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INFLUENCE OF CERAMIC PRIMERS ON MICROSHEAR BOND STRENGTH TO ZIRCONIA

Carlo Theodoro Raimundy LAGO[1], Bruna Luiza Tozatti LAGO[2], João Paulo De CARLI[3], Ana Paula FARINA[4]

[1] Department of Restorative Dentistry, School of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil.
[2] Department of Endodontics, School of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil.
[3] Department of Oral Medicine, School of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil.
[4] Department of Prosthetic Dentistry, School of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil.

Corresponding author:
João Paulo De Carli
Email: joaodecarli@upf.br

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Abstract
This study aimed to investigate the effect of two ceramic primers on the microshear bond strength (µSBS) of yttria-stabilized zirconia (Y-TZP) to two types of self-adhesive resin cement and one BisGMA-based resin cement. Zirconia specimens were sandblasted with 27-µm aluminum oxide and composite cylinders were cemented with resin cement with or without the prior use of ceramic primers. Nine groups (n=12) were randomly distributed according to the cement (self-adhesive RelyX U200/3M ESPE, self-adhesive Maxcem Elite/Kerr, and BisGMA-based dual-cure RelyX ARC/3M ESPE) and ceramic primer (Z-Primer Plus/Bisco and Porcelain Liner M/Sun Medical Co.). After luting, the specimens were stored in distilled water at 37°C for 24 hours and then submitted to the µSBS test. The data were analyzed with two-way ANOVA followed by the Scheffe post hoc test ($p<0.05$). There were significant differences between RelyX U200 and other groups. There were also significant differences between the RelyX U200 group without ceramic primer and other groups without ceramic primers ($p<0.05$). Self-adhesive resin cement (RelyX U200 and MaxCem) presented higher microshear bond strength (6.17 and 2.32 MPa) than the conventional resin cement (RelyX ARC) when a porcelain primer was not used (0.43 MPa). When using Porcelain Liner M, the results of RelyX ARC (2.94 MPa) were equivalent to the results of self-adhesive cement (3.93 and 2.11 MPa). When using Z-Prime Plus, the results of MaxCem (5.36 MPa) were lower than those of RelyX U200 (9.59 MPa) but equivalent to those of RelyX ARC (6.07 MPa). When using the RelyX ARC, the use of both ceramic primers improved bond strength to zirconia. When using self-adhesive resin cement, Z-Prime Plus improved microshear bond strength values. It can be concluded that, after 24 hours, the highest µSBS results were obtained when using Z-Prime Plus and RelyX U200 self-adhesive cement.

Keywords: Ceramics. Dental prosthesis. Shear strength.

1. Introduction

Oral rehabilitation with metal-free prostheses has been achieved due to the recent development of ceramics that present improved mechanical properties. Copings and frameworks made with yttria-stabilized zirconia (Y-TZP) can restore function and esthetics successfully. Different strategies have been proposed to improve the adhesion of resin cement to Y-TZP (Alves et al. 2016).

Zirconia is a polymorphic material that has three allotropes: monoclinic, tetragonal, and cubic. The monoclinic phase is stable at 1,170°C, turning to the tetragonal phase at 2,370°C, and reaching a stable cubic phase at 2,713°C (Özcan and Bernasconi 2015). During the cooling procedure, there is a volumetric expansion that induces expansion stress and potentially causes cracks. However, this zirconia polymorphic transformation can be prevented by adding oxides (3 to 6% of CaO, MgO, Y2O3, or CeO2), which restricts the volume increase by stabilizing the tetragonal phase. If a crack grows in the zirconia, the tetragonal crystals are transformed into the monoclinic phase, resulting in a volume expansion of 3% that induces compressive stress and interrupts crack propagation (Figueiredo et al. 2017). This mechanism represents an increase in material strength, thus zirconia is known as a "smart" ceramic (Badami and Ahuja 2014).

Different strategies have been proposed to activate the Y-TZP surface to increase roughness and improve the adhesion to resin cement. Sandblasting with aluminum oxide particles or abrasion with fine-grain diamond burs are strategies to create microretentions and increase flexural strength (Mehari et al. 2020), while tribochemical coating impregnates silica particles onto the zirconia surface and allows chemical bonding via silane. Apart from the surface treatment of Y-TZP, a new category of resin cement has been developed recently to improve adhesion. The self-adhesive cement is composed of Bis-GMA (Bisphenol-A-diglycidyl ether methacrylate) and multifunctional monomers with phosphoric acid groups (MDP, 4-META GMPD, MEPS, and 6-MHPA) that can react with ceramic oxides and chemically bond to Y-TZP (Lima et al. 2019). Recently, chemical bonding to zirconia has been improved by applying specific primers. These solutions are organophosphate mixtures that react with the methacrylate groups of resin cement and carboxylic acid monomers, which interact with metal oxides in the ceramic substrate (Pilo et al. 2016; Steiner et al. 2020).

Considering the need for improving the adhesion between resin cement and zirconia to provide long-term success to esthetic restorations, this study aimed to assess the effect of two ceramic primers on the microshear bond strength (µSBS) of Y-TZP ceramic to two types of self-adhesive cement and one Bis-GMA-based conventional resin cement. The hypotheses tested in this study were: 1) the self-adhesive resin cement presents superior bond strength to zirconia in comparison with the conventional resin cement; 2) the use of ceramic primers improves bond strength to zirconia.

2. Material and Methods

Study design

Chart 1 shows the materials and their respective compositions. Nine Y-TZP blocks (Vita in-Ceram YZ, Vita Zanhfabrik, Bad Sachingen, Germany) were cut into rectangular sections (14x15x2mm) with a low-speed diamond wheel saw. The zirconia sections were sintered in a specific furnace (inFire HTC Speed, Sirona, Germany) according to the manufacturer's instructions.

Chart 1. Materials used and compositions.

Materials	Composition
Vita In-Ceram YZ 40/15 (Vita Zanhfabrik)	ZrO_2 (91-94%), Y_2O_3 (4-3%), HfO_2 (2-4%), Al_2O_3 (<0.1%), SiO_2 (<0.1%), Na_2O (<0.1%)
RelyX U200 (3M ESPE)	Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiators, stabilizers, rheological additives, alkaline fillers, silanated fillers, initiator components, pigments
RelyX ARC (3M ESPE)	Bis-GMA, TEGDMA, zirconia/silica filler, amine, benzoyl peroxide
Maxcem Elite (Kerr)	GPDM, co-monomers (mono-, di-, and tri-functional methacrylate monomers, water, acetone, ethanol, minerals, ytterbium fluoride
Porcelain Liner M (Sun Medical Co.)	4-methacryloxyethyl trimellitic acid anhydride (4-META), methyl methacrylate (MMA), 3-(trimethoxysilyl) propyl methacrylate (ϒ-MPTS)
Z-Prime Plus (Bisco)	Biphenyl dimethacrylate, MDP, ethanol

Specimen preparation

The sintered zirconia sections were embedded in acrylic resin and polished with a #600-grain silicon carbide abrasive paper under water cooling. The surfaces were sandblasted for 10 seconds with 50-µm aluminum oxide particles, using 46-55 psi pressure and at a 10-mm distance from the surface. The surfaces were cleaned with 40% phosphoric acid (K-Etchant Gel, Kuraray, Japan) for five seconds, rinsed for 15 seconds with water spray, ultrasonically cleaned for five minutes, and blot-dried.

The specimens were randomly distributed in nine groups (n=12), corresponding to the combinations of ceramic primer (No primer, Porcelain Primer M, and Z-Primer Plus) and resin cement (RelyX U200, Maxcem Elite, and RelyX ARC).

To delimitate the bonding area, an Ainsworth perforator was used to punch 0.8-mm-diameter holes in an insulating tape positioned over the ceramic surfaces. Impressions of metallic tubes (0.8-mm-diameter x 1-mm-high) were made with vinyl polysiloxane material (Virtual, Ivoclar Vivadent) and used as matrices to create composite cylinders. A nanohybrid composite was placed inside the matrices and light-cured for 40 seconds using an LED light-curing unit with an output of 600 mW/cm2. The composite cylinders were sandblasted and silanized (Monobond-S, Ivoclar Vivadent).

Each combination of resin cement and ceramic primer was applied over the delimited bonding area according to the manufacturer's instructions and then the composite cylinders were positioned and cemented with the aid of tweezers. Resin cement excess was removed with disposable brushes and light-curing was conducted for 20 seconds on each specimen side, under irradiance of 1200 mW/cm2 (Optilight Max, Dabi Atlante, Ribeirão Preto, SP, Brazil). The specimens were stored in distilled water at 37°C for 24 hours.

The specimens were submitted to a µSBS test in a universal testing machine (Instron 4444, Instron Corporation, Canton, MA, USA) at a crosshead speed of 1 mm/min using a blade parallel to the ceramic surface. A notch in the blade could be adjusted to the bonding interface. Compressive stress was exerted at the interface until failure occurred. The µSBS values were calculated in MPa by dividing the load at failure by the surface area (mm2) of the specimens. The failure mode was determined using an optical microscope at 40x magnification and classified as adhesive (A), cohesive (C), or mixed (M).

Statistical analysis

The results were statistically analyzed with a two-way analysis of variance (ANOVA). Data normality was confirmed with histograms. Hence, a Welch correction was applied to two-way ANOVA. The Scheffe post hoc test compared bond strength among the groups. The level of significance was 5% ($p<0.05$). The data were analyzed with the BioEstat software (Version 5.3).

3. Results

Table 1 summarizes the mean µSBS values and standard deviations. All failures were classified as adhesive (100%). Regarding the results within the same resin cement subgroups, the use of Z-Primer Plus resulted in a significant increase of µSBS values for the three types of resin cement ($p<0.05$). When porcelain primer was not used or when Z-Prime Plus was used, RelyX U200 showed the highest results. When using Porcelain Liner M, the RelyX U200 and RelyX ARC cement showed similar results.

Table 1. Mean microshear bond strength (MPa ± standard deviation [SD]).

	RelyX U200	Maxcem	RelyX ARC
No primer	6.17 ± 1.38^{Ba}	2.32 ± 1.02^{Bb}	0.43 ± 0.43^{Cc}
Porcelain Liner M	3.93 ± 1.57^{Ca}	2.11 ± 1.26^{Bb}	2.94 ± 1.34^{Bab}
Z-Prime Plus	9.59 ± 1.59^{Aa}	5.36 ± 0.85^{Ab}	6.07 ± 1.89^{Ab}

Two-way ANOVA with Scheffe post hoc test ($p<0.05$). Uppercase letters compare the values in each column and lowercase letters compare the values in the same line.

4. Discussion

The results obtained in the present study show that the first hypothesis was partially accepted, considering that self-adhesive resin cement presented higher microshear bond strength than the conventional resin cement when a porcelain primer was not used. However, when using Porcelain Liner M, the results of Rely ARC were equivalent to the results of self-adhesive cement. When using Z-Prime Plus, the results of MaxCem were lower than those of RelyX U200 but equivalent to those of RelyX ARC. The second hypothesis was also partially accepted because, when using RelyX ARC, the use of both ceramic primers improved bond strength to zirconia. Conversely, when using self-adhesive resin cement, only Z-Prime Plus improved microshear bond strength values.

In this study, the zirconia surface treatment was standardized by sandblasting with 50-µm aluminum oxide particles for 10 seconds. This approach has shown improvement in flexural strength (1540 MPa) due to the significant increase of the monoclinic phase by about 9.5%, which induces the formation of a compressive stress layer to counteract the degradation (failures) caused by sandblasting (Barreto et al. 2020). However, Zhang et al. (2004) reported that sandblasting causes severe damage up to 4 µm below the surface and decreases fatigue resistance by 30%. Therefore, in this study, to prevent affecting the results and promote the same surface roughness for all specimens, the particle size (50 µm), time (10 seconds), and distance (10 mm) of the aluminum oxide were standardized.

The use of BisGMA-based resin cement alone cannot establish an effective bond to zirconia. This study corroborates this assertion because RelyX ARC alone reached a mean bond strength of 0.43 MPa. Similar results were found by Kern and Wenger (1998) during tensile tests in which a BisGMA-based cement bonded to a sandblasted or silanized zirconia showed low immediate bond strength values and pre-testing failures of all specimens after thermocycling. In our research, a conventional resin cement (RelyX ARC) was used to investigate the effectiveness of chemical bonding to Y-TZP provided by ceramic primers. The results showed an improvement when using the primers, which corroborates the study by Saleh et al. (2019) that found higher shear bond strength to zirconia when using a ceramic primer before the resin cement. The improvement is advocated by the copolymerization ability of organophosphate monomers, which have an organofunctional extremity similar to resin cement monomers. Additionally, these monomers in the primers have phosphoric acid groups that bind to the metallic oxides in the ceramic substrate and work as silane (bifunctional) (Özcan and Bernasconi 2015; Khan et al. 2019).

Following the same principle, self-adhesive resin cement is composed of phosphate monomers that simplify the luting procedure by eliminating the pretreatment of the dental substrate and restorative materials (Sathish et al. 2019; Yang et al. 2020). The results of this research suggest that phosphate monomers interact with zirconia because both RelyX U200 (6.17 MPa) and Maxcem Elite (2.32 MPa) self-adhesive cement reached higher values than the RelyX ARC conventional cement (0.43 MPa). Similar findings were reported by Piwowarczyk et al. (2004), who investigated the bond strength of a self-adhesive cement (RelyX Unicem) and a conventional BisGMA-based resin cement (RelyX ARC) to an aluminum oxide ceramic. After 14 days of water storage and thermocycling, only the self-adhesive cement could keep the initial bond strength values. However, the microtensile study conducted by Attia (2011) showed similar results for a self-etch and self-adhesive resin cement, regardless of zirconia surface pretreatment (sandblasting and silica-coating with or without silanization).

The µSBS test was applied to evaluate adhesion to zirconia, which is characterized by a small specimen size compared to the traditional shear test. It is known that a larger bonding area presents lower bond strength and more complex fractures (Albuquerque et al. 2019). Numerous benefits are reported for the µSBS test, such as preparing multiple specimens on the same material surface and the possibility of using materials that are usually too sensitive for specimen preparation procedures, that is, glass ionomer, enamel, and brittle materials (Asadzadeh et al. 2019). Although some studies report the use of a steel wire (Ozcan et al. 2011) and others describe the use of a stainless-steel blade (Magne et al. 2010) to apply shear forces, there is no difference between the results obtained (Torres et al. 2009).

The adhesion between dental ceramics and resin cement results from the physicochemical interaction of the ceramic/cement interface. The increase in surface energy ceramic obtained through chemical or mechanical treatments can therefore improve the bond strength between ceramic and cement

(Della Bona and Van Noort 1995). The use of primers is simple, easy, and low-cost for professionals, making it a very common treatment in clinical practice. Therefore, the use of ceramic primers seems to be a viable option regarding the adhesion between resin cement and Y-TZP. It is essential to understand the mechanism of bond strength tests for selecting the surface treatment of zirconia, as these tests allow predicting the clinical performance of the restoration. The µSBS test has been extensively used between zirconia and resin cement because it favors stress distribution, presents a favorable number of adhesive failures, and satisfactorily simulates clinical conditions (Scherrer et al. 2010; Özcan and Bernasconi 2015).

Delimiting the adhesive area is essential because bond strength values are lower than the set-up version without the delimitation, and the incidence of cohesive failures is lower (Woo et al. 2021). Conversely, the finite element analysis showed that during the shear test, bond strength is indicated by the cohesive strength of the substrate and not at the interface, which suggests that this traditional test is not adequate to measure the bond strength between composite and ceramic (Della Bona and Van Noort 1995). Ceramics composed of high oxide content (e.g., Y-TZP) require a different surface activation as an alternative to hydrofluoric acid (Barutcigil and Kirmali 2020). Promising results have been reported when using silica coating, silanization, and sandblasting with aluminum oxide particles (Guilardi et al. 2019). However, some authors reported that tribochemical coating with silica does not provide long-term bond strength (Scaminaci Russo et al. 2019). Silica particles seem to not adhere sufficiently to the zirconia surface, and siloxane bonds (Si-O-Zr) are sensitive to hydrolytic degradation, which affects the stability of this interface (Magne et al. 2010).

The interaction with self-adhesive or conventional resin cement, both associated with ceramic primers, showed promising results due to the combination of micromechanical retention and chemical adhesion. Some studies (Guilardi et al. 2019, Barutcigil and Kirmali 2020, Pulido et al. 2020, Ustun and Ayaz 2020) corroborate our findings, except for the fact that using Porcelain Liner M resulted in similar or even lower µSBS values than the groups without primer, for RelyX U200 and Maxcem cements. The sole use of RelyX U200 reached a mean µSBS of 6.17 MPa but when Z-Prime Plus was associated with RelyX U200, the value increased to 9.59 MPa and this difference was significant. The same results were obtained for RelyX ARC and Maxcem Elite with (6.07 MPa and 5.36 MPa) or without using the primer (0.43 MPa and 2.32 MPa, respectively).

However, the use of Porcelain Liner M did not improve the bond strength values to zirconia. Bond strength decreased (6.17 MPa to 3.93 MPa) when using the RelyX U200 self-adhesive cement. The use of Porcelain Liner M did not show significant differences in bond strength when associated with Maxcem Elite cement (2.32 MPa and 2.11 MPa). There was no evidence in the literature to explain the bond strength decrease and potential chemical interaction of the phosphate monomers in RelyX U200 or the GPDM monomer in Maxcem Elite with the Porcelain Liner M ceramic primer containing 4-META. Further studies are required to clarify these findings.

Based on the results obtained and the inherent limitations of this study, it can be assumed that using ceramic primers provided higher bond strength to Y-TZP. However, further studies should address not only the immediate but also long-term bond strength to standardize the luting protocol.

5. Conclusion

The use of Porcelain Liner M resulted in similar or even lower µSBS values than the groups without primer, for the RelyX U200 and Maxcem cements. Regardless of the resin cement, the use of Z-Prime Plus significantly increased bond strength to zirconia. After 24 hours, the best results were obtained by using Z-Prime Plus and the RelyX U200 self-adhesive cement.

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