A Powdering Technique for Veneering Zirconia and Its Effect on the Flexural Strength of Ceramic Bilayers

The aim of this study was to assess the powdering technique and its effects on the flexural strength of bilayered zirconia. Bars made of zirconia partially stabilized by yttrium (Y-TZP) received porcelain by the following techniques (n = 10 per group): (1) L: VM9 application; (2) P: powdering technique + VM9 application; (3) C: Y-TZP coloring before sintering + Y-TZP sintering + VM9 application; or (4) CP: Y-TZP coloring before sintering + Y-TZP sintering + powdering technique + VM9 application. The powdering technique consisted of the application of VM9 margin powder followed by sintering. The samples were subjected to a 4-point flexural strength test and contact angle. Data were analyzed by one-way ANOVA and Tukey test (α = 5%). Surface treatments did not affect the flexural strength of bilayered specimens (P = .1264), but contact angle was affected by surface treatments (P < .0001), wherein the association of coloring and powdering (CP) reached higher values of wettability. Powdering did not affect the flexural strength of bilayered specimens, but did increase the Y-TZP wettability. Int J Periodontics Restorative Dent 2018;38:865–871. doi: 10.11607/prd.3500

The use of metal-free restorations is currently a reality in dentistry. Ceramic based on zirconia partially stabilized by yttrium (Y-TZP) is indicated for crowns and fixed dental prostheses infrastructures due to its dimensional stability, high mechanical strength and toughness, biocompatibility, and Young’s modulus, which is close to that of stainless steel alloys. Although Y-TZP is more esthetically pleasing than a metal alloy, it may also need to be veneered with feldspathic ceramic (porcelain) due to its white coloration and high opacity when a full-contour zirconia is not used.

Clinical studies have shown success rates of between 67% and 97% for prosthetic restorations made of Y-TZP and porcelain after 5 years of use; however, the success of these crowns is limited by porcelain chipping-type failures. Thus, porcelain fails cohesively with or without exposure of the Y-TZP infrastructure. The literature attributes these failures to several factors, such as residual thermal stresses, differences between the elastic moduli of the infrastructure and veneering ceramics, residual stress at the Y-TZP/porcelain interface, and poor adhesion between the Y-TZP and porcelain. The procedures used for obtaining restorations also influence the results. To improve the adhesion between Y-TZP and porcelain,
one manufacturer (VITA Zahnfabrik) indicated that porcelain application should begin with the application of a thin and fluid layer onto a previously pigmented Y-TZP. Laboratory studies showed that the use of bonding agents (liners) did not affect the flexural strengths of zirconia and porcelain. Although most liners are no longer on the market, other techniques have been proposed to improve the union between Y-TZP and feldspathic ceramic. The increase in the superficial roughness of Y-TZP has been investigated by techniques such as airborne-particle abrasion with Al₂O₃ by itself or coated with silica oxide. Airborne-particle abrasion can reduce the flexural strength values of the materials, since the impact of particles with Y-TZP surfaces leads to microdefects and causes zirconia phase transformation (tetragonal to monoclinic [te→mo]), which could affect the longevity of the restorations.

However, the increase in Y-TZP roughness could reduce porcelain displacement during sintering shrinkage, as occurs in metal-ceramic restorations. Such displacement, as reported by prosthesis technicians, leads to a separation of Y-TZP and porcelain in the restoration margins, resulting in a gap that requires additional material and a new sintering cycle.

In an effort to increase Y-TZP roughness without harming the material structure, dental laboratories have been using the powdering of feldspathic porcelain on the Y-TZP surface. The objective is the creation of an intermediate layer of porcelain by using porcelain powder and sprinkling it on the Y-TZP surface previously made wet with modeling liquid. After porcelain sintering, the resulting Y-TZP has a superficial roughness that is visually perceptible and on which the porcelain can be applied. However, there are no scientific data supporting this technique and its influence on mechanical behavior of the bilayered ceramic structure. In clinical restorations, the cervical margins of ceramic crowns have been found to be the main area where fracture is initiated. Thus, the usage of a technique that can minimize such a condition is relevant.

Therefore the purpose of this study was to evaluate the effects of a powdering technique, with or without association with Y-TZP pigmentation, on the flexural strength of zirconia/ceramic bilayered specimens. The null hypotheses were that application techniques and types of porcelain powders have no influence on the flexural strength of bilayered zirconia and ceramic specimens or monolithic ceramic specimens, respectively.

### Materials and Methods

#### Preparation of Bilayered Specimens

Table 1 presents the materials used in this study. Forty Y-TZP (VITA In-Ceram YZ Cubes, VITA) pre-sintered blocks were cut with a diamond disk (IsoMet 1000, Buehler) under cooling. Bars with standardized dimensions of 26 × 5 × 1.3 (± 0.2) mm were obtained and wet-finished with #1200 sandpaper in a polishing machine (EcoMet/AutoMet 250, Buehler). After being ultrasonically cleaned with distilled water for 5 minutes, the bars were subjected to a cleaning cycle (Vacumat 6000 MP, VITA Zahnfabrik) and randomly

<table>
<thead>
<tr>
<th>Table 1 Firing Porcelain Cycles</th>
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<tbody>
<tr>
<td>Pre-drying</td>
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<td>(ºC)</td>
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<tr>
<td>Firing cleaning of Y-TZP</td>
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<tr>
<td>Powdering over Y-TZP</td>
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<tr>
<td>1st layer of VM9 over Y-TZP</td>
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<td>2nd layer of VM9 over Y-TZP</td>
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Arrows indicate temperature increase (angled up), maintenance (horizontal), or decrease (angled down). VAC = vacuum; Y-TZP = zirconia partially stabilized by yttrium.
divided into four experimental groups (n = 10 per group) according to the VM9 base dentin procedure for porcelain application. The coloring liquid was used as liner material for zirconia infrastructures. The four groups were:

- **L** (layering): Y-TZP sintering and direct application of porcelain;
- **P** (powdering): Y-TZP sintering, powdering, and porcelain application;
- **C** (coloring; control group): coloring liquid immersion for 2 minutes followed by 2 hours of drying at room temperature, Y-TZP sintering, and direct application of porcelain; and
- **CP** (coloring + powdering): coloring liquid immersion for 2 minutes, followed by 2 hours of drying at room temperature, Y-TZP sintering, powdering, and porcelain application.

Before porcelain powder was spread onto the zirconia surface, a thin layer of modeling liquid (VITA Akzent, VITA) was applied with a brush. A dry brush was then used to pulverize the porcelain powder (VM9 margin) on the wet Y-TZP surface, and, finally, a “VM9 margin” cycle was performed in a Vacumat 6000 MP furnace (VITA Zahnfabrik).

For the VM9 ceramic application, the powder and modeling liquid were mixed and applied on the Y-TZP with the aid of a stainless steel matrix. The application was performed in layers, which were condensed after excess liquid evaporation by means of a paper tissue. Two layers of VM9 base dentin were applied and individually sintered (“VM9 first dentin layer” and “VM9 second dentin layer” cycles; Table 2).

Under constant coolant irrigation and using #1200 sandpaper, the bilayered bars were polished in a polishing machine to standardized dimensions of $20 \times 4 \times 2$ (± 0.2) mm (the thicknesses of both materials were the same). The porcelain borders that would be subjected to tensile stresses were beveled in accordance with ISO 6872:2008. No aging procedure was performed.

### 4-Point Bending Test

The specimens were subjected to a 4-point bending test in a wet atmosphere by means of a device with fully articulated application and support rollers (Fig 1) in a universal testing machine (DL 1000, EMIC; 1,000 kgf load cell, 1 mm/minute). The fine adjustment among specimens, support, and rollers ensured the homogenous distribution of load at these points, even with the slight variations in specimens. The equations for flexural strength calculation were as previously described by Lima et al.

### Fractographic Analysis

The failure modes of the fractured specimens were analyzed by stereomicroscopy (Discovery V20, Zeiss). Representative samples were analyzed by scanning electron microscopy. For this, each sample was coated with a thin gold layer at low pressure by means of an ion sputter-coater (SC7620 ‘Mini’ Sputter...
Table 3  Mean Values (in MPa), Standard Deviations (SD), and Coefficients of Variation of 4-Point Bending Data

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean (SD)</th>
<th>Coefficient of variation (%)</th>
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<tbody>
<tr>
<td>L</td>
<td>51.31 (9.12)</td>
<td>17.79</td>
</tr>
<tr>
<td>P</td>
<td>40.34 (16.13)</td>
<td>39.98</td>
</tr>
<tr>
<td>C</td>
<td>44.74 (11.84)</td>
<td>26.46</td>
</tr>
<tr>
<td>CP</td>
<td>52.09 (11.50)</td>
<td>22.08</td>
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</tbody>
</table>

Contact Angle

The contact angles of treated surfaces were analyzed by means of an optical tensiometer (TL 1000, Attension Theta Lite, Biolin Scientific) by the sessile drop technique. The syringe (1-mL Gastight Syringes #1001, Hamilton) deposited a drop of distilled water onto the sample surface, and, after a waiting time of 10 seconds, a series of 30 images per second was recorded during 20 seconds. The OneAttension software (Biolin Scientific) calculated the mean values of contact angle for each sample from the images acquired.

Statistical Analysis

The results of 4-point bending tests of bilayered and monolithic bars, and the contact angles of treated specimens were statistically analyzed by one-way analysis of variance (ANOVA) and Tukey test ($P < .05$).

Results

Table 3 shows the mean values, standard deviations, and coefficients of variation for the groups. The mean values of the 4-point bending tests varied between 40.34 and 52.09 MPa (Table 3). According to one-way ANOVA, the powdering technique and the use of a liner did not significantly affect the flexural strength of Y-TZP/porcelain-bilayered specimens ($P = .1264$; $F = 2.034$).
All specimens showed failure beginning in the porcelain and extending to the Y-TZP interface (Fig 2). Some cracks had developed fractures that were also analyzed by stereomicroscopy and SEM (Figs 3 and 4). The fractures originated from pores or defects in the porcelain tensile surface.

The contact angle was affected by surface treatments of Y-TZP ($P = .0001; F = 132.0$). The powdering significantly affected the wettability of the Y-TZP surface, and the lowest mean angles were found in the CP group (Table 4).

Table 4  Mean Values (in Degrees), Standard Deviations (SD), and Homogeneous Groups of Contact Angle Data from Tukey Test

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean (SD)</th>
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<tbody>
<tr>
<td>L</td>
<td>78.03 (8.7)$^A$</td>
</tr>
<tr>
<td>P</td>
<td>24.41 (9.9)$^B$</td>
</tr>
<tr>
<td>C</td>
<td>81.83 (7.7)$^A$</td>
</tr>
<tr>
<td>CL</td>
<td>9.93 (3.0)$^C$</td>
</tr>
</tbody>
</table>

Different capital letters indicate statistically significant differences ($P = .00001; F = 132.0$).

Figure 5 shows Y-TZP surfaces with tested treatments. The surface of pulverized Y-TZP shows porcelain clusters, and the CP group presented the most homogeneous layer.

Discussion

This study intended to evaluate the effects of the powdering technique, with or without the use of a liner, on the flexural strength of bilayered
ceramic specimens. The flexural strength was the same, no matter the surface treatments, thus confirming the anticipated null hypothesis.

The powdering technique probably minimized both the centripetal contraction undergone by the porcelain during the sinterization process as well as the differences between thermal expansion coefficients of materials\(^{11}\) that led to a nonhomogeneous cooling contraction of the porcelain. This would be possible due to additional retention that reduces the porcelain displacement at the zirconia infrastructure margins. Indeed, the powdering technique did not appear to have a negative influence on the Y-TZP surface and flexural strength, differing from surface treatments—such as aluminum oxide airborne-particle abrasion—that change the Y-TZP microstructure, cause phase transformation,\(^{18}\) and, depending on pressure and particle size, lead to lower flexural strength.\(^{21,27}\)

Clinically, there is less exposure of the Y-TZP infrastructure and protection against low-temperature degradation is obtained if porcelain displacement is avoided. Further, fewer porcelain layers and a reduced number of firing cycles on the Y-TZP surface and flexural strength, differing from surface treatments—such as aluminum oxide airborne-particle abrasion—that change the Y-TZP microstructure, cause phase transformation,\(^{18}\) and, depending on pressure and particle size, lead to lower flexural strength.\(^{21,27}\)

The surface treatments influenced the wettability of Y-TZP. Powdering reduced the contact angle of the Y-TZP surface with water, and the association of liner and powdering showed the highest wettability values. The increased surface wetting resulted in more adhesion between materials.\(^{13}\) This improved adhesion could also be achieved by surface treatments such as silica coating\(^{19}\) and airborne-particle abrasion,\(^{18}\) but these can reduce the long-term survival of the material, as previously noticed.

Microscopically, it could be observed that powdering without the previous coloring of Y-TZP generated a less-uniform, roughened pattern, with porcelain agglutination in some areas, validating what was observed during the preparation of the specimens: that the spreading of the glaze liquid during powdering on colored zirconia was more homogeneous in relation to that on the noncolored zirconia. The pigmentation liquid has oxygen, chlorine, and iron in its composition, with less than 1% of chromium, erbium, aluminum, cobalt, and sulfur. Despite the improvement in the wettability of colored surfaces, there is no evidence of interaction between coloring liquid and Y-TZP after sintering.\(^{16}\)

All specimens failed due to porcelain cracking. The defects causing fracture evolved from the porcelain surface under tensile stress and propagated until the interface was reached. From the fracture surfaces, there is no evidence of higher interaction between porcelain and zirconia.

All things considered, powdering did not change the flexural strength of bilayered flat zirconia specimens. So far, residual stresses resulting from different thermal coefficients between porcelain and zirconia, associated with chewing, clenching, and moisture, are the main issues proven to cause failures in veneered zirconia crowns.\(^{28,29}\) Additional studies should tackle the behavior of curved surfaces in concert with the powdering method.

Conclusions

Within the limitations of this study, the authors conclude that powdering did not increase/decrease the flexural strength of bilayered zirconia compared to a traditional technique that was used as control, whereas the surface roughness was increased by the association of coloring liquid with the powdering technique.

Acknowledgments

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References


