Original Research
Dental Materials

Can low-fusing glass application affect the marginal misfit and bond strength of Y-TZP crowns?

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Abstract: To evaluate the effect of different surface treatments on the marginal misfit and retentive strength between Y-TZP crowns and an epoxy resin. Forty (40) epoxy resin (G10) abutments (height: 5mm, conicity: 60, finish line: large chamfer) with equal dimensions were milled and included in polyurethane to simulate the periodontal ligament. Next, 40 Y-TZP crowns (thickness: 1mm) were milled (Cerec inLab) and randomly divided into four groups (n=10) according to the surface treatment: GS(glaze spray), GP(glaze powder/liquid), P(zirconia primer) and RS(tribochemical silica coating).The conditioned surfaces were cemented with dual self-adhesive cement, light cured and submitted to thermomechanical cycling (2x106, 100N, 4Hz, 5º/55ºC). Marginal misfit was analyzed by a stereomicroscope and SEM. Retentive strength test was performed (1mm/min) until crown debonding. Glaze layer thickness was also performed to GS and GP groups. Marginal misfit data were analyzed by Kruskal Wallis and Dunn tests; one-way ANOVA and Tukey (5%) analyzed the tensile strength data. The marginal misfit of the GS (48.6±19.9µm) and GP (65.4±42.5µm) were statistically lower than the RS (96±62.9µm) and P (156±113.3µm) (p=0.001).The retentive strength of the GP (470.5±104.1N) and GS (416.8±170.2N) were similar to the P (342.1±109.7N), but statistically higher than those of the RS (208.9±110N). The GS and GP glaze layer was 11.64µm and 9.73µm respectively. Thus, glaze application promoted lower marginal discrepancy and higher retentive strength values than conventional techniques.

Keywords: Biomedical and Dental Materials; Crowns; Ceramics.

Introduction

An important factor for successful ceramic restorations is a correct cementation protocol. However, the ideal cementation protocol for Y-TZP ceramic crowns is still not well defined, since these ceramics cannot be etched by hydrofluoric acid, as occurs with silica-based ceramics.[1,2] Therefore, several surface treatments have been proposed to improve bonding between Y-TZP and adhesive cements such as sandblasting with aluminum oxide particles,[3,4] Er:YAG laser irradiation,[1,5] sandblasting with aluminum oxide particles coated with silica followed by silanization,[2,6,7,8] use of MDP (methacryloxydecyl dihydrogen phosphate) monomer-based resin cement,[5,7,8,9] metallic primers,[1,3,4,5,8,9,10,11,12] selective infiltration,[8,13] and plasma spraying.[7,8]

Another proposed treatment is the vitrification of the Y-TZP surface through the application of a thin layer of glaze on the inner surface of the crown, which can then be etched by hydrofluoric acid and silanized, creating micro retentions and chemical reactivity similar to the union mechanisms of vitreous ceramics. The micromechanical retentions help establishing a strong, stable, and durable bond of the resin cement to the ceramic substrate[2,8,13,14,15,16,17]. In a recent study, Bottino et al.[2] evaluated the effect of vitrification on shear bond strength between dentine and Y-TZP ceramic, and noted that this surface treatment improved adhesion between zirconia and resin cement. Similarly, Vanderlei et al.,[18] concluded that vitrification significantly improved adhesion between Y-TZP and resin cement.

Although vitrification seems to be an efficient method,[16,19,20] the influence of this treatment on the marginal misfit of Y-TZP crowns should be investigated. Thus, the purpose of this study was to evaluate the influence of surface treatments on marginal misfit and retentive strength of Y-TZP crowns. The hypotheses tested were that a) vitrification increases the retentive strength of Y-TZP crowns bonded with resin cement; and that b) vitrification increases the marginal discrepancy of these Y-TZP crowns.

Methodology

The manufacturer, trademark, and batch number of the materials used in this study are listed in Table 1.

Epoxy resin preparations

An epoxy resin reinforced by glass fiber mesh (NEMA grade G-10, International Paper, Hampton, USA) was used to simulate a natural tooth. This material is considered analogous to dentine.[21]

A 3-D model of a right maxillary second molar with crown and root preparation was constructed in the CAD program (Rhinoceros version 4.0 Robert McNeel and Associates, USA). The coronal part had 5 mm in height, conicity of 6° and the preparation was finished in a large chamfer. Next, 20 mm diameter sticks of NEMA grade G-10 epoxy resin were milled in a specific machine (Romi D600, Santa Bárbara d'Oeste, Brazil) with a 1.5 mm spherical carbide bur to generate 40 bases with the pre-defined dimensions (Figure 1A). The milling cutter was replaced every ten preparations.

After milling, the G-10 bases were marked 2 mm below the cement-enamel junction (pre-determined as finishing line of the preparation) using a digital caliper (500, Mitutoyo Sul Americana Ltda, Suzano, SP, Brazil). The root portion was covered with polyether (Impregum Soft, 3M ESPE, Seefeld, Germany) to simulate the periodontal ligament[22]. A uniform coverage of approximately 0.3 mm was obtained by immersing the root portion in a specific plastic wax (PW 1 Plastic - Kota Imports, São Paulo, SP, Brazil) for two seconds up to the marked distance of 2 mm. The constant flow of the wax was obtained by means of an electric wax heating apparatus (Mega Bell, Cera Matic Júnior, São Paulo, Brazil) with temperature control at 90°C confirmed by thermometer.

Table 1. Commercial brand, material type, manufacturer, and batch number of materials used in the research.

Commercial brand	Material type	Manufacturer	Batch number
IPS e.max® ZirCAD	Y-TZP ceramic	Ivoclar-Vivadent/ Schaan, Liechtenstein	R55609 R83002
Rocatec® Soft	Silica-coated alumina	3M ESPE, St. Paul, Minnesota, USA	424975
VITA Akzent® Plus	Glaze Spray	Vita Zanhfabrik, Bad Sachingen, Germany	33301
Vita Akzent Glaze Fluid	Glaze powder/liquid	Vita Zanhfabrik, Bad Sachingen, Germany	21740 22601
Signum Zirconia Primer	Zirconia primer	Heraeus Kulzer GmbH, Germany	10123
Condac Porcelana	10% Hydrofluoric acid	Dentsply, Petrópolis, RJ, Brazil	9630446
Rely X U200	Self-adhesive resin cement	3M ESPE, Sumaré, SP, Brazil	544887
Rely X Ceramic Primer	Silane	3M ESPE, Sumaré, SP, Brazil	2721

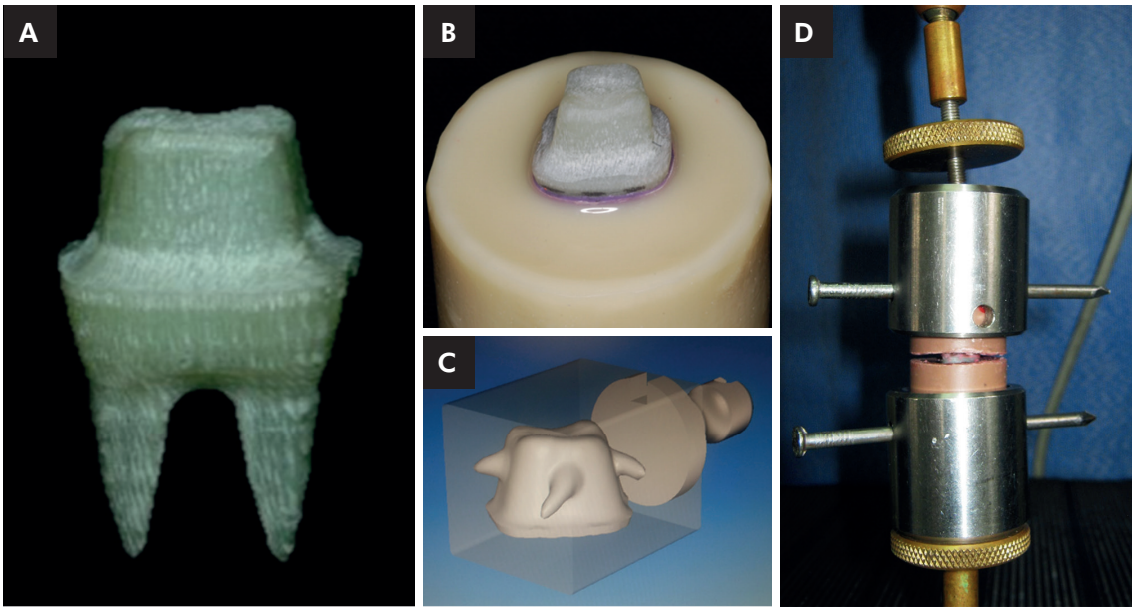

Figure 1. Preparation for total crown in a right maxillary second molar milled in G10 (A). Epoxy resin base was used for periodontal ligament simulation (B). Design of the crown milled by CAD/CAM (C). Sample in the universal test machine for the retentive strength test (D).

Next, G-10 was positioned inside a PVC cylinder matrix with 15 mm diameter and 20 cmm high. Polyurethane (F16; Axson, Cergy, France) was inserted in this matrix. After polyurethane polymerization, the set was immersed in water at 75°C for 1 min to remove the wax layer and then polyether was applied around the root surface at the cervical level, 2 mm short of the marked area (Figure 1B). Before cementation, the preparations were cleaned with pumice and water paste, water-sprayed, and air-dried, as recommended by the resin cement manufacturer.

Manufacture of Y-TZP crowns

The CAD/CAM system (Cerec inLab; Sirona; Bensheim, Germany) was used to manufacture 40 right maxillary second molars Y-TZP (Vita Zahnfabrick, Germany). Crowns were prepared with standardized anatomy and dimensions with 50 µm of internal relief. A Step Bur 20 cylindrical diamond bur was used (Sirona; Bensheim, Germany), and the milling cutter was replaced every ten preparations.

The crown design and occlusal anatomy were standardized according to the Cerec program archives. The dimensions of all crowns were standardized by epoxy resin bases. Small "hooks" were placed on the buccal, lingual, distal, and mesial areas of the crown surfaces to prevent debonding from the inclusion material during the retentive strength test (Figure 1C). Afterwards, the integrity of the crowns was evaluated by stereomicroscope (Discovery V-20, Zeiss, Germany) at a 40× magnification and no crown was discarded. The crowns were after sintered in a Zyrcomat T oven (Vita Zahnfabrick, Germany). The integrity of crowns was checked again and the internal relief of one crown from each randomly selected group was evaluated using the replicate technique with a light-weight vinyl polysiloxane/silicone film (Elite HD Light Body, Zhermack, Badia-Polesine, Rovigo, Italy).[23] The thickness of the film was measured in a stereomicroscope in different areas (Discovery V-20, Zeiss, Germany) at 30× magnification. A uniform 50-µm thickness pattern obtained from the internal relief was observed for all experimental groups. Random Allocation Software (Isfahan University of Medical Sciences, Isfahan, Iran) was used to allocate the samples among the groups.

Surface treatments and crown luting

The crowns were cleaned by ultrasound (Ultrasonic Washing Machine, Cristófoli, Campos Mourão, Brazil) with distilled water for 5 min and randomly assigned to four groups (n = 10) according to surface treatment:

RS - sandblasting with silica-coated alumina (Rocatec Soft, 3M ESPE, St. Paul, Minnesota, USA), GS - glaze spray application (Glaze Spray Vita Akzent Plus (Vita Zanhfabrik, Bad Sachingen, Germany), GP - glaze powder/liquid application (Glaze Powder/Liquid Vita Akzent Plus, Vita Zanhfabrik, Bad Sachingen, Germany), and P - zirconia primer (Signum Zirconia Bond, Heraeus Kulzer, Germany).

The RS group had the internal surfaces sandblasted using a specific apparatus (Dento-PrepTM, RØNVIG A/S) with 30-μm silica-coated alumina particles. The pressure exerted was 2.8 bar for 15 s at a 10 mm distance between the surface and the apparatus tip. Then, silane RelyX Ceramic Primer (3M ESPE, St. Paul, Minnesota, USA) was actively applied with a microbrush onto the crown, and left for 60 seconds.

In the GS group, the glaze spray was applied for two seconds at a 5-mm standard distance of the ceramic's inner surface. Then, the samples were taken to a VITA VACUMAT 6000 MP oven (VITA, Zahnfabrik, Germany) for the glaze firing process. Soon after, the crown was etched with 10% HF for 60 s, washed with air-water spray for 15 s, dried and then RelyX Ceramic Primer was applied.

In the GP group, the cement surface was treated with Powder/Liquid Glaze in the correct proportion (0.2 g of powder to 8 drops of liquid). A single layer was applied to the inner surface of the Y-TZP crown with a thin brush, and then taken to the oven for glaze firing. The HF etching and the silane application was done the same way as for the GS group.

In the P group, a Signum Zirconia Bond I + II layer primer was applied. The excess was removed with air jets and the surface was light cured for 90 s with 1200 Wm/cm^2 LED (Radii Cal, SDI, Australia).

Luting of the crowns was performed with RelyX U200 self-adhesive resin cement (3M ESPE, St. Paul, Minnesota, USA) applied to the inner surface of the treated crowns. After adaptation to the G10 preparation, a 750-g controlled pressure was exerted onto the crown. The cement excess was removed with a microbrush and each face of the crown was light cured for 20 s with 1200 mW/cm^2 LED (Radii Cal, SDI, Australia). The light intensity was measured on a radiometer (Kondortech-Kondentech, São Paulo, Brazil).

Thermomechanical cycling

A total of 2×10^6 thermomechanical cycles were performed in the ERIOS Cycler (ERIOS Cycler ER - 37000, Paraná, Brazil) with 100 N load, 4 Hz frequency and a 4 mm stainless steel piston. The piston touched the internal inclines of the buccal and lingual cusps of the Y-TZP crown's occlusal surface, establishing a 3-point contact. During the test, all samples reached a thermal equilibrium between 5° and 55°C with 60-s duration of each cycle, and a 30-s intermediate pause maintained by a thermostatic controller, which totaled 5555 thermal cycles for each group.

Marginal Misfit Analysis

After the thermomechanical cycling, the samples were evaluated by stereomicroscope (Discovery V-20, Zeiss, Germany) at 40× magnification to observe the crown's marginal misfit. The measure was obtained from the end of the preparation to the margin of the crown. Eight measurements (mesiobuccal, buccal, distobuccal, distal, distopalatine, palatine, mesiopalatine and mesial) were performed perpendicular to each crown using a ruler coupled to the stereomicroscope software (AxioVision Rel 4.8, Zeiss, Germany). The mean of the eight measurements was used for statistical analysis. Scanning electron microscopic (SEM) examination under high-resolution close-ups (Inspect S50, FEI Company, Eindhoven, Netherlands; 1000×) was also conducted.

Glaze layer thickness measurement

Glaze layer thickness was measured on the GS and GP additional crowns after the HF etching. Crowns were then mesiodistally cut in an IsoMet 1000 Precision Saw (Buehler, Lake Buff-IL, USA) and cleaned in a distilled water sonic bath for 5 min. The surfaces were then sputtered for SEM analysis at 1000× magnification. Measurements were performed in the cervical, axial, and occlusal regions. The mean of these three measurements was calculated to obtain the glaze thickness result.

Retentive strength test

The root portion of the NEMA grade G-10 was removed from the polyurethane and included in chemically activated acrylic resin (JET, Campo Limpo Paulista, Brazil). After resin polymerization,

the entire crown/G-10 interface was protected with wax (NeWWax, Quintino, Brazil) and the Y-TZP crowns were embedded in the same resin. After resin polymerization, the wax was removed and the sample was mounted on a universal testing machine (EMIC DLM 1000 - São José dos Pinhais, Brazil). All samples were submitted to the retentive strength test using a 1000-Kgf load cell at a speed of 1 mm/min (Figure 1D).

Failure analysis

A failure analysis was conducted in three steps as follows: examination of the crowns and of the G10 specimens under stereomicroscope at 50× magnification (Discovery Z-20, Zeiss, Jena, Germany) for failure classification (adhesive or cohesive) and selection of significant specimens for further SEM investigation. The chosen crowns were cut in the lingual-buccal direction and mounted on aluminum stubs; SEM examination under high-resolution magnification was conducted to observe the presence of cement on the zirconia crown and the substrate.

Statistical Analysis

For the marginal mismatch analysis, the data were submitted to the Kruskal-Wallis test and the Dunn's test. One-way ANOVA and Tukey's test were used for the retentive strength analysis. The significance level considered for all tests was 5%.

Results

Marginal misfit

The Kruskal-Wallis ($p = 0.001$) and the Dunn's test ($p = 0.0005$) indicated that the marginal misfit of the GS and GP groups were statistically lower than the RS and P groups (Table 2). The P group had statistically higher values than all the other groups (Figure 2).

Glaze layer thickness

A glaze layer thickness of 11.64 μm was observed in the sectioned extra GS crowns, and a thinner layer of 9.73 μm in the GP sample (Figure 3).

Retentive strength test

One-way ANOVA revealed a significant interaction ($p = 0.001$), indicating that bond strength was dependent on surface treatment. The retentive strength values of the GP and GS groups were similar and statistically higher than the RS group. The P group was statistically similar to the others (Table 2).

Failure analysis

The failures observed through stereomicroscopy were predominantly adhesive. The images reinforced that the resin cement remained adhered in the internal structure of the Y-TZP crowns of all groups (Figure 4).

Discussion

The difficulty of bonding the Y-TZP to the resin cement is reported as one of the main limitations for the use of this type of ceramic.[1,2,24] To evaluate the retentive strength of Y-TZP ceramics to resin cements, this study treated the Y-TZP crowns inner surface with four different surface treatments: sandblasting with silica-coated alumina, application of a zirconia primer with MDP, and application of a glaze layer by two different vitrification methods. The marginal discrepancies were also measured for each of these treatments, since the application of the glaze layer has been indicated as the cause of marginal mismatches.[8]

Table 2. Tukey's test results for tensile strength (n) and ANOVA for marginal misfit (μm).

Groups	n	Shear Strength ± SD (N)	Marginal mismatch ± SD (μm)
Glaze (powder/liquid)	10	470.5 ± 104.1[a]	48.6 ± 19.9[a]
Glaze (Spray)	10	416.8 ± 170.2[a]	65,4 ± 42.5[a]
Primer	10	342.1 ± 109.7[ab]	156 ± 113.3[b]
Rocatec	10	208.9 ± 110.0[b]	96 ± 62.9c

Different letters indicate significant statistical differences.

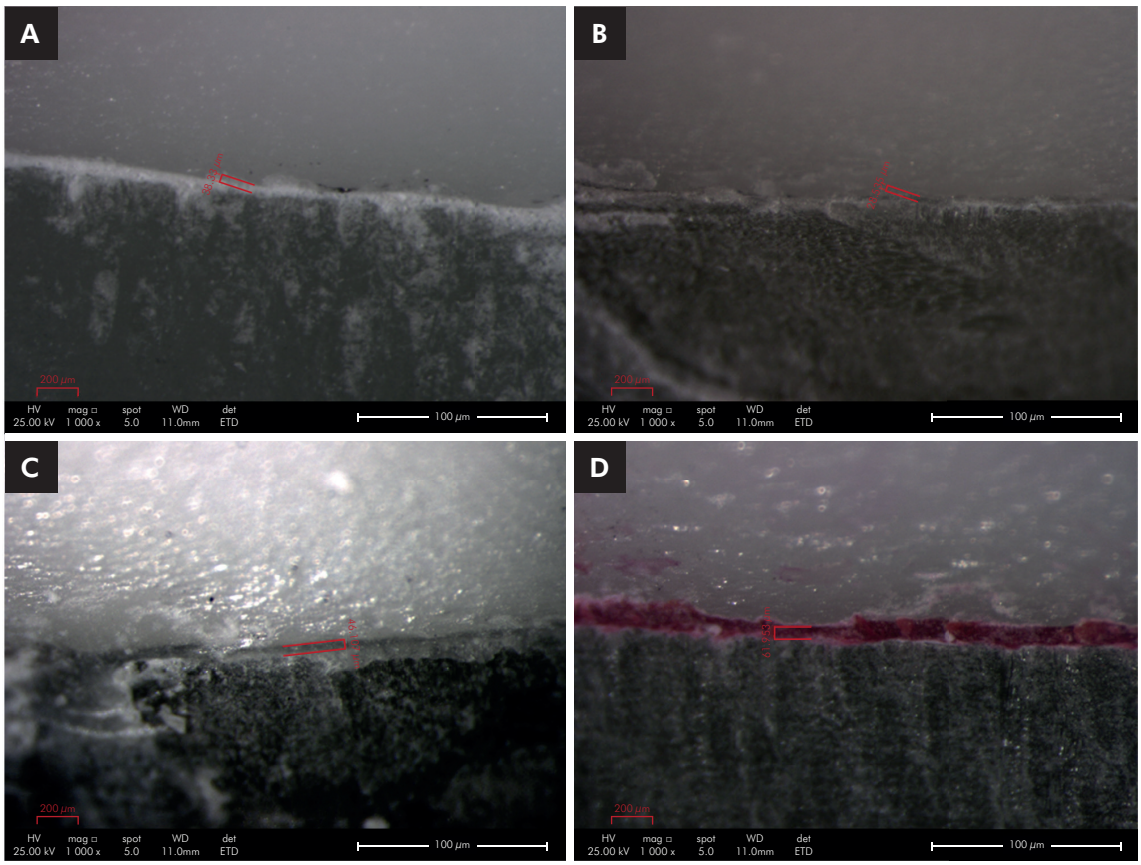

Figure 2. SEM images (40×) of the vestibular marginal discrepancy of GS (a), GP (b), RS (c) and P (d) groups after luting with resin cement. Smaller dimensions of marginal mismatch occurred in the groups in which a glaze layer was applied to the Y-TZP crowns inner surface.

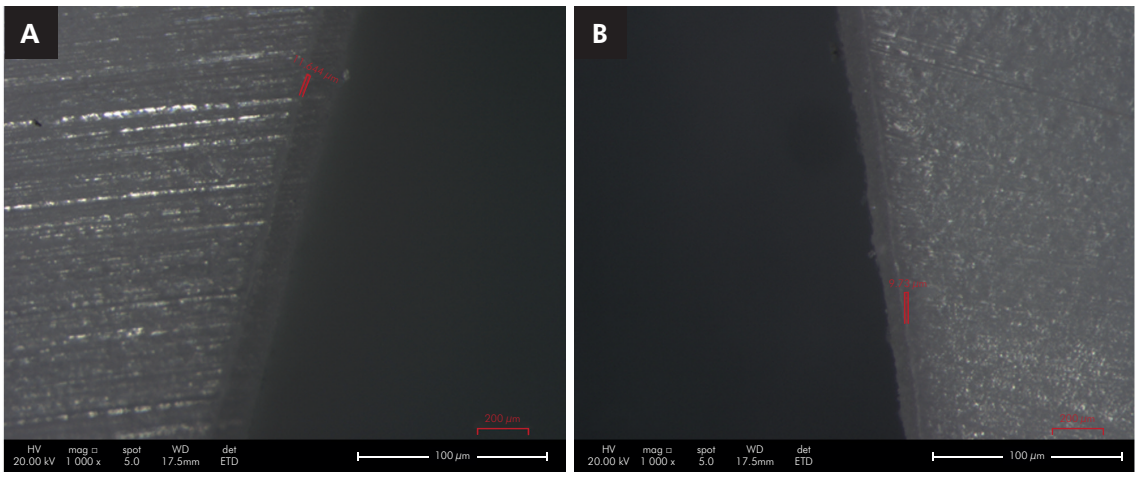

Figure 3. a) Glaze layer thickness of the GS group (11.64 µm) and b) the GP group (9.73 µm) observed in SEM using 1000× magnification.

Ideally, under laboratory conditions, the material used as a substrate for crown luting should have an elastic behavior similar to human dentine. Kelly et al.[21] evaluated the retentive strength of human dentine and a woven glass fiber-filled epoxy material (NEMA G10), obtaining reasonable results and suggesting that they have similar elastic modules and bond strength to the composite resin. This epoxy resin also showed adhesive

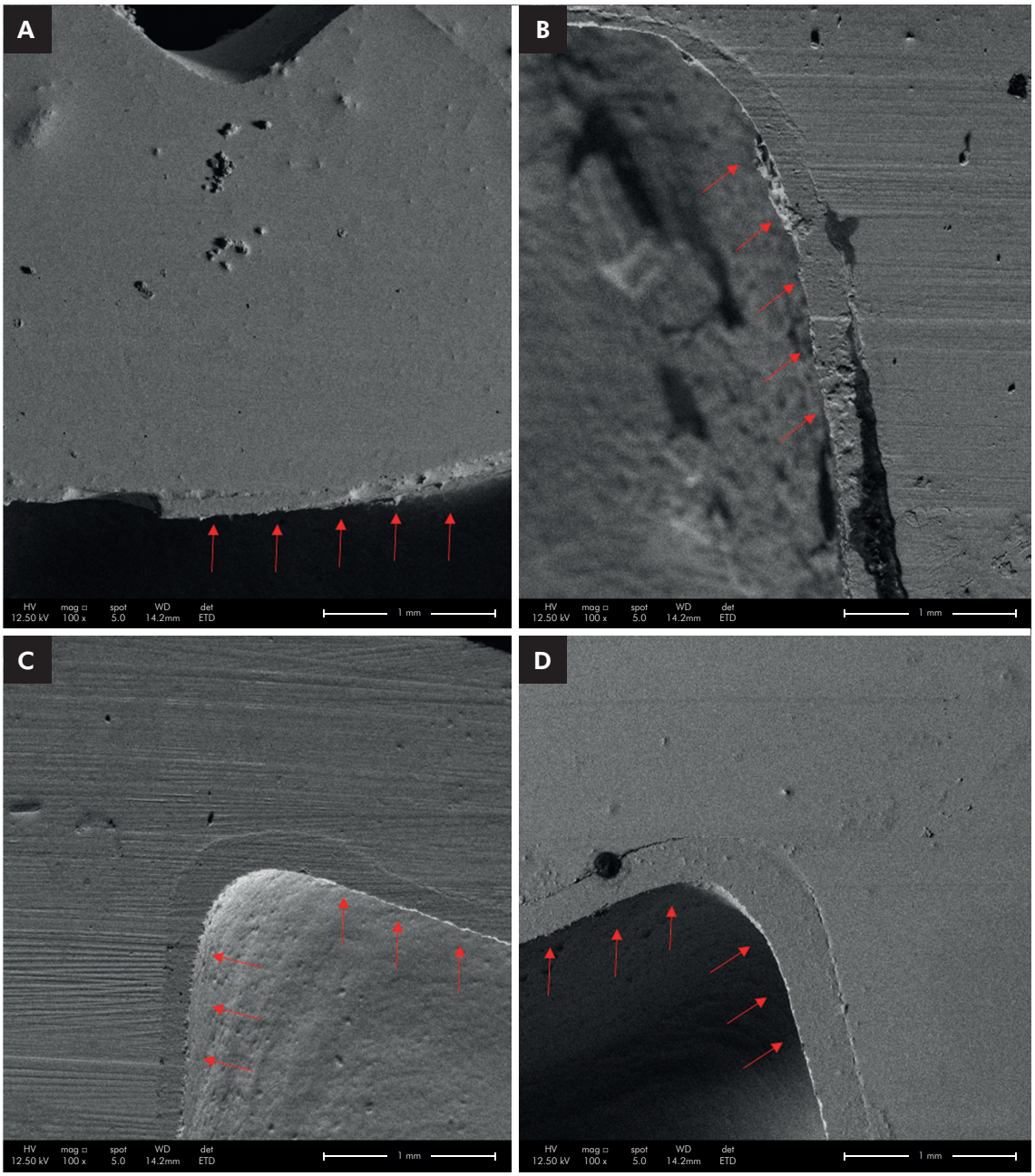

Figure 4. Micrographs showing the presence of cement on the inner surface of the Y-TZP crowns (indicated by arrows), which characterizes adhesive failures of the cement with the preparation on epoxy resin. Groups GP (A), GS (B), RS (C) and P (D).

properties[21,25,26] and is currently considered a material similar to dentine, been used as a reliable substrate in some laboratory studies[27,28]. Standardization of the substrate is crucial to verify the pretreatment factor alone in zirconia crowns.[20] To avoid possible debonding, cracking, and fracture of the covering ceramics crowns,[29,30] the present study used monolithic Y-TZP crowns.

The two methods of glaze application differ by product presentation; one is a spray (GS) and the other has a powder and a liquid component (GP). This study evaluated which method is more viable and effective. It was observed that the GP and GS presented statistically similar retentive strength values between each other and the P group, but the bond

strength value of the vitrified groups was statistically superior to the RS. This data are similar to those found by Vanderlei et al.,[18] which state that the application of low-fusing glass on the surface of Y-TZP followed by hydrofluoric acid etching and silanization significantly improved the bond between Y-TZP and resin cement.

Therefore, regardless of the application method, these two vitrification technics were effective, thus corroborating the literature[18] and leading to the acceptance of the first hypothesis. The glaze application on the Y-TZP crowns created a thin glass layer on the inner surface of this ceramic, allowing the hydrofluoric acid to create microretentions in the surface and facilitating the penetration of the luting agent.[2,13,14,15,16,17] Previous studies[17,18,31,32,33] that used tensile and shear bond strength tests reported that the application of a thin layer of low-fusing porcelain glaze on the zirconia surface followed by HF etching generated similar or higher adhesion values compared with conventional surface-treatment methods. This was correlated to a better chemical interaction mediated by the silane application process on the Y-TZP vitrified inner surface.[2,9,10,18]

The lower results of the RS group are also due to the questionable stability of sandblasting bonding over time, because hydrolytic degradation occurs together with aging and causes the silica particles to peel off the Y-TZP, causing infiltration and detachment of the crowns.[33] The thermomechanical cycling laboratorial aging was employed based on studies described in the literature[34,35] showing that applying thermal stress alone does not lead to clinically relevant deterioration.[36] In addition, thermomechanical cycling was reported[34] as being responsible for decreasing by 80% the fracture resistance of posterior crowns luted with resin cements. In the present study, the authors used 2×10^6 cycles aiming to simulate a long aging period, which corresponds to 10 years of mouth functioning on average.[36,37,38] However, the RS group achieved a retentive strength similar to the P group. This finding corroborates the literature,[12,39,40]

Although the zirconia crowns were manufactured with similar internal relief, the marginal misfit was different among the groups with different surface treatments. Contrary to some previous research[4,18] that states that the glaze on the inner surface of Y-TZP crowns may affect their adaptation, the GS (48.69 ± 19.90 μm) and GP (65.48 ± 42.57 μm) groups presented the lowest marginal misfit, and were statistically similar between them, thus rejecting the second hypothesis. The misfit values of our research were still higher than those found by Bottino et al.[2] (12 μm)[2] and Ntala et al.[17] (31.8 μm), however the clinically recommended maximum misfit is around 120 μm,[2,18] so the low-fusing glass application is not capable of causing significant marginal damages in the Y-TZP restorations.[32] In group P, the misfit value was higher than that considered acceptable (156 ± 113.3 μm). This probably occurred because this primer requires a light cure prior to the resin cement insertion and this additional stage may have favored the formation of a first layer that did not exist in the other groups in this study.

The present study had some limitations. It is difficult to compare our results with the current literature because most studies did not use crown specimens with dental preparation.[9,41,42] Maybe the restoration shape in crowns favored the maintenance of the cement on the zirconia crown. However, even though the epoxy resin used has some similarities to dentine, it cannot be directly compared to the complex bond procedure. Thus, caution should be taken in extrapolating the results to a clinical situation. Nevertheless, considering the results obtained in the retentive test and the presence of cement on the crown's inner surface, it is believed that the inner glaze application techniques can solve the Y-TZP luting problem, since it is a simple and affordable method, and the material used is readily available. With this technique, the clinician can receive from the prosthodontic lab a zirconia crown with an etchable intaglio surface. However, additional studies should be conducted to confirm the stability and durability of the bond in order to assess the time of hydrofluoric acid conditioning; follow-up studies are also needed.

Conclusion

Within the limitations of this study, it can be concluded that glaze application in the inner surface of the Y-TZP crowns promoted lower marginal discrepancy values and higher retentive strength data compared to the conventional techniques.

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