Influence of number and position of porcelain specimens in the furnace on flexural strength and translucency


Aim: The aim of this study was to investigate the influence of the quantity and positioning of feldspathic ceramic specimens inside the furnace on their flexural strength and translucency. The tested hypotheses were that the arrangement of specimens in the furnace would not influence 1) the translucency or 2) the biaxial strength of the porcelain.

Methods: Ninety porcelain specimens were made (1.2 mm thickness and 13.5 mm diameter) and assigned into two main groups (n=15): G1 group - 15 firing cycles containing only one specimen each, always at the center of the refractory; and G5 group - 15 firing cycles containing five specimen each, where one specimen was at the center of the refractory and four specimens positioned equidistantly on the periphery. The translucency test was performed using a spectrophotometer, followed by the flexural strength test, according to ISO 6872:2015. T-student test was performed for both the mechanical and optical obtained data.

Results: The flexural strength of the porcelain was not affected by the positioning (center x periphery) of the specimens inside the furnace (p =0.08), but the translucency was affected (periphery > center; p =0.009). Regarding to the number of feldspathic ceramic specimens, the biaxial flexural strength was affected (p =0.025), as well as the translucency (p <0.05). Conclusion: A higher quantity of feldspathic ceramic specimens for each firing cycle decreased its biaxial flexural strength and translucency. Also, specimens positioned at the center of the refractory became less translucent than those positioned at the periphery.

Keywords: Dental porcelain. Flexural strength. Optical phenomena.
Introduction

Nowadays, the aesthetic demand of patients is a major concern during the clinical practice. Although metal-ceramic prostheses present satisfactory clinical results and longevity\(^1\), they also present an aesthetic limitation due to its opacity and the metal grayish shade. In this sense, all-ceramic restorations appear as a good option due to their biocompatibility, excellent aesthetic and mechanical properties, like chemical durability, adequate resistance after bonded and low degradation\(^2-5\). Among the options of ceramic materials, feldspathic ceramic is widely used in metal-free restorations as veneering material for bilayer systems and veneers\(^6\).

When applied through sequential increments, the porcelain requires several firings to achieve the desired thickness and coloration. According to Tang et al.\(^7\) (2012), several firings could improve hardness, density, and decrease the porosity of ceramics used in veneering zirconia restorations. On the other hand, the number of firings, material type and porcelain thickness are factors that can influence optical properties and color changes on dental ceramics\(^8,9\). According to Vanini\(^10\) (2011), the color of an indirect ceramic restoration is influenced by lightness (which includes opacity and translucency), chroma (intensity), and hue (the color itself); moreover, literature has shown that translucency and light interaction are also important aspects on color perception of ceramic restorations\(^2,11\).

From the laboratory standpoint and as already known, many ceramic restorations can be simultaneously introduced into the furnace at different positions for the same firing cycle in order to save time due the exponential growth of aesthetic and restorative demand in dental clinics, which consequently increases the workflow for the prosthetic laboratories. However, the effects of these procedures on ceramic properties, mainly in terms of mechanical and optical behavior, are not clear in the literature.

Therefore, the aim of this study was to evaluate if the flexural strength and translucency of a porcelain are affected by the specimen position in the furnace (center or periphery), or by the number of specimens in each firing cycle. The tested hypotheses were that the positioning and the quantity of specimens in the furnace would not affect 1) the translucency or 2) the biaxial strength of the feldspathic ceramic.

Material and Methods

Preparation of specimens

Ninety (90) feldspathic ceramic discs (VITA VM9, 3M3 shade, VITA Zahnfabrik, Bad Sackingen, Germany) were manufactured according to ISO 6872:2015\(^12\). The porcelain was manipulated by mixing the ceramic powder with the modeling liquid (VITA VM Modelling liquid, VITA Zahnfabrik) in standardized proportions (1/1) until a slurry solution was obtained. This slurry was condensed in metal templates with a thickness of 1.8 mm by the layering technique using a metal spatula until the complete thickness of the templates was reached. For each applied layer of ceramic inside the template, the excess of liquid was removed with a pressed absorbent tissue (softy’s, Elite, Brazil). After complete filling and excess liquid removal, the porcelain
was removed from the template, and the specimens were placed on a refractory to the firing cycles in the Vacumat 6000MP furnace (VITA, Zahnfabrik Bad Sackingen, Germany), according to the manufacturer recommendations (Table 1). A prolonged slow cooling of 600 °C was used until the achievement of 200ºC, when the furnace finally opened.

Table 1. Temperature oscillations of the firing cycle in the “first dentin firing” mode.

<table>
<thead>
<tr>
<th>Pre-Drying °C</th>
<th>Min.</th>
<th>Temp. approx. °C</th>
<th>Min.</th>
<th>Vac min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>6.00</td>
<td>55</td>
<td>910</td>
<td>1.00</td>
</tr>
</tbody>
</table>

Porcelain firing (Coefficient of thermal expansion (CET): 9.0–9.2 \(10^{-6} \text{ K}^{-1}\)) was performed as follows:

- G1 group: 15 specimens, where only one specimen was placed inside the furnace for each firing, thus 15 firing cycles containing only one specimen were performed (n=15).
- G5 group: 75 specimens, where 5 specimens were placed inside the furnace simultaneously, thus 15 firing cycles containing five specimens for each firing cycle were performed (n=15).

The distribution of the samples inside the furnace was standardized according to each group. For G1, each specimen was placed in the center of the furnace, while for G5 one specimen was placed in the center (G5 center) and the other four discs were positioned equidistantly on the periphery (G5 periphery), through markings on the base of the furnace as reference points (Fig. 1).

![Figure 1. Disposition of the specimens inside the furnace for each sintering cycle. The G1 group, with one specimen placed in the center of the furnace (left); G5 group, with one specimen placed in the center (G5 center) and the other four positioned equidistantly on the periphery (G5 periphery) (Right).](image)

After firing, the specimens were ground and polished with silica carbide (SiC) papers (#200, #400, #600 and #1200; 3M, Sumare, Brazil) under constant water coolant irri-
In a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, USA) to standardize the specimen dimensions and surfaces (final diameter of 15 mm and thickness of 1.2 mm, according to ISO 6872). All specimens were inspected under an optical microscope (Stereo Discovery V20; Carl Zeiss, Göttingen, Germany) to verify the presence of porosities or irregularities on their surface, if so the specimen was discarded and replaced by a new one, according to the groups of firing.

Translucency evaluation

A spectrophotometer (SP60, X-Rite, Grand Rapid, USA) was used to measure CIE L* (lightness) parameter (Commission Internationale de l’Eclairage). Measurements were carried out over a white (L*=92.95, a*=-0.78, b*=3.57) and a black background (L*=27.94, a*=-0.01, b*=0.03) (Leneta Card, model 12H, Cor & Aparência) under the followed parameters: aperture setting 8 mm; illumination 12 mm; and measuring time of 2 seconds. The spectrophotometer was recalibrated every 20 measurements to avoid relevant discrepancies between evaluations. A coupling solution (glycerol C3H8O3/Vetec Química Fina Ltda.) was used to minimize light scattering between the specimen and the standard card. Each specimen was positioned into the spectrophotometer chamber, that inhibited the influence of the ambient illumination, and measured three times by a single trained operator (P.S.M), and the mean values of L*, a* and b* over the white and the black backgrounds were used in Contrast Ratio (CR) equations (1) (2):

\[ CR = \frac{Y_b}{Y_w} \]  
\[ Y = \left(\frac{L+16}{116}\right)^{\frac{3}{2}} \times 100 \]

in which \( Y_b \) denotes the reflectance over the black background, and \( Y_w \) denotes the reflectance over the white background. In these calculations CR=0 is considered the most translucent, and CR=1 is the opaquest material, as described by Liu et al. (2010).

In this study, we considered the clinical threshold (CR=0.06) of CR for ceramic materials.

Biaxial flexural strength test

The biaxial flexural strength test was performed in a universal testing machine (EMIC DL 2000, São Jose dos Pinhais, Brazil), according to ISO 6872:2015. An adhesive tape was glued to the disc on the compression side before the test to avoid spreading the fragments and for better contact between the piston and the disc; a cellophane paper (2.50 μm) was placed between the three support balls of the base and the specimen avoid contact damage and improve the load distribution. A flat circular tungsten piston (Ø=1.6 mm) was used to apply an increasing load of 1 mm/min until the fracture. The tests were performed under immersion of distilled water.

The test machine recorded the load for failure in newtons (N). After data collection, biaxial strength values were calculated using equations 3, 4, and 5:
\[ \sigma = \frac{-0.2387P (X-Y)}{b^2} \]  \hspace{1cm} \text{Eq. (3)}

\[ X = (1 + v) \ln \left( \frac{r_2}{r_3} \right) + \left[ \frac{1 - v}{2} \right] \left( \frac{r_2}{r_3} \right)^2 \]  \hspace{1cm} \text{Eq. (4)}

\[ Y = (1 + v) \left[ 1 + \ln \left( \frac{r_1}{r_3} \right)^2 \right] + (1 - v) \left( \frac{r_1}{r_3} \right)^2 \]  \hspace{1cm} \text{Eq. (5)}

where “\( \sigma \)” is the maximum tensile stress (MPa), P is the load used until fracture (N), “b” is the thickness at the fracture point of origin (mm), “v” is the Poisson coefficient (0.21-ratio that relates to the nature and symmetry of the interatomic bonding forces of the specimens), \( r_1 \) is the radius of the support circle (5 mm), \( r_2 \) is the radius of loaded area (0.8 mm) and \( r_3 \) is the radius of the specimen (mm).

**Statistical analysis**

Data of strength and contrast ratio were subjected to Shapiro Wilk and Levene tests to verify their normality and homoscedasticity, respectively. Student t-test was performed to analyze the influence of the position and the quantity of specimens in the furnace on biaxial strength and constrast ratio (significance level was set as 5%). The comparisons were performed as follows:

- Position: in order to verify if the position (center x periphery) of the specimen influences the biaxial strength and the CR, a mean of the four specimens from the periphery (G5) was calculated for each firing. This value was compared to the center specimen value of that same firing;

- Quantity: the center specimens from G5 were compared with the G1 specimens, in order to compare specimens fired at the same position in the furnace (center), however with one group containing five specimens in the furnace and another with only one.

Considering that failure in ceramic materials originates from internal defects, the size and distribution of those defects justify the need for a statistical approach for structural reliability. Thus, the Weibull statistical analysis was performed\(^{16}\), as a way to describe the strength variation by obtaining the Weibull modulus (m) determined in a diagram\(^{17}\):

\[ \ln \ln \left( \frac{1}{1-F} \right) = m \ln \sigma_0 - m \ln \sigma \]  \hspace{1cm} \text{Eq. (6)}

where F is the failure probability, \( \sigma \) the initial strength, \( \sigma_0 \) the characteristic strength, and m is the Weibull modulus. The characteristic strength is considered to be the strength at a failure probability of approximately 63%, and the Weibull modulus is used as a measure of the distribution of strengths, expressing the mechanical structural reliability of the material.

**Fractographic analysis**

All the fractured specimens were cleaned and then analyzed under a light microscope (Stereo Discovery V20, Carl Zeiss, Gottingen, Germany) to determine the location of
the fracture origin. Representative specimens of G1 and G5 (center and periphery) groups were selected and sputter-coated with platinum. These samples were then analyzed by field emission Scanning Electron Microscopy (SEM-Vega3, Tescan, Czech Republic) under high-vacuum with 20.00 kV at a working distance of approximately 13.5 mm, in order to investigate the influence of the specimen’s arrangement for sintering on the fracture origin pattern. The fractured surfaces were qualitatively analyzed under 200 ×, 700 × and 7000 × magnification.

Results

The CR and Flexural Strength data were normal and homogeneous. Means and standard deviations of all experimental groups are described in Table 2.

In terms of ‘positioning effect’, no significant difference was found between periphery and center specimens for biaxial strength (p=0.082); however, the position was statistically significant for the CR (p<0.009), since periphery specimens were more translucent than the center specimens, and this difference was 0.07 which is clinically relevant according to the adopted threshold\(^\text{14}\).

Taking into consideration the ‘quantity effect,’ the number of specimens influenced the biaxial strength values (p=0.025) and the CR (p<0.05); a higher number of specimens inside the furnace during firing showed lower strength and less translucency when compared with specimen fired alone inside the furnace. However, the translucency was not clinically relevant, since the difference between the mean CR values of the two groups was 0.03\(^\text{14}\).

Scanning electron microscopy images evidenced that the fracture of all tested specimens originated from the middle of the bottom surface, where tensile stress concentration took place. In addition, all specimens presented similar fracture parameters caused by critical defects present near the surface. Thus, the location of the failure origin occurred in a subsurface defect (Fig. 2). In terms of reliability, the Weibull’s modulus showed no significant difference among the study groups (Table 2).

Table 2. Means (Standard Deviation) of biaxial strength (MPa), contrast ratio data, Weibull’s moduli (confidence intervals).

<table>
<thead>
<tr>
<th>Tests</th>
<th>Specimen’s position</th>
<th>Specimen’s quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>G5 Center</td>
<td>G5 Periphery</td>
</tr>
<tr>
<td>Biaxial Strenght (MPa)</td>
<td>52.52 (5.16)(^A)</td>
<td>55.58 (3.69)(^A)</td>
</tr>
<tr>
<td>Contrast Ratio</td>
<td>0.91 (0.03)(^A)</td>
<td>0.84 (0.03)(^\ast)</td>
</tr>
<tr>
<td>Weibull’s Moduli</td>
<td>11.7 (6.7 – 16.3)(^A)</td>
<td>17.33 (10 - 24.2)(^A)</td>
</tr>
</tbody>
</table>

Distinct letters show significant statistical differences (p < 0.05).

*Clinically significant differences (threshold for perceptibility of contrast ratio = 0.06; Liu et al., 2010).
Figure 2. Scanning electron microscopy (SEM) of specimens selected from groups G1, center of G5 and periphery of G5 analyzed at 200, 700 and 7000 × magnification, depicting similar failure pattern for all groups. The dotted semicircles show the failure origin area, which started on the bottom surface (tensile side), while the black arrows show the direction of the crack propagation (DCP). The filled white arrows show the aspect of the Wake Hackles, which start from an internal defect in the material and project in the form of whitish lines, also showing the DCP.

Discussion

In the translucency analysis, both the position and the quantity of specimens in the furnace generated statistically significant differences; therefore, the first tested hypothesis was rejected. Feldspathic ceramic is composed mainly by vitreous phase, with silica as its main matrix content (55-65%), which is responsible for the translucency and aesthetic of the porcelain. Previous studies reported that multiples firings generate microstructural differences in terms of vitreous and crystalline phase sintering of ceramics, besides of density and porosity of the material. Moreover, even the firing temperature may affect the translucency ceramic materials.

Gonuldas et al. (2014) evaluated the effect of several firings on the color changes in ceramic specimens, and observed an increase in ∆L* as the number of firings increased, indicating the effect of more heat energy. As can be observed in Contrast Ratio equations (1) (2), L* is related to a material’s translucency. Following this line,
since the present study is limited to only one firing cycle, the interaction between the
heat energy of the furnace and the ceramic properties was dictated by the organi-
zation of the specimens inside the refractory. Due to the proximity of the periphery
specimens of the G5 group to the resistance of the furnace, attached to the lateral
walls, the heat energy that reached these specimens was more direct and intense
than for the center ones, which is corroborated by the thermodynamics concepts22.
Such differences may have affected the sintering level of the vitreous phase of the
porcelain, thus modifying the microstructure arrangement and consequently the light
interaction, increasing its translucency.

In relation to the quantity of specimens during firing, there also significant differ-
ences between G1 (which was more translucent) and the center specimens of
G5. However, the difference between the mean CR values of the two groups was
just 0.03, which despite being statistically significant, is not clinically perceptible
according to the adopted threshold (0.06)14. Thus, the heat energy present in the
furnace for one or five specimen during firing may be enough for a satisfactory
contrast ratio.

The results of our study only indicate that the positioning of the specimens in the
sintering furnace did not affect its biaxial strength, so the second hypothesis was
partially accepted. The literature is scarce regarding positioning and the quantity of
dental ceramics inside the furnace for each firing cycle in relation to biaxial strength.
According to thermodynamics concepts22, since there is no direct contact between
resistance and specimens, the energy in the form of heat is transferred to the speci-
mens by radiation inside the furnace. Although the furnace’s resistance closer to the
peripheral specimens of G5, the distance between center and periphery does not
seem to be enough different to generate more relevant porosities and critical defects
along the sintered microstructure, which are important aspects for the strength of the
material23,24. Thus, a similar mechanical performance was observed between central
and peripheral porcelain specimens after firing.

On the other hand, the quantity of specimens per firing cycle affected the porce-
lain biaxial strength, since the larger the number of specimens, the lower was their
strength. This implies that there may be a division in the amount of heat from the
furnace between specimens present in the refractory, where the G1 group only had
one specimen and it absorbed all the heat transmitted from the furnace to itself,
which may have generated less porosities and defects. In this sense, Aurélio et al.25
(2015) showed a significant increase in the flexural strength values of a leucite-re-
inforced glass ceramic when the glaze firing time was extended to 15 min instead
of 1.5 min. In addition, according to Ban et al.26 (2013), without the presence of
contaminating factors, the increase of the furnace temperature led to an increase
in the flexural strength of a polycrystalline tetragonal zirconia (Y-TZP). Although
the current study investigated a feldspathic ceramic, it is hypothesized that the
greater the amount of heat transmitted to a ceramic, the greater its flexural strength.
Thus, at G5 group the heat energy was divided by 5 specimens, decreasing the
mechanical strength.

Even though there were discrepancies of biaxial strength between groups G1 and G5,
there were no changes in the fracture pattern between the analyzed specimens. All
failures originated from the bottom surface of each specimen, where the tensile stress took place during the test\textsuperscript{15}. The failure started from a critical subsuperficial defect, where the stress concentrated and the crack propagated until catastrophic failure. The direction of crack propagation was represented by the wake hackles present along the microstructure of the tested specimen (Fig. 2). Also, there were no differences among Weibull modulus, despite the significant differences in strength, according to Weibull parameters\textsuperscript{16}. This demonstrates that the position and quantity of specimens promoted equal structural mechanical reliability in the material (Table 2).

The interaction between porcelain firing conditions in a single cycle, biaxial strength and translucency evaluated in this study is an approach that has not been explored in the literature. The differences in flexural strength and the translucency between the groups have a major impact on the patient’s greatest desires when receiving new fixed prosthesis: longevity and aesthetics. In addition, the obtained results were standardized, as the standard deviation of the findings was small. Therefore, the firing process of feldspathic ceramics must be considered and a standard protocol defined, maintaining all crowns close to the heat source for each firing.

On the other hand, there are some limitations such as the manual layering technique used to make each specimen; the use of powder and liquid may have incorporated internal flaws and bubbles\textsuperscript{27}, which may have influenced the results\textsuperscript{23}, however it is an inherent limitation of the layering technique. Besides that, more studies are necessary using thermocouples to verify the real temperature of the specimens during sintering, and with other types of furnaces and dental ceramics, such as feldspathic reinforced by leucite, lithium disilicate, zirconia and bilayered samples. Thus, it might be possible to corroborate the findings on the relationship between sintering conditions and flexural strength/translucency of the ceramics.

**Conclusion**

So, with the limitations of the present study, we conclude that:

1. the specimen’s position inside the furnace led to a clinically significant difference in translucency of the feldspathic ceramic. However, the translucency difference caused by the quantity of specimens seems to be clinically insignificant.

2. the specimen’s position inside the furnace during firing did not have any effect on biaxial strength of the feldspathic ceramic. However, the quantity of specimens in the furnace may affect the ceramic strength, since the specimens sintered alone presented greater strength values.

**Conflicts of interest**

None.

**Data availability**

This study was part of the fulfilment for the requirements of the Dentist degree (P.S.M) at the Faculty of Dentistry, Federal University of Santa Maria (UFSM)-RS, Brazil.
Datasets related to this article can be found at the Link: https://repositorio.ufsm.br/handle/1/2512?locale-attribute=en, hosted at UFSM.

Acknowledgements

We thank Luís Felipe Guilardi for his time and availability in helping with the fractographic analysis. This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Authors Contribution

Pablo Soares Machado - Conceptualization, data curation, formal analysis, methodology, writing – review & editing. Jeanni Gonçalves Camponogara - Conceptualization, data curation, methodology, writing – original draft. Camila da Silva Rodrigues - Formal analysis, methodology, writing – review & editing. Leticia Borges Jacques - Formal analysis, writing – review & editing. Luiz Felipe Valandro - Conceptualization, supervision, formal analysis, writing – review & editing. Marilia Pivetta Rippe - Conceptualization, supervision, formal analysis, data curation, writing – review & editing.

All authors actively participated in the manuscript’s findings, revised, and approved the final version of the manuscript.

References


