Fatigue behavior and colorimetric differences of a porcelain-veneered zirconia: effect of quantity and position of specimens during firing

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Abstract

Purpose: To evaluate the influence of quantity and positioning of zirconia specimens during firing of porcelain on their fatigue performance and colorimetric differences.

Methods: Bilayer discs (Ø=15 mm) were made, following ISO 6872 guidelines, using a Y-TZP core (yttria-stabilized tetragonal zirconia polycrystal ceramic; VITA In-Ceram YZ) and a feldspathic veneering material (VITA VM9), being both layers with 0.7 mm thickness. Y-TZP discs were sintered, the veneering material was applied over it, and the bilayer specimens were fired according to two factors (n=20): ‘quantity’ (1 or 5 samples per firing cycle; G1 and G5 groups respectively) and ‘positioning’ of the specimens inside the furnace (center or periphery of the refractory tray; G5C and G5P groups, respectively). The CIEL*a*b* parameters were recorded with a spectrophotometer and the color difference (ΔE00) was calculated using CIEDE2000 equations. The step-stress fatigue test was performed with the veneer facing down (region of tensile stress concentration), 10 Hz frequency, initial tension of 20 MPa for 5,000 cycles, followed by steps of 10,000 cycles using a step size of 10 MPa, up to 100 MPa; data from strength and number of cycles for failure were recorded for statistical analysis.

Results: Unacceptable color differences (ΔE00>1.8) were observed comparing G5C vs. G1 (quantity) and G5C vs. G5P (positioning), meanwhile translucency parameters were not affected. Besides, only the ‘quantity’ factor influenced the fatigue performance (G1>G5C). None of the tested specimens survived beyond 90N and/or 75000 cycles.

Conclusions: The quantity and position of the specimens during firing influence the final color of porcelain-veneered zirconia, and firing one specimen per cycle improved the fatigue performance of the bilayer system.

Keywords: Dental porcelain, Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), Firing, Mechanical Phenomena, Mechanical Cycling, Optical Phenomena

1. Introduction

The demand for all-ceramic restorations in dentistry has been growing over the years. Several ceramics offering satisfactory mechanical properties are available on the market, with the advantage of providing better aesthetics and biocompatibility than porcelain-fused-to-metal restorations [1]. In this sense, crystalline ceramic materials such as yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) has been used in dental restorations, since it has high strength and fracture toughness, mainly due to the toughening mechanism through phase transformation (tetragonal to monoclinic), which gives it a higher resistance to crack propagation when subjected to the cyclical masticatory loading [2,3].

However, its white color and low translucency is far from the natural appearance, which affects the esthetics of monolithic Y-TZP crowns [4]. Thus, a glass-ceramic is still necessary to veneer the zirconia core and provides better esthetic appearance [5], especially when restoring scenarios with non-favorable substrate color, e.g. darkened teeth or metallic based cores. In this context, feldspathic ceramic is highly indicated due to their excellent optical properties [6]. Thus, from a clinical perspective, the success of the bilayer systems relies on the success of both the veneering porcelain and the Y-TZP core [7].

The oral environment is challenging for this type of material since it generates different stimuli such as cyclical masticatory loading, temperature and pH variations, and humidity. Therefore, it is fundamental that veneering ceramics (such as feldspathic) have good mechanical properties through appropriate confection and firing processes. It is already known that the multiple firing cycles of the veneering feldspathic ceramic applied over zirconia cores can effectively improve its hardness [8,9]. Also, according to Hassijia et al. [10], repeating firings did not affect the color parameters of ceramics with metal core. However, it is still unknown if variations in the same firing, like the position and quantity of these veneered zirconia restorations inside the furnace, might influence their mechanical and color properties, which, could be an important issue concerning the clinical performance of restorations. Besides, the great demand for all-ceramic materials in dentistry has led to a very large workflow in...
prosthetic laboratories, which consequently produce and sinter many crowns simultaneously. This results in an overloaded furnace of porcelain crowns, without proper knowledge of the influence of this action on the final properties of the ceramic materials.

Considering these circumstances, the objective of this study was to evaluate the effect of the position (at the center or periphery of the refractory) and quantity of specimens (one or five) inside the furnace during the veneering firing cycle on the color difference, translucency, biaxial flexure fatigue strength, number of cycles for failure and survival rate of zirconia specimens veneered with a feldspathic ceramic. The assumed null hypotheses were that the (1) quantity and the (2) position of the specimens inside the furnace would not significantly affect the fatigue strength, color or translucency of the bilayer specimens.

2. Materials and methods

2.1. Preparation of the specimens

An yttria-stabilized zirconia polycrystal ceramic (3 mol% Y-TZP) was veneered with a feldspathic ceramic. The specimens were made following the ISO 6872:2015 [11] guidelines for biaxial flexure test (piston-on-three-ball test). Details of the materials are described in Table 1.

The zirconia discs were obtained from pre-sintered blocks (VITA In-Ceram YZ for iInLab 40/19, Bad-Säckingen, Germany). Two metal cylinder guides of 18 mm diameter were attached parallel to both sides of the Y-TZP blocks, and the lateral surfaces were then ground using a polishing machine (EcoMet/AutoMet 250, Bucheler, Lake Bluff, USA) with 600 grit silicon carbide sand paper until a cylinder was shaped. Next, the cylinders were cut, under water irrigation, with a diamond disc in a precision cutting machine (Isomet 1000, Bucheler) in slices of 1 mm thickness, polished with #600 and #1200-grit silicon carbide sand papers on both sides and then cleaned in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 10 minutes. After, the zirconia discs were sintered (VITA Zyrocomat 6000 MS, VITA) according to the manufacturer’s recommendations (Table 1), resulting in zirconia discs with final dimension of 15 mm in diameter and 0.7 mm thickness, inspected with a digital caliper.

Next, the feldspathic ceramic (VITA VM9, VITA) was applied using the layering technique. It was not executed surface treatments of the zirconia prior to the application of porcelain, as a previous study shown absence of positive influence of a liner application on adhesion with feldspathic ceramic [12]. The ceramic powder was handled with softy’s Elite, Melhoramentos, Sao Paulo, Brazil. All specimens were prepared by the same trained operator. The ceramic set was then placed around the zirconia to compensate the porcelain contraction during the firing cycle, hence avoiding the discovery of the infrastructure. The excess liquid was removed at each increment with absorbent paper (softy’s Elite, Melhoramentos, Sao Paulo, Brazil). All specimens were fired over black (L*= 27.94, a*= -0.01, b*= -0.03), white (L*= 92.95, a*= -0.78, b*= 3.57), and gray (L*= 50.30, a*= -1.41, b*= -2.37) backgrounds, and a coupling solution (glycerol C3H8O3, Vetec Química Fina Ltda, Rio de Janeiro, Brazil) was used to limit light scattering between the product and the background [14]. All the measurements were carried out in the same place and period of the day. The spectrophotometer was recalibrated every 20 measures to avoid discrepancies between assessments. Each specimen was measured three times, and the averages of L*a*b* coordinates were used to evaluate the effect of the position (at the center or periphery of the refractory) and quantity of specimens (one or five) inside the furnace during the veneering firing cycle on the color difference, translucency, biaxial flexure fatigue strength, number of cycles for failure and survival rate of zirconia specimens veneered with a feldspathic ceramic. The assumed null hypotheses were that the (1) quantity and the (2) position of the specimens inside the furnace would not significantly affect the fatigue strength, color or translucency of the bilayer specimens.

2.2. Evaluation of color and translucency parameters

Color and translucency parameters were evaluated using a spectrophotometer (VITA Easyshade, VITA). CIE L*a*b* coordinates were recorded, in which L* is the luminosity axis with values varying from 0 (black) to 100 (white), and a* and b* are the color coordinates in the green-red axis and the blue-yellow axis, respectively. The measurements were performed over black (L*= 27.94, a*= -0.01, b*= -0.03), white (L*= 92.95, a*= -0.78, b*= 3.57), and gray (L*= 50.30, a*= -1.41, b*= -2.37) backgrounds, and a coupling solution (glycerol C3H8O3, Vetec Química Fina Ltda, Rio de Janeiro, Brazil) was used to limit light scattering between the product and the background [14]. All the measurements were carried out in the same place and period of the day. The spectrophotometer was recalibrated every 20 measures to avoid discrepancies between assessments. Each specimen was measured three times, and the averages of L*a*b* coordinates were used to evaluate the effect of the position (at the center or periphery of the refractory) and quantity of specimens (one or five) inside the furnace during the veneering firing cycle on the color difference, translucency, biaxial flexure fatigue strength, number of cycles for failure and survival rate of zirconia specimens veneered with a feldspathic ceramic. The assumed null hypotheses were that the (1) quantity and the (2) position of the specimens inside the furnace would not significantly affect the fatigue strength, color or translucency of the bilayer specimens.

The specimens were visually inspected under an optical microscope (Stereo Discovery V20; Carl Zeiss, Göttingen, Germany) to verify the presence of porosities or defects on their surface, if so the specimen was discarded and replaced by a new one.

The difference pair in L*, a*, b* coordinates, and the parametric factors (factor) were calculated by CIEDE 2000 (equation 1) from the CIE L*a*b* coordinates evaluation. The specimens were cleaned with alcohol and dried with absorbent paper. Color differences (ΔE<sub>ab</sub>) between the central G5 specimens (GSC) and the G1 group specimens (quantity factor), and between the center (GSC) and the periphery (GSP) specimens (position factor) were calculated by CIEDE 2000 (equation 1) from the measurements performed over a gray background. The perceptibility and unacceptability thresholds (0.8 and 1.8, respectively), as described by Paravina et al. [15], were considered for evaluating if the color differences (ΔE<sub>ab</sub>) caused by quantity or position of specimens during firing were clinically relevant.

$\Delta E_{ab} = \left( \frac{\Delta L^*}{K_{L^*}} \right)^2 + \left( \frac{\Delta a^*}{K_{a^*}} \right)^2 + \left( \frac{\Delta b^*}{K_{b^*}} \right)^2 + R_1 \left( \frac{\Delta \alpha^*}{K_{\alpha^*}} \right) \left( \frac{\Delta \beta^*}{K_{\beta^*}} \right)$

(1)

where $\Delta L^*$, $\Delta a^*$, and $\Delta b^*$ are the differences in luminosity, chroma, and hue, respectively, for a pair of measurements. RT is a rotation function which accounts for the interaction between chroma and hue differences in the blue region. SL, SC, and SH are weighting functions that adjust the total color difference for variation in the location of the color difference pair in L*, a*, b* coordinates, and the parametric factors kl, KC, and KH are correction terms for deviation from reference experimental conditions. These parametric factors were set as 1.

Measurements performed over the black and white backgrounds were used to calculate the translucency parameter (ΔΔTP<sub>ab</sub>) of each experimental group, using the same aforementioned equation, where the perceptibility and unacceptability thresholds to verify if there are clinically relevant differences between G1 and GSC, and GSC and
GSP, were considered as 0.62 and 2.62, respectively [16].

2.3. Fatigue strength test

The specimens were subjected to the biaxial flexure fatigue strength test in an electro-dynamic machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, United States) by applying cyclic intermittent loads using the assembly preconized by ISO 6872:2015 [11] for ceramic testing. The specimens were placed in the device on a metal base over three equidistant supporting steel balls (10 mm distance a part). The set was submerged in distilled water and the specimen was concentrically placed on the supporting balls of the device with the feldspathic side facing down (tensile side). Next, a tungsten piston with a 1.6 mm diameter flat cylindrical tip was used to apply the cyclic load on the specimen [11]. An adhesive tape was glued to the specimens on the compression side before the test for better contact between the piston and the specimen [17]. A permanent marker was used to mark the center of the disc and ensure the same positioning of the specimen in relation to the piston after each step.

The fatigue strength was determined using the step-stress methodology described by Collins [18]. The method was started by applying a tension of 20 MPa, at a frequency of 10 Hz for 5,000 cycles (preconditioning phase to guarantee predictable positioning of the sphere with the specimen), followed by steps of 30, 40, 50, 60, 70, 80, 90 and 100 MPa at a maximum of 10,000 cycles each. The specimens were analyzed by transillumination at the end of each step until the first crack was found, which was considered as the main outcome for failure. Data related to the strength for occurrence of the crack and number of cycles for such outcome were collected for statistical purposes.

2.4. Qualitative fractographic analysis

As only cracks were detected, the samples were cut (Isomet 1000, Buehler) perpendicularly to the crack, then cleaned in 78% isopropl alcohol for 10 minutes by ultrasonic immersion for analysis under a stereomicroscope (Stereo Discovery V20) to determine the fracture origin. Representative specimens were selected for analysis in a Scanning Electron Microscope (SEM - Vega3, Tescan, Czech Republic). First those specimens were gold-sputtered, under high-vacuum, and the SEM analysis was executed also under high-vacuum with 20.00 kV at a working distance of approximately 13.5 mm, using secondary electron imaging at 175 and 300× magnification.

2.5. Statistical analysis

Color difference and translucency data were subjected to the Shapiro–Wilk and Levene tests, (normality and homogeneity, respectively), and to the Mann-Whitney test in order to evaluate the effect of specimen quantity (G5C Vs G1) and position (G5C Vs G5P) in the furnace during the firing process. The fatigue strength, number of cycles for failure were analyzed by the Kaplan-Meier and Mantel-

### Table 1. Description of the materials used in the study, their composition and firing cycle.

| Material (manufacturer) | Composition | Firing cycle |
|-------------------------|-------------|--------------|
| In-Ceram Yz (VITA) Lot. 48330 | ZrO₂ 3 mol%, Y₂O₃, HD₃O, Al₂O₃, SiO₂, Na₂O | Cycle starts on 50 °C; temperature increases 17 °C/min until 1,000 °C (±55 min); temperature increases 17 °C/min until 1,530 °C (±31 min); remains at 1,530 °C for 120 min; cooling until 200 °C. |
| VITA VM9 Dentine (VITA) Lot. 46310 | Pure-grade potash and albite feldspar materials | Pre-drying 500 °C for 6 min; temperature increases 55 °C/min (±7 min); remains at 910 °C for 1 min; cooling until 600 °C; vacuum for ±7 min. |

### Fig. 1. Disposition of the G1 (A) and G5 groups (B) inside the furnace for each firing cycle.

Cox post-hoc tests. Besides the survival rates for each step considered during fatigue test were obtained.

3. Results

The quantity (G5C Vs G1) and position (G5C Vs G5P) of the specimens in the furnace led to a color difference above the unacceptable threshold (ΔEₐp > 1.8). However, the translucency parameter was not affected by the quantity (G5C Vs G1, p= 0.26) and position (G5C Vs G5P, p= 0.32) factors, and the differences in the TPₐ values were not enough to reach clinically perceptible and unacceptable thresholds (ΔTPₐ < 0.62) (Table 2).

Regarding the mechanical fatigue behavior, G1 group presented higher fatigue strength and number of cycles for failure when compared to the G5C group (p= 0.000) (quantity factor). However, the specimens’ position had no statistically significant influence (G5P vs G5P, p= 0.131) (Table 3). In terms of survival rate, G1 specimens lasted for more time before failure; for instance, when considering 60 MPa, the rate for G1 is 0.50 (50% of chance to exceed the such stress), while G5C and G5P had 0 and 0.05 rates, respectively (0 and 5% of chance to exceed it) (Table 4), corroborating the other mechanical findings.

The fractography evidenced a similar pattern of failure for all groups considered, where the crack origin started at the porcelain surface from the bottom side during the fatigue test (tensile stress concentration side), being always parallel to the load application (Fig. 2).

4. Discussion

From the results of the present study, the quantity of the bilayer specimens in the furnace during the feldspathic ceramic firing affected their fatigue strength, number of cycles for failure and color measurements, but it did not alter their translucency. Therefore, the first hypothesis was partially accepted. Moreover, the specimens’ position only influenced the color measurements promoting clinically perceptible and unacceptable alterations (ΔEₐp > 1.8). Therefore, the second hypothesis was also partially accepted.

According to thermodynamic concepts [19], the porcelain firing occurs through the amount of heat generated by the furnace’s resistance, which is transferred to the specimens by radiation in the form of electromagnetic waves, since there is no direct contact between the heat source and the ceramic material. The heat treatment of the porcelain leads to the union of particles at an atomic level and consequently generates a solid structure [20]. This suggests that a higher temperature would lead to a greater amount of heat absorbed by each specimen, and therefore it results in a more compact structure, with less defects and better mechanical properties. Also, Hammad and Stein [21] reported that the increase in firing temperature increased the bond strength of ceramometals.

It is important to remember that during manufacturing restorations that are consisted of a zirconia substructure covered with a feldspathic ceramic using the layering technique, explored herein, its usual to execute many firing cycles during the application of the 1st dentin layer, 2nd dentin layer, enamel, and/or even finally with the glaze
material to achieve the final thickness of restoration [9]. So, our data corroborate that such processing would inherently lead to alterations of color, optical characteristics, and also potentially impact the mechanical properties of the future restoration, in accordance with prior studies [9,22-24]. In fact, a previous study [22] reported that veneering ceramics fired 10 times showed higher density and less porosity than those fired twice.

Thus, as the number of specimens to be fired in the same cycle increases, it could be hypothesized that the heat energy generated by the resistance is divided between the specimens, which may lead to a worst/different firing process among samples in terms of crystalline and not towards the center, which implies that the furnace indeed generates present heating differences between the center and the periphery.

Ceramic materials used in clinical practice are brittle and may fracture unexpectedly when mechanically overloaded. However, the most usual scenario for failure is mainly motivated by fatigue [25]. Fatigue failure usually occur due to cumulatively loads over time, which lead to defect’s coalescence until it reaches a critical size and the crack propagates [26]. Still on this sense, our data corroborates that to place only one specimen in the furnace per cycle is mandatory to guarantee that the firing process is optimized and the final surface characteristics and consequently the mechanical properties of the restorative assembly is enhanced, which is in accordance with prior literature [8].

We adopted a fatigue testing scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28]. Clinically there has been seen with this kind of restorations high incidence of chipping and delamination [29]. Thus, in this scenario, recommended by ISO, that successfully resulted on the desired pattern of failure, that was the crack of the veneer without complete fracture of the set. Besides this testing assembly has already been at prior used by published articles of similar thematic [27,28].

It is important to highlight that after firing completion, starts the cooling of the restoration. Such step may be executed rapidly or under a slow cooling rate. A rapid cooling rate may potentiate negative effects at strength by inducing residual stress concentration [30]. For such reasons, a slow cooling rate was adopted, where the furnace was only opened at temperatures below 200°C. So, the influence of different cooling rates on the evaluated outcomes should be further considered on future studies.

In addition, our results show relevant esthetic differences according to the quantity of specimens in each firing cycle. There was a clinically
unacceptable color difference ($\Delta E_{00} > 1.8$) between the specimens of the G1 and G5C groups. Hammad and Stein [31] evaluated the effect of the increase in firing temperature on the hue, chroma and luminosity of metal-ceramics, and observed that the increase in furnace heat led to higher hue values and lower luminosity. Considering that the heat generated by the furnace is distributed among the specimens [19], at the G1 group the specimens absorbed all the heat inside the furnace, while those from the G5 group divided the energy among them, which probably caused the differences in terms of vitreous and crystalline phase sintering, besides of density and porosity of the material [22,24], generating different light interaction and consequently the observed color difference.

The specimens’ position inside the furnace had no influence on the fatigue strength, the number of cycles for failure, survival probability or translucency. However, it caused a color difference in a clinically perceptible and unacceptable way ($\Delta E_{00} > 1.8$). According to the location of the furnace resistance, which is coupled to the lateral walls of the internal chamber, the first heating occurs in proximity to where the periphery specimens are located (G5P). According to the concepts of thermodynamic [19,32], in an attempt to compensate this effect and to allow a similar amount of heating energy between periphery and center of the furnace, a convection current is created and the energy is transferred from the greater heating zone (periphery) to the lesser heating zone (center). Nevertheless, $L^*a^*b^*$ measurements showed that specimens from the periphery became lighter and slightly less reddish than the central ones, which resulted in a clinically perceptible color difference (Table 2). Previous studies have reported instability of certain porcelain metal oxides after thermal treatments, which led to significant color changes [33,34]. Metal oxides are added to the ceramic to obtain the appropriate color shades. The heterogeneous heat distribution inside the furnace probably affected the porcelain metal oxides of each specimen in a different way, resulting in a relevant color difference. Regarding the translucency, the absence of differences between the groups probably occurred due to the zirconia being the core material and its consequently opaque nature [35]. Thus, even in a thin layer (0.7 mm), the zirconia core seems to have masked any difference in the translucency of the ceramic veneer, so it was the reason why differences in the veneering ceramic translucency were not observed, despite the differences in luminosity (Table 2), in accordance with Bacchi et al. [36].

As an in vitro study, there are some limitations that must be considered. The outcomes tested in regards of the performance of bilayer specimens are directly related to the geometry and specifications of the furnace used (chamber size and position of the heating resistance), our data clearly encourage that new studies evaluate and characterize the effect of such parameters on the aforementioned outcomes. Additionally, each restoration in clinical practice is composed of different colors used during the layering technique, for esthetic improvements, so this interaction of shades could be affected differently from the present study, which evaluated only one porcelain shade. Finally, it is crucial to mention that the porcelain veneered Y-TZP restorations might assume a distinct fatigue behavior when the restorations are luted on a substrate, since the stress distribution on the assembly may differ from the biaxial set-up applied in this study [37-39].

5. Conclusion

- The quantity of bilayer specimens in the furnace for porcelain firing affected the biaxial fatigue strength, i.e. only one specimen should be sintered rather than five for fatigue behavior improvements. Moreover, the color difference caused by the quantity of specimens was clinically significant, but translucency did not seem to be affected.
- The specimen’s position in the furnace during firing had no effect on fatigue strength and translucency. However, the color difference between the specimens was clinically significant.

Conflict of Interest Statement

The authors deny the presence of any conflict of interest.
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