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Evaluation of Tensile Retention of Y-TZP Crowns After Long-term Aging: Effect of the Core Substrate and Crown Surface Conditioning

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Clinical Relevance

The retention of Y-TZP crowns seems to be higher when luted to dentin rather than resin composite. When cementing a Y-TZP crown using an MDP-based resin cement, tribosilicatization and application of a thin layer of low-fusing porcelain glaze might improve the retention.

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SUMMARY

This study evaluated the effect of the core substrate type (dentin and composite resin) on the retention of crowns made of yttrium oxide stabilized tetragonal zirconia polycrystal (Y-TZP), submitted to three inner surface conditionings. For this purpose, 72 freshly extracted molars were embedded in acrylic resin, perpendicular to the long axis, and prepared for full crowns: 36 specimens had crown preparations in dentin; the remaining 36 teeth had the crowns removed, and crown preparations were reconstructed with composite resin plus fiber posts with dimensions identical to the prepared dentin. The preparations were impressed using addition silicone, and 72 Y-TZP copings for the tensile test were produced. Cementation was performed with a dual-cured cement containing phosphate monomers. For cementation, the crown preparation (dentin or resin) was conditioned with the adhesive sys-

tem, and the ceramic was subjected to one of three surface treatments: isopropyl alcohol, tribochemical silica coating, or thin low-fusing glassy porcelain layer application plus silanization. After 24 hours, all specimens were submitted to thermocycling (6000 cycles) and placed in a special tensile testing device in a universal testing machine to determine failure loads. The failure modes of all samples were analyzed under a stereomicroscope. Two-way analysis of variance showed that the surface treatment and substrate type (α=0.05) affected the tensile retention results. The dentin substrate presented the highest tensile retention values, regardless of the surface treatment. When the substrate was resin, the tribochemical silica coating and low-fusing glaze application plus silanization groups showed the higher retention values.

INTRODUCTION

Yttrium oxide stabilized zirconia polycrystal (Y-TZP) ceramic has high flexural strength (around 1000 MPa)[1] and fracture toughness ($>$ 9-10 MPa $m^{1/2}$) in comparison with other dental ceramics.[2] Due to these outstanding mechanical properties, Y-TZP has been successfully used to build the infrastructure for all-ceramic crowns and bridges using computer-aided design – computer-aided manufacture (CAD-CAM) technology. Despite the good mechanical behavior, Y-TZP is a highly crystalline material, without a vitreous phase,[1] which impairs adhesion to resin cements. Therefore, different adhesion strategies have been proposed to improve the bond strength of Y-TZP to resin cements, such as ceramic surface conditioning methods, application of ceramic primers, and/or cements containing phosphate monomers.

Surface treatments on Y-TZP substrates have been extensively explored in the recent literature to improve the bond strength of resin cement to this type of ceramic.[3-10] However, the results of these studies are contradictory. For example, Ernst and others[11] compared 12 different types of cements and showed that the greatest retention value was obtained by a self-cure dental adhesive resin cement based on methyl methacrylate when tribochemical silica coating was used as the ceramic surface treatment, but the reported retention value was not statistically different from that obtained for the group with the same resin cement without the application of tribochemical silica coating.

An alternative adhesive approach has been recently introduced to improve the bond strength between Y-TZP and resin cements without the necessity of using air abrasion procedures, which may result in permanent damage of the inner surface of the crowns.[6,7,9,10,12-14] This novel surface treatment involves the application of a thin layer of low-fusing glass to the cementation surface of Y-TZP crowns in order to enrich that surface with silicon oxides and enable hydrofluoric acid etching, which can create micromechanical retention along the surface, improving bond strength.

Even though there are several laboratory studies showing that both tribochemical sandblasting and the application of a glass film can lead to improved bond strength values, few of those studies evaluated the effect of these surface treatments using an *in vitro* design that takes into account the geometry of prosthetic dental crowns, such as using the retention force test on CAD-CAM machined Y-TZP crowns.[11,15]

Another key factor regarding all-ceramic restorations is the substrate over which it will be cemented. In some clinical situations, there is a lack of remaining dental tissue so that intracanal anchorage is necessary to retain a resin composite core and enhance retention of the prosthetic crown. However, the bond strength between the resin core and the resin cement is different from that observed between a dentin core and the resin cement, which involves the formation of a hybrid layer.

The objective of this current study was to evaluate the retention force of Y-TZP crowns as a function of the cementation substrate (dentin or resin composite) and the surface treatment of the intaglio surface of the Y-TZP crowns. The hypotheses were that: 1) a dentin substrate would promote higher crown retention than a composite substrate; 2) the Y-TZP surface treatment would influence the retention values, regardless of the substrate.

METHODS AND MATERIALS

In order to determine the number of teeth per group, a sample calculation was made based on two other articles.[15,16] In order to achieve a statistical power of 80%, mean standard deviation of 1.7 and a detectable difference of 2.3 MPa, the experimental group size was determined to be 12, for a total of 72 teeth divided into six experimental groups.

The 72 teeth were numbered and, using a computer program (www.randomizer.org), divided randomly into six testing groups (Table 1).

Tooth Embedding

The coronal part of the tooth was bonded to an adapted surveyor for keeping the long axis of the

Table 1: *Testing Groups*

Groups (n=12)	Substrate	Y-TZP Surface Treatment
D-alc	Dentin	Isopropyl alcohol
D-sil		Silicatization + silanization
D-vit		Vitrification
C-alc	Fiber post + composite resin	Isopropyl alcohol
C-sil		Silicatization + silanization
C-vit		Vitrification

tooth perpendicular to the ground (horizontal plane). Self-cured acrylic resin (Dencrilay, Dencril, Caieiras, SP, Brazil) was prepared and poured into a matrix. The tooth was then inserted into the resin up to 3 mm below the cementoenamel junction.

Dental Preparation

The occlusal surface of all teeth was cut by a diamond blade mounted on a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) 4 mm above the cementoenamel junction. Conical trunk diamond burs (KG 3139 and KG 3139FF, KG Sorensen, Cotia, Brazil) were mounted in a high-speed handpiece and fixed to a modified optical microscope, enabling tooth reduction to be obtained as parallel as possible to the long axis of the tooth. Thus, the axial part was reduced to a depth of 1.5 mm (same as the bur diameter), and a standard angle of convergence was created. The height of the preparation was 4 mm.

Composite Substrate

The 72 prepared teeth were divided into two groups according to the type of coronal substrate: 36 teeth were prepared for a full crown with the exposed dentin; 36 teeth received a fiber post and composite core.

The following procedures were performed for making the resin substrate:

1) An impression of each prepared crown was made using polyvinylsiloxane (Elite Light + Body Normal Set, Zhermack, Badia Polesine, Italy). After producing the master die, a silicone matrix was fabricated to reproduce the preparation. Thus, the future reconstruction in composite had the same characteristics as the full crown preparation.
2) For post cementation, the greatest root canal of each molar received prosthetic preparation with a custom no. 2 drill of a glass fiber post system (White Post DC, FGM, Joinville, Brazil). Afterwards, the coronal and root dentin was prepared using a two-step etch and rinse adhesive system (Ambar, FGM). The dentin was etched with 37% phosphoric acid, rinsed with water for 5 seconds and dried with absorbent papers. The adhesive agent was then applied as recommended by the manufacturer and light-cured for 20 seconds (Radii-cal, SDI, Bayswater, VIC, Australia). The posts received silane (Prosil, FGM) and were cemented with a dual-cure resin cement (Allcem, FGM). The core was built up with a composite resin (Opallis), using the previously fabricated silicone matrix. The specimens were stored in water (37°C) for 24 hours. All preparations were finished with fine conical trunk diamond burs (3139FF, KG Sorensen).

Crown Manufacture

The preparations of the 72 specimens were impressed using polyvinylsiloxane (Elite Light + Body Normal Set, Zhermack), and the dies obtained were taken to the CEREC MC XL inLab (Sirona Dental Systems GmbH, Bensheim, Germany) to manufacture the crowns using the Software inLab 3.60. The copings were designed with retention on the occlusal surface for subsequent tensile testing, and the milling was performed with VITA In-Ceram 2000 YZ CUBES (VITA Zahnfabrik, Bad Säckingen, Germany), followed by sintering the crowns in a furnace (Zircomat, VITA Zahnfabrik).

Cementation

All of the crowns were cemented using a resin cement containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) (Panavia F, Kuraray Medical Inc, Okayama, Japan). The dentin and the composite resin core of all teeth were pretreated with the self-etching adhesive system (Panavia F ED Primer, Kuraray) and were luted with the resin cement according to the manufacturer's recommendations. Each infrastructure was cemented using a device that induced a force of 750 g over the Y-TZP infrastructure, according to the strategies described in Table 1.

The inner surface of the Y-TZP copings of groups 1 and 4 was only cleaned with isopropyl alcohol and silanized with silane agent (ESPE-Sil, 3M ESPE, St Paul, MN, USA), which was left undisturbed for 5 minutes for complete evaporation.

The inner surface of the copings of groups 2 and 5 was air-abraded with 30 μm particles of aluminum

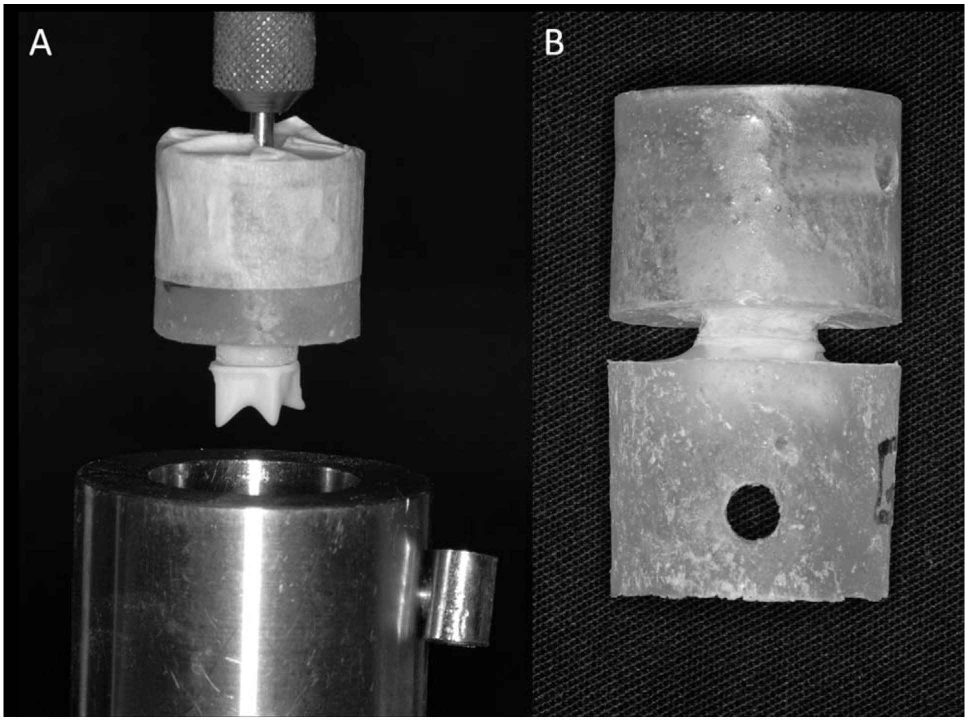

Figure 1. *Preparation of the specimen for tensile testing. (A) Specimen positioned for embedding the Y-TZP crown inside of acrylic resin. (B) Specimen with both tooth and crown embedded in resin to be tested.*

oxide coated with silicon (CoJet sand, 3M ESPE). The sandblasting was performed using an adapted device[17] and a constant pressure of 2.8 bars at a distance of 15 mm from the occlusal infrastructure region, with circular movements. The infrastructure was silanized with a silane agent (ESPE-Sil, 3M ESPE), which was left undisturbed for 5 minutes for complete evaporation.

The inner surface of the copings of groups 3 and 6 received application of a thin layer of a low-fusing porcelain glaze (VITA AKZENT, VITA Zahnfabrik) using a brush. The infrastructure was subjected to a sintering cycle of the glaze material as recommended by the manufacturer. The surface was then etched with hydrofluoric acid for 1 minute, washed with water, and air-dried. Subsequently, the copings were cleaned again in a sonic device (5 minutes in distilled H_2O). The infrastructure was silanized with a silane agent (ESPE-Sil, 3M ESPE), which was left undisturbed for 5 minutes for complete evaporation.

Thermocycling

After cementation, all specimens were stored in distilled water at 37°C for 24 hours and then were submitted to thermocycling (6000 cycles) between baths of 5°C and 55°C, according to Ernst and others[11] and Palacios and others.[15]

Tensile Test

The infrastructure was embedded in acrylic resin (Dencrilay, Dencril) until it covered the retention areas that had been prepared on the crowns (Figure 1). This procedure was performed following the same axis of the root embedding, with the aid of a suitable surveyor.

For testing, the lower base of the assembly was fixed on a universal testing machine (DL-2000, Emic, São José dos Pinhais, PR, Brazil) and the upper base was connected to a mobile device, using a load cell of 1000 N; the tensile strength test was performed at a speed of 0.5 mm/min (Figure 2).

Failure Analysis

The fractured interfacial surface of all the specimens was analyzed using a stereomicroscope (SteREO Discovery V20, Carl Zeiss, Göttingen, Germany). The failure mode was classified as:

- predominantly adhesive at core-cement interface, when over 50% of cement remained on the crown inner surface;
- predominantly adhesive at Y-TZP-cement interface when over 50% of cement remained on the prosthetic preparation; and
- catastrophic failure (post debonding and root fracture).

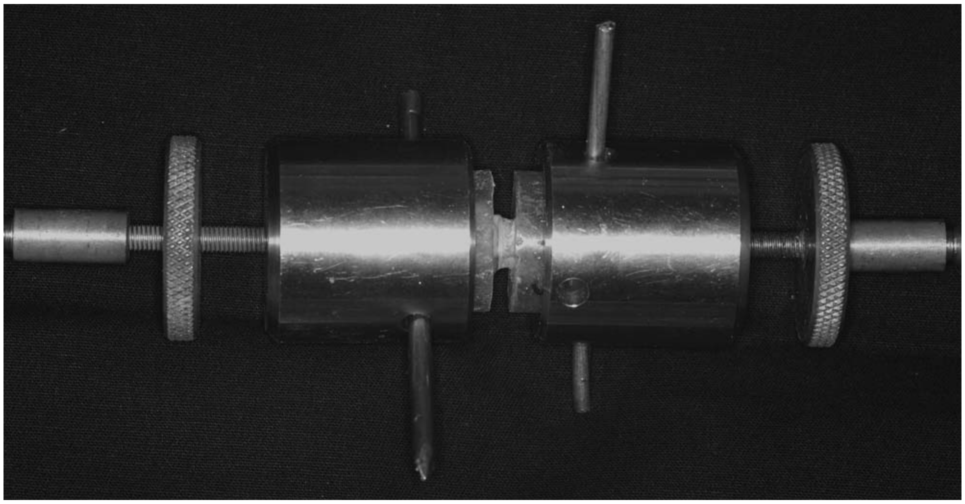

Figure 2. *Specimen placed for tensile test. The universal joints at the two superior and inferior sides can be noted at the ends.*

Statistical Analysis

The tensile retention data were compared using two-way analysis of variance (ANOVA) (substrate and surface treatment factors) and further by Tukey and the *post hoc* Student *t*-tests ($p<0.05$) for pairwise comparisons.

RESULTS

The two-way ANOVA showed that both Y-TZP surface treatment ($p<0.001$) and core substrate type ($p<0.0001$) significantly affected the retention force of the crowns. There was not significant interaction between the surface treatment and substrate ($p>0.105$). Table 2 shows the descriptive analysis and the Tukey test to elucidate the comparisons of the groups.

For crowns cemented on the dentin substrate, no difference was observed among the surface treatments tested ($p>0.05$) (D-alc = D-sil = D-vit) (Table 2). On the other hand, when crowns were cemented on a composite substrate, ANOVA showed that the surface treatment significantly influenced the retention force, with the tribosilicatization and low-fusing porcelain glaze application resulting in significantly higher mean retention values when compared to the control group (alcohol) (Table 2).

When the two core substrates (dentin and composite resin) were compared (Table 2), it is possible to note that bonding to dentin resulted in significantly higher retention mean values when compared to resin composite for both the control and silicatization groups. For the glazed specimens, there was no significant effect of the substrate on retention force.

The failure analysis indicated that for the groups in which the crowns were cemented on dentin, there was a higher percentage of adhesive failure at the Y-TZP-cement interface (cement on the crown inner surface), except for group D-alc (C-alc also), which had failures at the Y-TZP-cement interface (cement on the dental preparation). In general, when conditioning the Y-TZP surface, the failure occurred at the core-cement interface (cement remained on the Y-TZP crown inner surface) (Table 3). Most catastroph-

Table 2: *Mean (and Standard Deviation) of the Results of the Tensile Retention (N) for Different Groups**

Substrates	Y-TZP Treatments		
	Alcohol	Silicalization	Vitrification
Dentin	20.8 ± 8.1^{Aa}	24.7 ± 8^{Aa}	25.4 ± 3.4^{Aa}
Composite resin	5.2 ± 5.3^{Bb}	14.2 ± 6.9^{Ab}	20.5 ± 7.7^{Aa}

Abbreviation: Y-TZP, yttrium oxide stabilized zirconia polycrystal.
** The different lowercase letters indicate a significant difference (p<0.05) between the substrate types maintaining the same surface treatment. Different capital letters indicate a significant difference (p<0.05) between surface treatment types maintaining the same substrate.*

Table 3: *Number and percentages of failure types of the specimens from different groups.*

Groups	Failure Types		
	Adhesive at Core-Cement Interface[a]	Adhesive at Y-TZP-Cement Interface[b]	Catastrophic Failure[c]
D-alc	2 (16.7%)	10 (83.3%)	0 (0%)
D-sil	5 (41.6%)	2 (16.7%)	5 (41.7%)
D-vit	7 (58.4%)	1 (8.3%)	4 (33.3%)
C-alc	0 (0%)	10 (83.3%)	2 (16.7%)
C-sil	7 (58.3%)	2 (16.7%)	3 (25%)
C-vit	1 (8.3%)	6 (50%)	5 (41.7%)

Abbreviation: Y-TZP, yttrium oxide stabilized zirconia polycrystal.
[a] *Over 50% of cement on the crown inner surface.*
[b] *Over 50% of cement on the prosthetic preparation.*
[c] *Catastrophic failure: post debonding and root fracture.*

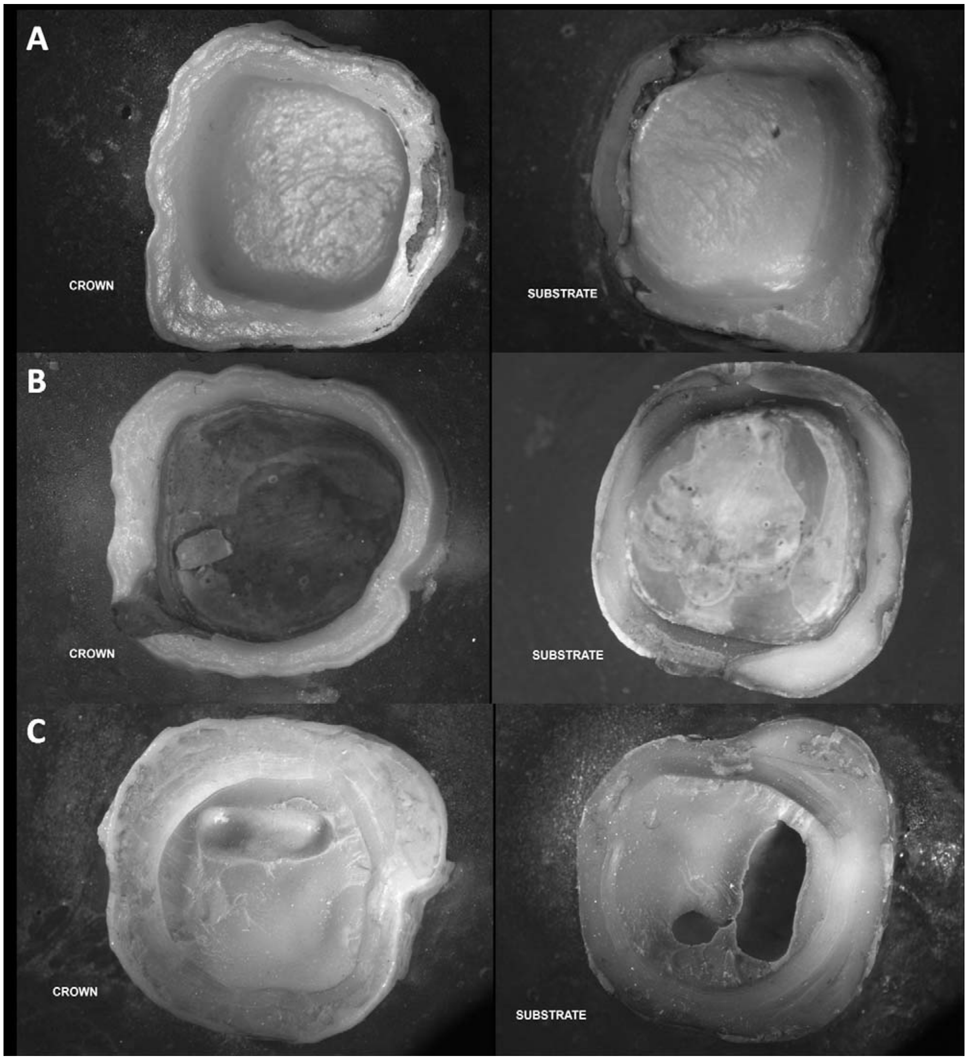

Figure 3. *Photographs from stereomicroscopy of the failure types.*

ic failures (41.7%) occurred in the groups D-sil and C-Vit. Representative stereomicroscope images of fractured specimens are presented in Figure 3.

DISCUSSION

The first hypothesis of this present study was partially accepted because the dentin substrate only resulted in significantly higher retention force for the control and silicatization groups. For the glazed specimens, both substrates resulted in similar retention values. It is possible that the better retention observed for dentin in two of the experimental groups may have occurred because the chemical composition of the resin cement used in the present investigation had a better adhesive interaction with the dentin substrate than with the resin composite. The self-etching adhesive from Panavia forms a hybrid layer with the dentin by dissolving/modifying the smear layer. Therefore, the Panavia cement penetrated into the dentinal tubules, providing micromechanical retention between adhesive and dentin. Additionally, this cement contains the MDP molecule with two ends. One end has vinyl groups that react with the monomers of the resin cement when polymerized. At the other end, hydrophilic phosphate ester groups bond strongly with metal oxides, such as alumina (Al_2O_3) and zirconia (ZrO_2),[18] and with the calcium hydroxyapatite of the dentin and enamel, which does not occur with resin substrate.

In addition, the lower retention values obtained for the resin substrate may be related to a reduction in the number of unreacted methacrylate groups at the resin surface over time, which reduces the potential for bonding to a new composite resin.[19] Another factor that reduces the bonding potential of resin composite cores is the grinding process that takes place during preparation of the core. The application of a diamond bur to a resin composite surface removes reactive monomers, exposing inorganic filler particles that lose their silane layer and therefore will have reduced bonding ability.[20]

The second hypothesis was partially accepted because a significant effect of the surface treatment on the retention forces was only observed for the resin composite surfaces. For this substrate, the application of a thin layer of a low-fusing glassy porcelain resulted in significantly higher retention values between the cement and ceramic. It is probable that the thin layer on the inner surface (approximately 10 μm of thickness)[9] might have caused increased friction to the preparation walls because the cement space in the software of the CAD-CAM system was 30 μm for all groups, and consequently, an improvement in retention for the two substrates may have resulted. Favorable adhesive behavior of the internal glaze treatment has also been observed in previous studies using other types of adhesion tests.[3,6,7,9,10,21] As a negative issue, the glaze application approach can cause a slight modification in marginal adaptation.[9]

The weak bonding interaction observed between the cement and Y-TZP for the other types of surface treatments (control and silicatization) was also reflected by the great number of failures at the Y-TZP/cement interface (83.3%) (Table 3). This unacceptable bonding behavior of untreated zirconia surfaces was also noticed in previous studies.[4,5,9,10]

With regard to tribosilicatization, though this surface treatment resulted in a very good retention force for the dentin substrate (24.72 N), previous studies have shown this method might create a critical damage zone involving grooves and defects that can act as crack initiators.[22,23] On the other hand, other investigations did not find a negative effect of this method on the Y-TZP material,[24,25] and the clinical failures of Y-TZP crowns appear to have no association with crown inner surface conditioning as well.[26,27] Thus, tribosilicatization appears to be a good option for the treatment of the inner surface of the Y-TZP crown.

One of the limitations of this current study was the occurrence of failures classified as "catastrophic" (post debonding and root fracture), which generates unreal force values. Palacios and others[15] stated that catastrophic failures occur before reaching the maximum load supported by the adhesive interface. In this current study, the incidence of this type of failure type varied from 0% to 41.7%, depending on the experimental group (Table 3). As for the statistical analysis, it is important to point out that the data concerning catastrophic failures were indeed taken into account, because if this type of failure had not occurred, the retention values would probably have been higher. According to Palacios and others,[15] catastrophic failures occur before the maximum load supported by the adhesive interface is reached. Furthermore, had these data been removed from the statistical analysis, the retention values would have been underestimated. Another important limitation of this present study was the small convergence (taper) of the preparation, which made acquiring the image difficult for the CAD-CAM processing. This problem can be overcome in future studies using greater taper of the preparations. This modification will most likely facilitate capturing the preparation image, although the resultant increase in convergence may affect the retention of zirconia crowns.[28]

The clinical relevance of this study is that it simulated different cementation protocols of Y-TZP crowns in an *in vitro* design: freshly extracted human teeth were used and core build-ups with composite resin cores and fiber posts were fabricated to simulate prosthetic cases. To the knowledge of the current authors, no previous investigation has evaluated the factors in the current study and their effect on the retention force of zirconia crowns. Further studies should be conducted to investigate other factors involved in the retention of Y-TZP crowns, such as longitudinal fatigue testing, evaluation of different cementation strategies, and other surface treatments on the inner surface of Y-TZP frameworks.

CONCLUSION

a) The cementation of Y-TZP frameworks on dentin substrates resulted in significantly higher retention values when compared to those cemented on composite resin substrates, except when glazing was applied on the inner surface of the crown.
b) For the dentin substrate, the type of treatment performed on the Y-TZP cementation surface did not affect the retention force of the crown. On the other hand, both silicatization/silanization and glaze application resulted in higher retention values for the composite resin substrate when compared to the control (no surface treatment).

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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