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Evaluation of shear bond strength and shear stress on zirconia reinforced lithium silicate and high translucency zirconia.

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Abstract: This study evaluated the shear stress distribution on the adhesive interface and the bond strength between resin cement and two ceramics. For finite element analysis (FEA), a tridimensional model was made using computer-aided design software. This model consisted of a ceramic slice (10x10x2mm) partially embedded on acrylic resin with a resin cement cylinder (Ø=3.4mm and h=3mm) cemented on the external surface. Results of maximum principal stress and maximum principal shear were obtained to evaluate the stress generated on the ceramic and the cylinder surfaces. In order to reproduce the *in vitro* test, similar samples to the computational model were manufactured according to ceramic material (Zirconia reinforced lithium silicate - ZLS and high translucency Zirconia - YZHT), (N=48, n=12). Half of the specimens were submitted to shear bond test after 24h using a universal testing machine (0.5mm/min, 50kgf) until fracture. The other half was stored (a) (180 days, water, 37ºC) *prior* to the test. Bond strength was calculated in MPa and submitted to analysis of variance. The results showed that ceramic material influenced bond strength mean values (p=0.002), while aging did not: YZHT (19.80±6.44)[a], YZHTa (17.95±7.21)[a], ZLS (11.88±5.40)[b], ZLSa (11.76±3.32)[b]. FEA results showed tensile and shear stress on ceramic and cylinder surfaces with more intensity on their periphery. Although the stress distribution was similar for both conditions, YZHT showed higher bond strength values; however, both materials seemed to promote durable bond strength.

Keywords: Finite elements analysis; ceramics; indirect restoration; shear bond strength; Zirconia.

INTRODUCTION.

The long-term success of ceramic restorations is directly dependent on a suitable cementation technique capable of establishing durable bond strength between restoration and substrate. Total ceramic restorations depend on adhesive cementation with resin cements to achieve a good clinical prognosis.[1] In addition to providing adequate adhesiveness, resin cements may prevent oral biofilm formation at the adhesive interface,[2] as well as promoting fracture resistance of restorations, including zirconia.[3] However, even if a silane agent is indicated for ceramic restorations, there are generally several materials that can be used by the clinician and each one has different properties and characteristics that may influence its interaction with the resin cement.[4]

Of all ceramic materials used in dentistry, yttria partially stabilized

zirconia (Y-TZP) ceramic is the most resistant and has the best mechanical properties.[4] Recently, its optical properties have been optimized, and the possibility of increasingly conservative preparations have enabled the use of this material in monolithic form.[5] Among the indirect materials available for the preparation of monolithic restorations, Y-TZP with high translucency stands out. This ceramic was recently introduced in the market and associates the traditional zirconia mechanical resistance with high translucency.[6] Despite this, few investigations can be found on the adhesive strength of this material with the bonding agent. Zirconia can also be found inside glass ceramics as a mechanical reinforcing material, such as observed in zirconium reinforced lithium silicate (ZLS). However, the literature is not conclusive about the adhesive or mechanical benefit of incorporating zirconia inside this material.[7,8]

The shear, microshear, tensile and microtensile tests are available to evaluate adhesive interfaces.[9,10,11] Despite the limitations of the mechanical shear test, the possibility of standardization, facility of specimen preparation and suitability to laboratory equipment[12] make this a widely-used method to evaluate adhesive resistance of dental materials.[13] In addition to the *in vitro* test, associating an analysis of the generated stress in the interfaces through an in silico (computational) test using finite element method can contribute to understanding the results.[14]

Thus, the aim of this paper was to analyze the shear bond strength of two ceramic materials indicated for monolithic restorations with dual resin cement, and also to evaluate the stresses generated at the adhesive interface of these materials. The hypotheses of this study were: 1) the adhesive strength of the glass ceramic would be superior to the zirconia; and 2) there would be no difference between the materials in the distribution of stresses during the shear test.

MATERIALS AND METHODS.

Shear stress

FEA methodology was used to verify the shear stress generated at the adhesive interface between resin cement and ceramic. For this, a three-dimensional (3D) model was constructed using computer-aided engineering software containing the same geometry of the samples used in

the *in vitro* test. Thus, the 3D model was composed of a resin cylinder (Ø = 3.4mm and h=3mm) located in the center of a ceramic slice (10x10x2mm). After checking the geometries as volumetric solids, the models were exported to the computer-aided engineering software.

During the pre-processing, the elastic modulus (E) and Poisson's ratio (V) were calculated based on the literature, and defined for each simulated material in the structural static analysis: resin cement (E=8; V=0.33),[15] zirconia reinforced lithium silicate (E=65.6; V=0.26)[16] and high translucency zirconia (E=210; V=0.33).[17] All materials were considered isotropic, linear and homogeneous, while the adhesive interface was considered perfectly bonded.

The subdivision of the geometry into finite elements followed the mesh convergence test at 10% significance, thereby defining 209,836 nodes and 113,156 elements for the final model. The fixation of the system was around the ceramic slice and the loading (100 N, Z axis) was performed at 0.2mm (wire thickness used in the *in vitro* test) of the adhesive interface. The required results were at maximum principal stress and maximum shear stress, both on the ceramic surface and on the resin cement cylinder surface. The quantitative stress peaks were selected from the center and from the periphery of the adhesive area and plotted on a bar graph for further comparison.

Shear Bond Strength

For sample preparation, the CAD/CAM (Computer-Aided Design/Computer-Aided Machine) blocks of both ceramics were used: a zirconia reinforced lithium silicate (ZLS) and a high translucency zirconia (YZHT), both of Vita Zahnfabrick, Bad Sackingen, Germany. Twenty-four slices (10x10x2mm) of each material were prepared using a precision cutter (Isomet 1000, Buehler, Lake Bluff, USA) under constant irrigation.

The ceramic surfaces were regularized with the aid of sandpaper (#600) in order to remove defects and standardize the flat faces. The samples were then polished (#600, #800, #1000 and #1200) in an automatic polisher (EcoMet/AutoMet250, Buehler) with constant pressure of 20N and 450RPM. The samples were cleaned in an ultrasonic bath (Cristófoli Equipamentos de

Biossegurança LTDA, Paraná, Brazil) for 280 seconds with isopropyl alcohol. After, YZHT slices were sintered and ZLS slices were crystallized according to the manufacturer's recommendation. Finally, the samples were partially included in acrylic resin. The YZHT samples were surface treated by silica-coating (30μm) at 2 bar. ZLS samples were subjected to superficial chemical conditioning with 10% hydrofluoric acid for 20 seconds, followed by washing and drying.

Then a silane agent (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein) was applied to the surfaces. Next, a resin cement cylinder (Fill magic dual cement, Vigodent, Rio de Janeiro, Brazil) (Ø=3.4mm and h=3mm) was built. For this, the base paste and catalyst paste of the cement were mixed and then the material was taken into the silicon matrices with the help of a centrix syringe and photopolymerized for 20s (1200mW/cm² - Radii Cal, SDI, Australia).[18]

The matrices were removed and half of the samples were stored in distilled water and submitted to a shear test after 24 hours. The other half was stored for 180 days for aging simulation (a). The shear strength test was performed in a universal testing machine (DL-1000, EMIC, São José dos Campos, Brazil), and the load was applied at the base of the cylinder by a steel wire (0.2mm in diameter) at a speed of 0.5mm/min and a load cell of 50kgf until specimen fracture (Figure 1). Figure 1 consists of the reproduction of the test pieces used in both the *in vitro* and the computational tests. The bond strength was calculated by the formula: R=F/A, where R=adhesive strength (MPa); F=force (N); and A=interfacial area (mm). Statistical analysis was performed using two way analysis of variance – ANOVA (Material and Aging) and Tukey test for group comparison, both with significance of 5%.

Failure analysis

The surfaces of fractured specimens were examined in stereomicroscope (Stereo Discovery V20, Zeiss, Göttingen, Germany) and the failure types were classified as: A) Adhesive along the ceramic/cement interface; B) Cohesive of ceramic; C) Cohesive of cement; D) Mixed (adhesive failure along ceramic/cement interface + cohesive failure of cement).

Figure 1. (a) Representation of the final specimen submitted to a shear bond test with metallic wire; (b) Representation of the rupture moment of the adhesive interface between cement and ceramics.
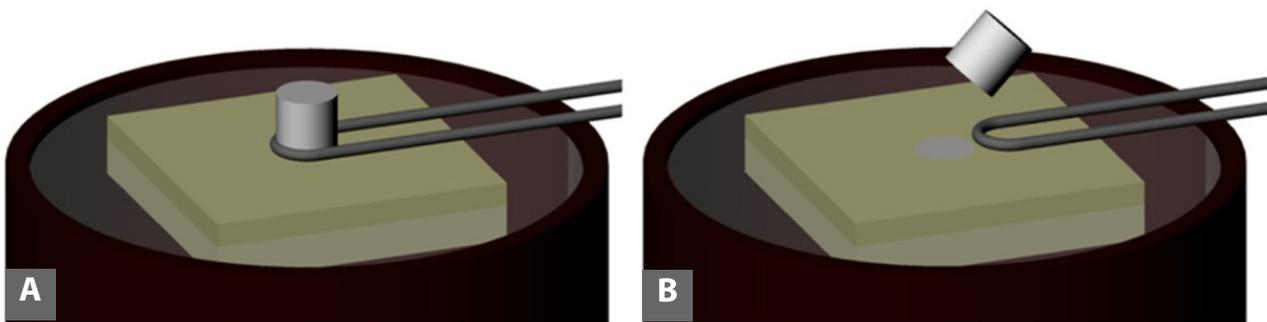
RESULTS.

Two-way ANOVA revealed that the "Material" factor (*p*=0.002) presented statistical significance.

On the other hand, the "Aging" factor (*p*=0.26) was not statistically significant for the average values of bond strength. Using failure analysis, it was observed that ceramic/cement adhesive failure occurred in 100% of the cases (Figure 2).

An analysis of the generated stresses showed that both ceramics showed both tensile and shear stress on the ceramic surface and on the cement cylinders surfaces, and always with greater magnitude at the periphery of the adhesive interface (Figure 3).

The stress peaks (Figure 4) show that the difference between the ceramics was not significant (10%), while the tensile result represented approximately 87% of the value of shear stress in the ceramics and 89% in the cement cylinder. Another important fact is that the higher values are found in the cement cylinders, thus suggesting that the failure could be originated from this material.

Figure 2. Micrographs of representative samples of failure analysis,respectively:
(a-c) YZHT, (d-f) YZHTa, (g-i) ZLS, and (j-l) ZLSa.
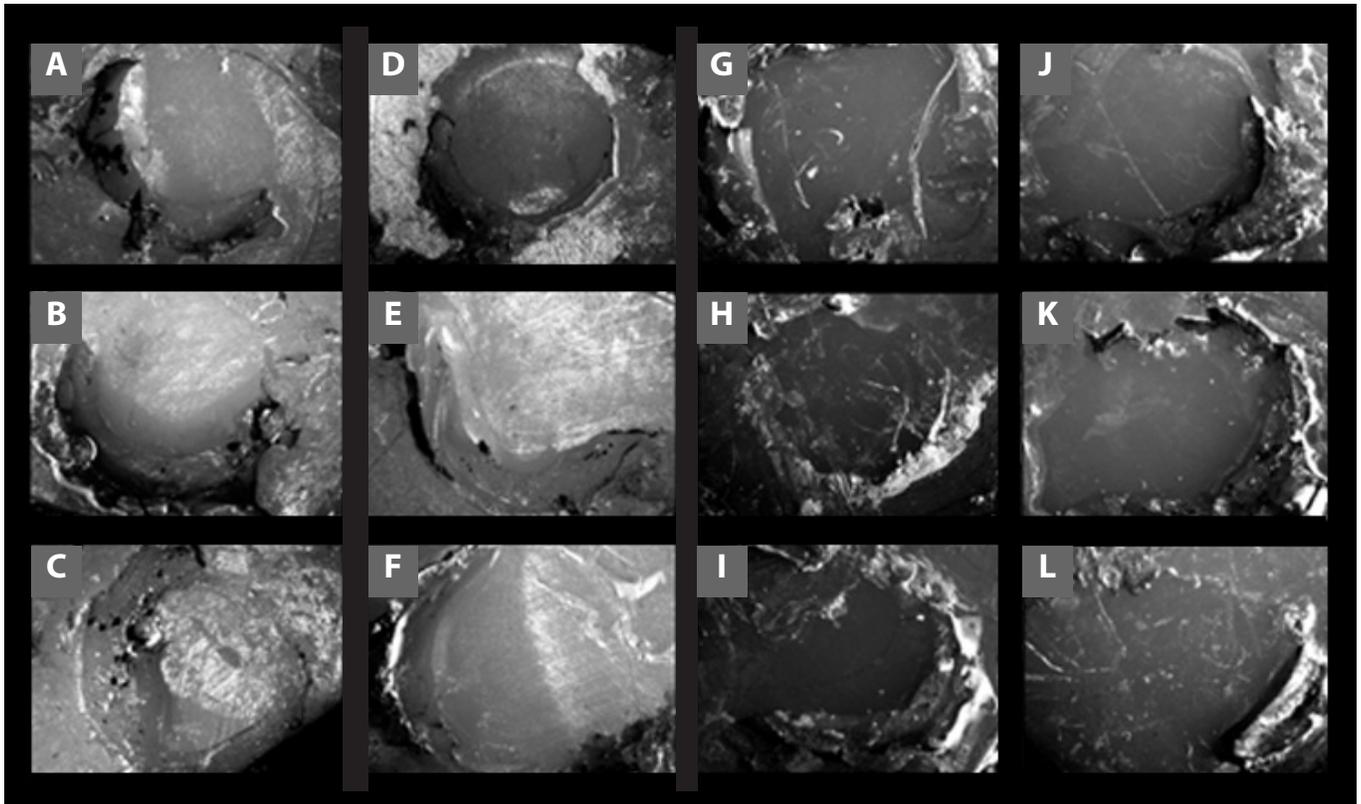
Figure 3. Results of shear and tensile stress at the surface of the resin cylinder and the ceramic for both groups. (a) shear stress at the surface of the ZLS, (b) tensile stress at the surface of the ZLS, (c) shear stress at the surface of the YZHT, (d) tensile stress at the zirconia surface, (e) shear stress in the cement on the ZLS, (f) tensile stress in the cement on the ZLS, (g) shear stress in the cement on the YZHT and (h) the tensile stress in the cement on the YZHT.
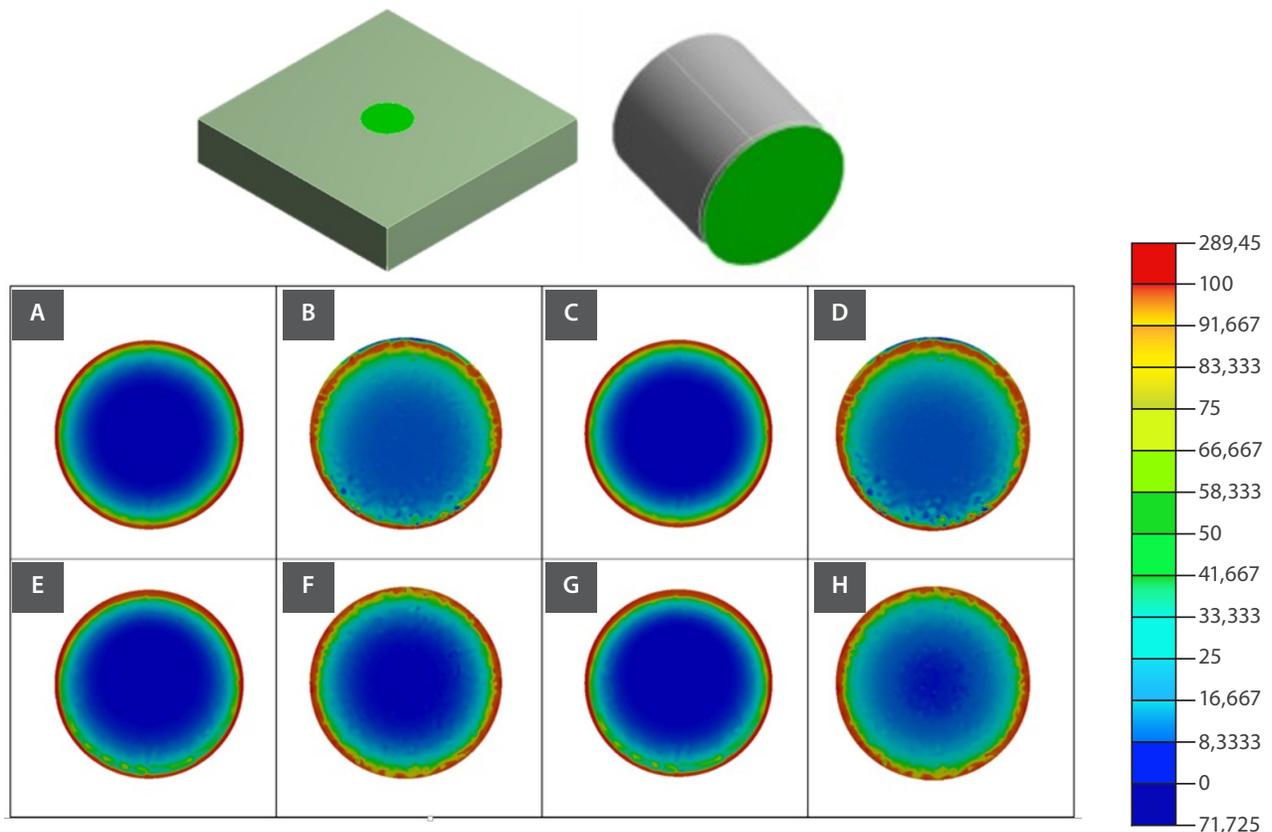
Figure 4. Bar graph of tensile and shear stress peaks on the ceramic and resin cement surfaces.
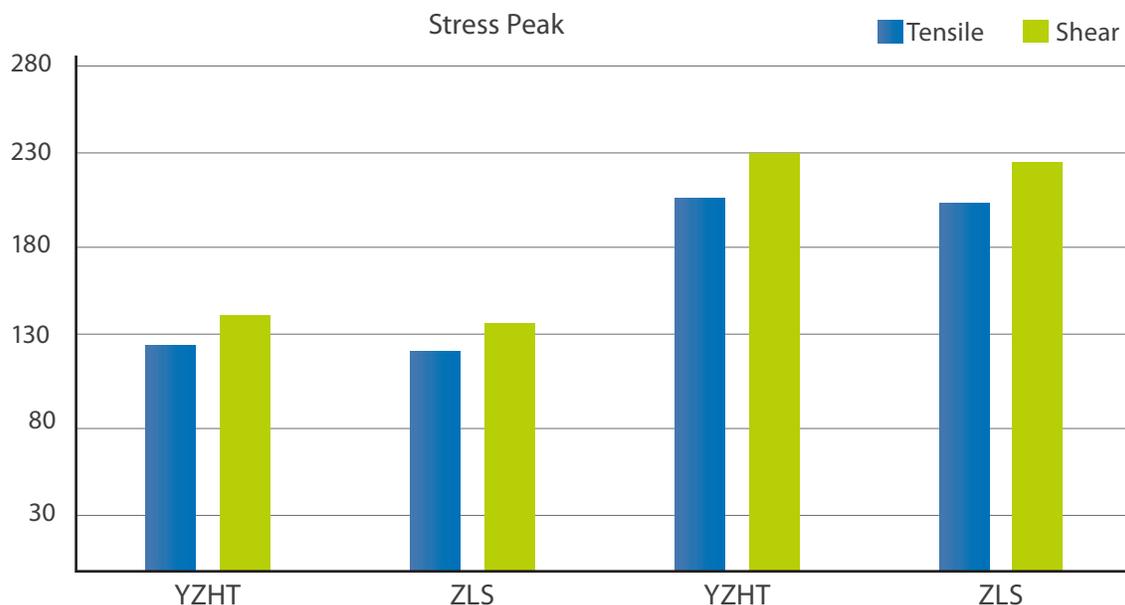
Table 1. Descriptive statistics of the mean values of bond strength (MPa) and standard deviation.

Material (p=0.002)	Aging (p=0.26)	MPa
ZLS	No	11.88±5.40[B]
ZLSa	Yes	11.76±3.32[B]
YZHT	No	19.80±6.44[A]
YZHTa	Yes	17.95±7.21[A]

Groups that share the same letter do not differ statistically (>0.05).

DISCUSSION.

The present study evaluated the bond strength of two different ceramic materials through shear bond test. The results showed that zirconia presented higher bond strength than the glass ceramic, thus rejecting the first hypothesis. The second hypothesis was accepted because no significant difference was found between the generated stresses at the bond interface of both materials.

De Hoff *et al.*,[19] questioned if the specimen submitted to the shear bond test actually failed due to shear or tensile stresses. This doubt exists due to the complexity of the resulting material involved during the load application in this experimental model. For this, the authors used finite element analysis and found both tensile and shear stresses at the interface periphery of the tested materials. The results herein corroborate with the study. Although the magnitude of the shear stress was higher on both

ceramic surfaces and also on the resin cement cylinder surface, it cannot be affirmed that tensile stresses were not responsible for part of the generated failures. Another paper suggests using a shear test instead of a microshear bond test due to less tensile stress generated on the bond surface,[20] even though the smaller adhesive surface has a lower concentration of defects.[21] Finite element analysis consists of a mathematical method that provides absolute values of the stress distributed between different structures.[22] It is widely used because of the possibility of providing more complete results compared to other *in vitro* methodologies,[23,24] saving time and being less onerous.

Tensile results suggest that even if the ceramic materials have different elastic modulus and mechanical properties, and if adhesion were ideal, little or no difference would be expected between them. This is because the stress was distributed in a similar way. In spite of this, the *in vitro* test

demonstrated that high translucency zirconia ceramic was superior to zirconia reinforced lithium silicate ceramic for bond strength. For that, each ceramic received the surface treatment indicated by the manufacturer. The zirconia was sandblasted with silica-coated alumina particles; a procedure already based on the literature as a protocol to promote greater bond strength between zirconia and resin cement.[11] However, the application of primers without the previous blasting of particles has already been suggested.[10] Blasting is capable of promoting mechanical cleaning and creating micro-retentions on the surface when done properly. The technique used allows the deposition of silica on the ceramic surface, further improving the zirconia adhesive property.[25] For zirconia reinforced lithium silicate ceramic, the surface treatment consisted of conditioning with hydrofluoric acid. This material is consecrated as a treatment method of acid-sensitive vitreous ceramics and which generally promotes values of bond strength higher than zirconia.[26] Nevertheless, this behavior was not observed in the results herein. In this way, we can suggest that another property is influencing the bonding to the cementing agent. The surface free energy consists of a surface property directly related to the material's adhesiveness. When it is evaluated, the contact angles formed between the material surface and a polar and apolar liquid show that polished or glazed high translucency zirconia has higher free energy than zirconia reinforced lithium silicate.[5] This suggests that this zirconia presents a promising adhesive property. This fact can still be reaffirmed through the durable bond strength observed through the aged groups. Storage in water is a method already based on the literature[10] and is capable of accelerating the adhesive interface degradation. Although statistically inferior, the glass ceramic was also able to maintain the adhesive strength in the long term.

A silane bonding agent was used for both materials. Silane is responsible for combining the inorganic particles of the ceramic with the organic particles of the resinous cement.[27] This material is described as essential for adequate adhesive strength, and although it has been described that the adhesive interface may undergo degradation over time,[28] no aging effect was observed by storage in any of the tested materials.

As limitations of our study, we may suggest that longer storage or association with a protocol of 10,000 cycles of thermocycling could be implemented to promote even greater aging. Other studies may also address different surface treatments and bonding systems for both evaluated ceramics in order to suggest an application protocol. Although the stress distribution is similar for both evaluated ceramic systems, the high translucency zirconia presented durable and superior bond strength to the zirconia reinforced lithium silicate.

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