551-558.
- Seabra B, Arantes-Oliveira S, Portugal J. Influence of multimode universal adhesives and zirconia primer application techniques on zirconia repair. *J Prosthet Dent* 2014;112:182-187.

Received December 12, 2017

Accepted May 18, 2018