RELATÓRIO DE ATIVIDADES

1 – DADOS CADASTRAIS

1.1 Nome do Beneficiário	1.2 CPF / Passaporte
GABRIEL KALIL ROCHA PEREIRA	**006.656.640-10/GA570010**
1.3 Instituição	1.4 Programa CAPES/ nº do AUXPE
UFSM	**GERAL**
1.5 Projeto	1.6 Coordenador Projeto
GERAL	**PAULO RENATO SCHNEIDER**
1.7 Programa de Pós-Graduação	
PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS ODONTOLÓGICAS	

2 – BENEFÍCIO

2.1 Modalidade		
(X) Bolsa **CAPACITAÇÃO EM CURSOS DE CURTA DURAÇÃO**		
2.2 Instituição de Destino (nome da instituição e nome do centro/instituto/departamento/grupo de pesquisa)	2.3 Período da Atividade	
	2.3.1 Início	2.3.2 Término
DEPT. OF DENTAL MATERIALS SCIENCE, ACADEMISCH CENTRUM TANDHEELKUNDE AMSTERDAM, UNIVERSITY OF AMSTERDAM AND VRIJE UNIVERSITY AMSTERDAM (THE NETHERLANDS)	01/02/2020	29/02/2020

3 – RECURSOS RECEBIDOS (R$)

3.1 Auxílio-deslocamento	PAGAS PELA CAPES
3.2 Auxílio-instalação	€1300,00
3.3 Seguro-saúde	€90,00
3.4 Adicional-localidade	€400,00
3.5 Mensalidade	€1300,00

4 – DESCRIÇÃO DAS ATIVIDADES

4.1 Objetivos da missão:

A presente missão se enquadrou no contexto do projeto Materiais Inteligentes/*Smart Materials*. Em suma essa proposta de curso de capacitação objetivou no treinamento em ensaios de fadiga com base em diferentes metodologias de ensaio, na execução de ensaios e na posterior discussão dos dados obtidos para a escrita de artigos neste contexto. Ademais, também foi objetivo da proposta o treinamento em análises topográficas dos materiais envolvidos e análises fractográficas para caracterização do padrão de falha observado durante os ensaios. Portanto, três pilares de metas podem ser destacados na presente proposta:

1- a qualificação de recursos humanos pela participação do docente de graduação e pós-graduação promovendo aperfeiçoamento da formação acadêmica, amadurecimento científico e tecnológico em área de vital importância no contexto do trabalho desenvolvido pelo Programa de Pós-Graduação em Ciências Odontológicas da UFSM;

2- o desenvolvimento e propagação de conhecimento científico pela obtenção de evidência científica no contexto explorado e posterior inserção nas práticas diárias de atuação do docente na instituição; e

3- o fortalecimento da cooperação interinstitucional (Universidade Federal de Santa Maria e Academic Centre for Dentistry Amsterdam), pela aproximação entre pesquisadores e instituições copartícipes.

4.2 Atividades Realizadas:

Todas as atividades previstas no plano de atividades previamente estabelecido foram cumpridas em sua plenitude. Nesse sentido foram realizadas:

- discussões da temática que convergiram principalmente para a submissão de artigos (expostos na seção 4.3 abaixo, e em anexo neste relatório).

-participação como ouvinte em defesa de doutorado, em 14 fevereiro de 2020, intitulada “The protective effect of topical fluoride treatments in dentine lesions”, defendida pela Ms. Marwa Alhothali.

- treinamento na obtenção de amostras para ensaios (Apêndice).

- ensaios de fadiga objetivando no desenvolvimento de uma nova metodologia de ensaio para acelerar caracterização de materiais em flexão de 3 pontos (Apêndice).
- treinamento na análise topográfica de fraturas (Apêndice).
- discussão de novos contextos a serem explorados no futuro, em decorrer do fortalecimento das atividades interinstitucionais.

Diversos tópicos foram discutidos no contexto de desenvolvimento de ensaios de fadiga, na busca de novos métodos e de delineamentos que permitam a aprofundização na temática que têm sido desenvolvida pelos grupos de pesquisa. Ademais, salienta-se que dentre o grande rol de infraestrutura e de conhecimento dos profissionais da ACTA destaca-se o conhecimento na predição de comportamento de materiais restauradores e dentários em relação a análises de abrasão, onde enfatiza-se que a instituição possui uma máquina que fora totalmente desenvolvida por estes e validada para predição de performance clínica. Desta forma, foram discutidos aspectos para que pesquisas nesse sentido também possam vir a ser desenvolvidas, e desta forma, prevê-se o fortalecimento dessa linha de pesquisa em um futuro próximo.

4.3 Resultados e/ou Impactos:

Artigo publicado (submetido ao longo da missão):

1- Kiara Serafini Dapieve, Renan Vaz Machry, Rafaela Oliveira Pilecco, Cornelis Johannes Kleverlaan, Gabriel Kalil Rocha Pereira, Andressa Borin Venturini, Luiz Felipe Valandro. One-step ceramic primer as surface conditioner: effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations. Journal of the Mechanical Behavior of Biomedical Materials; Volume 104, April 2020, 103686. https://doi.org/10.1016/j.jmbbm.2020.103686. Revista de Qualis A1; FI 3.485.

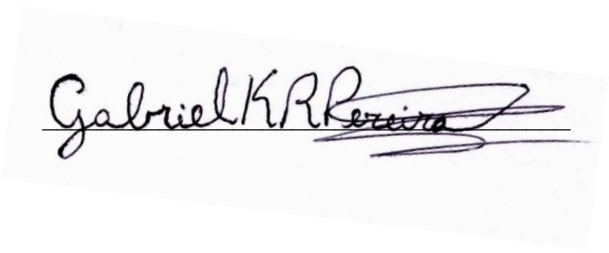
Artigos submetidos (Anexo):

1- Effect of grinding, polishing and glazing on the optical and surface characteristics of a lithium disilicate glass-ceramic. Felipe Brescansin, Catina Prochnow, Atais Bacchi, Cornelis Johannes Kleverlaan, Luiz Felipe Valandro , Gabriel Kalil Rocha Pereira. Artigo submetido para o periódico *Journal of Prosthodontic Research* (Qualis A1; FI 2.636).

2- Influence of testing environment at the fatigue performance of lithium disilicate ceramics and yttria stabilized zirconia. Sara Fraga; Luís Felipe Guilardi, Liliana Gressler May, Luiz Felipe Valandro, Cornelis Johannes Kleverlaan, Gabriel Kalil Rocha Pereira. Artigo submetido para o periódico *Journal of the Mechanical Behavior of Biomedical Materials* (Qualis A1; FI 3.485).

Compactuamos com a ideia essencial de que uma colaboração científica é justificada quando existem diferentes competências que podem ser complementadas com o propósito de qualificar o conhecimento científico e seus produtos (artigos científicos, desenvolvimento de novos materiais e patentes), assim como quando existem anseios mútuos de ampliação de relações que tenham repercussões fundamentalmente na formação de recursos humanos mais qualificados e impacto institucional. Com esse pensamento, entendemos que a presente proposta tem tido e prospecta-se que ainda terá forte impacto exatamente nesse 2 âmbitos fundamentais. Ademais, a interação e troca de experiências dos grupos de pesquisa têm qualificado o conhecimento científico dos temas de pesquisa envolvidos, e por consequência têm se proporcionado uma contribuição mais consistente para a literatura científica. Os docentes-pesquisadores da instituição holandesa apresentam muita experiência na caracterização de materiais, representada pelo histórico de produção intelectual qualificada, fruto de conhecimento acumulado e estrutura física qualificada e a interação com esta instituição têm se mostrado profícua e pretende ser ainda intensificada no futuro.

Santa Maria, 03 de março de 2020.



ANEXOS

1- Artigo 1

Artigo submetido para o periódico *Journal of Prosthodontic Research* (Qualis A1; FI 2.636).

Original research article

Effect of grinding, polishing and glazing on the optical and surface characteristics of a lithium disilicate glass-ceramic

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Conflict of Interest Statement: The authors deny the presence of any conflict of interest.

ACKNOWLEDGEMENTS

This manuscript is based on the final graduation work of F.B. to obtain the title of DDS under the guidance of G.K.R.P. The authors state that they did not have any conflicts of interests. In addition, they gratefully thank Ivoclar Vivadent for donating the ceramic materials and the *CAPES/NUFFIC* Program (*CAPES* – Agency for the High-Standard Promotion of Graduate Courses, Brazil; *NUFFIC* – Netherlands Organization for International Cooperation in Higher Education, The Netherlands) for the support (Grants CAPES #056/14).

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Felipe Nascimento Brescansin (brescansin.felipe3@gmail.com), contributed on: Experimental procedures; Surface characteristic analysis, Optical measurements, Discussion of data, Writing and final review of the manuscript.

Catina Prochnow (catinaprochnow@hotmail.com), contributed on: Optical measurements, Final review of the manuscript.

Cornelis Johannes Kleverlaan (c.kleverlaan@acta.nl), contributed on: Designing the study, Discussing the data, Final review of the manuscript.

Ataís Bacchi (atais_bacchi@yahoo.com.br), contributed on: Statistical analysis, Discussing the data, writing and final review of the manuscript.

Luiz Felipe Valandro (valandrolf@gmail.com), contributed on: Designing the study, Discussing the data, Final review of the manuscript.

Gabriel Kalil Rocha Pereira (gabrielkrpereira@hotmail.com), contributed on: Designing the study, Experimental procedures, Surface characteristic analysis, Optical measurements, Statistical analysis, Discussing the data, Writing and final review of the manuscript.

Short title: Optical and surface alterations triggered by surface treatments of glass-ceramic

Number of Pages: 18, Tables: 3; Figures: 4. Quantity of Reprints: 0.

Abstract

Purpose: To evaluate the effect of adjustments (grinding with diamond burs) and posterior processing treatments on the optical and surface characteristics of a lithium disilicate glass-ceramic.

Methods: Ceramic specimens were randomly allocated into 6 groups (n=5) according to their surface treatment: ***mirror-polished*** (baseline): surfaces polished on both sides with SiC papers; ***as-cut***: mimicking a CAD/CAM milled rough surface; ***ground***: grinding with medium-grit diamond bur (90-120μm); ***ground polished***: after grinding, finished with fine (46μm) and extra-fine (30μm) diamond burs, polished with diamond cups and a nylon brush with diamond paste; ***ground glazed***: glazed after grinding; **ground polished glazed**: association of the three aforementioned methods. A spectrophotometer was used over 4 backgrounds (black, white, gray and shaded A2) to estimate the translucencies (TP_{00}) and color changes (ΔE_{00}), CIEDE2000. Light transmittance and irradiance were assessed with a colorimeter considering 3 light wavelengths (blue: 440 to 485nm; green: 500 to 565nm; and red: 625 to 740nm). Surface characteristic were analysed through Scanning Electron Microscopy and using a profilometer considering *Ra* and *Rz*.

Results: Grinding harmfully affected the optical properties (TP_{00}, ΔE_{00}, and light transmittance) and the surface characteristics (topography and roughness), regardless of background color. Only application of a glaze material was not able to reestablish the deleterious impact on optical characteristics, despite achieving topographical features comparable to the baseline.

Conclusions: Grinding harmfully affects the optical and surface characteristics of a lithium disilicate ceramic; polishing after grinding is mandatory to revert the deleterious influence on optical outcomes; glaze is advocated to enhance topographical characteristics.

Keywords: Translucency; Color change; Light transmittance/irradiance; Topography; Roughness.

1. Introduction

Monolithic glass-ceramic restorations have been extensively indicated nowadays [1-3]. The main reason for this is the attempt to achieve restorations which unify high aesthetic and superior mechanical properties [4,5]. Monolithic restorations enable using a monolayer material which contributes to reducing the risk of failure due to incongruent layers (e.g. chipping). In addition, it reduces tooth preparations (the thickness depends on the employed material, i.e. the microstructural characteristics and intrinsic properties) in accordance with actual minimum invasive restorative concepts [1-3].

Monolithic restorations are not a new restorative concept [6], but based on enhancements/advances in the processing techniques in recent years, especially regarding CAD/CAM systems (Computer-Aided Design/Computer-Aided Manufacturing), their application has been re-explored [1]. Miyazaki et al. [7] states that CAD/CAM milling permits a substantial reduction in working time together with higher precision in the final restoration. In addition to these aspects, it also results in better mechanical properties for ceramic restorations [8] since CAD/CAM technology enables producing materials with less intrinsic defects, reducing the crack propagation and failures in restorations when submitted to tensile forces [9-11].

The use of lithium disilicate glass-ceramic can be highlighted among available restorative materials. This material has been widely used for manufacturing indirect restorations [2,3,12], depicting a 5-years survival rate of 96.6% for single crowns [13] and presenting superior mechanical properties, adhesive predictability (higher bond strength and bond stability) and optical properties in comparison to monolithic zirconia [14-16].

Despite all the advancements regarding the accuracy and precision of CAD/CAM tools, adjustments are still commonly necessary (grinding, finishing and polishing procedures) to enhance the emergence profile and occlusal/proximal relations of restorations [17-19]. These adjustments inherently influence the surface characteristics of ceramics, and in doing so they may potentially change the optical and mechanical properties of these materials [21-23]. From this viewpoint, the literature has shown that these procedures may introduce different types of damage (defects) into the material's surface, creating different roughness and surface topography patterns [18,23,24].

It is well-known that an increase in roughness inherently affects the light transmittance through a restorative material [22,25,26]. Thus, when light is transmitted through a roughened surface, the direction and incidence of light is altered, consequently increasing the material's opacity, further altering other optical characteristics. Therefore, the restorative surface should be subjected to subsequent processing treatments after clinical adjustments with diamond burs for smoothening and restoring the optical properties. However, scarce information exists on this theme to guide clinicians' choice.

Thus, this study aimed to evaluate and compare the surface characteristics (topography, roughness) and the optical characteristics (translucency, light transmittance and color variation ΔE) of a lithium disilicate glass-ceramic after different surface treatments which mimic clinical adjustment and subsequent processing conditions. The assumed study hypothesis is that increased roughness will deleteriously impact the optical and surface characteristics of the ceramic.

2. Material and Methods

2.1. Specimen preparation

Thirty (30) disc-shaped ceramic specimens (final dimensions: 10 mm diameter and 1.5 mm thickness) were obtained from a lithium disilicate glass ceramic (IPS e.max CAD HT/A2 B40, Ref #634587, Lot #S04802, Ivoclar Vivadent, Schaan, Liechtenstein), using a similar methodology previously described by Pereira et al. [27]. To do so, the blocks were first removed from the metallic holder and then two metal cylinders (10 mm in diameter and 2 mm in thickness) were glued to each block side to serve as a reference for shaping the blocks into cylinders by manually grinding their lateral surfaces on a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA), first with diamond grinding discs (Dia-Grid Diamond Discs #120 – average grit size 160 mm, Allied High Tech Products, Inc./Rancho Dominguez, CA, USA), and then silica carbide (SiC) papers (600-2000 grit).

Finally, the former metallic holders were again glued to the ceramic cylinders and slices were produced on a cutting precision machine (ISOMET 1000, Buehler) with 2 mm thickness to guarantee safe dimensions for executing the surface treatments and still standardize the final ceramic thickness at 1.5 mm (minimum thickness recommended by manufacturer on the occlusal surface), and always inspected with a digital micrometer (Quantumike 0-25mm/0.001mm, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan). The specimens were submitted to the heat treatment according to the manufacturer's instruction to promote the final crystallization (initial temperature 403°C, heat rate 1 - 90°C/min until 820°C, hold time 10 seconds, heat rate 2 - 30°C/min until 840°C, hold time 7 minutes, long-term cooling at 700°C; vacuum 1 at 550 until 820°C, vacuum 2 at 820 to 840°C), and then randomly allocated using a computer software (Random Allocator) into 6 groups (n= 5) considering the factor "surface treatment".

2.2. Surface treatments

The study design is described in Table 1, taking into consideration the distinct procedures shown below.

The baseline comparison condition assumed in this study was a mirror-polished condition (on both sides of the samples), which was executed on a polishing machine (EcoMet/AutoMet 250, Buehler) with SiC papers (600–2000 grit). Moreover, the ceramic sample surfaces to be analyzed were first mirror-polished in order to guarantee that the assessments of optical and surface characteristics were in response to the surface treatment and not to inherent defects introduced by cutting. Next, this mirror-polished side was treated and its opposite side was then mirror-polished until achieving the final thickness of 1.5 mm. A statistical analysis based on one-way ANOVA with posterior Tukey tests were run to assure final similar thickness through all specimens in all conditions and is described in Table 1.

2.2.1. As-cut condition

As CAD/CAM milling inherently leads to a rough internal surface of ceramic restorations, this condition aims to mimic this scenario (similar to the one shown in Fraga et al. [28] which shows mean values of Ra 1.8 μm and Rz 11.0 μm). Thus, the ceramic surface was polished with a diamond grinding disc (Dia-Grid Diamond Discs #120, Allied High-Tech Products) using a polishing

machine (EcoMet/AutoMet 250, Buehler).

2.2.2. Grinding

Grinding was executed with a medium-grit diamond bur (#4219 – grit size 90-120 μm, KG Sorensen) in a high-speed handpiece (Kavo Dental, Biberach, Germany) under constant water-cooling (≈30 ml/min). Specimens were attached to a metal base which served as reference for maintaining the diamond bur tip parallel to the specimen surface, and then grinding was manually executed in a horizontal direction. Caution was taken in conducting this procedure to standardize it in order to increase its reproducibility; as such, the entire ceramic surface to be ground was marked with a permanent marking pen (Pilot, São Paulo, Brazil); grinding was executed until completely eliminating this mark; the diamond bur was also replaced after each specimen (1 bur per specimen). This method was previously employed by Zucuni et al. [19] and Pereira et al. [29].

2.2.3. Polishing

Firstly, a finishing procedure was executed with fine (#4219F – grit size 46 μm, KG Sorensen) and extra-fine (#4219FF – grit size 30 μm, KG Sorensen) diamond burs using the same methodology described in the "*Grinding*" section.

Next, an Optrafine System (Ivoclar Vivadent) polishing kit was used. This consists in 2 tips (Light-blue and Dark-blue) and a nylon brush which is used combined with a diamond paste (OptraFine HP Polishing Paste – diamond particle size of 2–4 μm) to generate a smooth and shiny surface. Procedures were also cautiously performed for standardization and reproducibility: the specimens were affixed in a similar metal base to the one used in the grinding procedure, and the area to be polished was divided into two regions considering the size of the polishing tips; this procedure was executed for 25 s in each region for each specific tip [19].

2.2.4. Glazing

A thin glaze layer was applied on the ceramic surface (IPS Ivocolor Glaze Paste, Ivoclar Vivadent) following the manufacturer's guidelines. To do so, the paste was mixed with distilled water until obtaining an adequate consistency, and then applied onto the whole ceramic surface with a specific brush, and sintered in a Vacumat 6000MP furnace (Vita Zahnfabrik; drying temperature 403°C, furnace closing time 6 min, heating rate 45°C/min, final temperature of 710°C, maintenance of 1 min, with a vacuum at 450°C and at 709°C, slow cooling). One of the researchers (G.K.R.P.) developed pilot studies prior to performing this step in order to standardize and improve reproducibility of this protocol until a homogenous glaze layer became noticeable under Scanning Electron Microscopy (SEM - Vega3, Tescan, Czech Republic).

2.3. Optical measurements

2.3.1. Translucency (TP_{00}) and Color changes (ΔE_{00})

An SP60 spectrophotometer (X-Rite, Grand Rapids, MI, USA) was used according to the CIE 1976 L*a*b* color scale relative to the CIE standard illuminant D65 (as defined by the International Commission on Illumination), which corresponds to "average" daylight (including ultraviolet wavelength region with a correlated color temperature of 6504K) to estimate the translucency (TP_{00}) and the color change (ΔE_{00}) of each specimen with values comparing each surface condition to the Mirror-Polished (baseline). Color parameters were obtained in three coordinate dimensions of L* (from 0=black to 100=white), a* green-red (−a*=green; +a*=red), and b* blue-yellow (−b*=blue; +b*=yellow) over white (CIE L* = 90.1, a* = −0.1 and b*=9.6), black (CIE L*=100, a*=−0.6 and b*=15.1), grey (CIE L* = 53.9, a* = −1.0 and b*=0.6) and shaded A2 (CIE L* = 87.7, a* = −1.1 and b*=15.1) backgrounds.

Measurements for each specimen were collected three times, and the average of those readings was used for statistical analysis for the different parameters. Two measurement directions in relation to the specimens' treated surface and the backgrounds were considered: one with the treated surface facing down (in contact with the background), and the other facing up (opposite to the background). A coupling substance (glycerol, $C_3H_8O_3$) (Vetec Química Fina Ltda, Rio de Janeiro, Brazil) with a refractive index of 1.47 was used to minimize light scattering by eliminating the presence of an air layer between the specimen and the background [30,31]. Calibration of the spectrophotometer was executed before measurements. The specimens were rinsed after each measurement in order to remove the coupling substance and dried with gauze afterward.

The TP_{00} of each specimen was obtained by calculating the color difference between the specimen against the white background and against the black backgrounds using the equation of CIEDE2000:

$$TP_{00} = \left[\left(\frac{L'_B - L'_W}{K_L S_L}\right)^2 + \left(\frac{C'_B - C'_W}{K_C S_C}\right)^2 + \left(\frac{H'_B - H'_W}{K_H S_H}\right)^2 + R_T\left(\frac{C'_B - C'_W}{K_C S_C}\right)\left(\frac{H'_B - H'_W}{K_H S_H}\right)\right]^{\frac{1}{2}} \text{(Eq. 1)}$$

where the subscripts "B" and "W" refer to lightness (L′), chroma (C′), and hue (H′) of the specimen over the black and the white backgrounds, respectively. R_T is a function (the so-called rotation function) which accounts for the interaction between chroma and hue differences in the blue region. SL, SC, and SH are the weighting functions and KL, KC, KH are the correction terms to be adjusted according the experimental conditions. These parametric factors of the CIEDE2000 color difference formula were set as 1 in the present study.

The same equation of CIEDE2000 was used for the color change (ΔE_{00}) assessment, however considering the different surface treated conditions in comparison to the Mirror-Polished surface always on the same background. The ΔE_{00} values described by Paravina et al. [32] of 0.8 and 1.8 were considered as clinical thresholds for perceptibility and acceptability, respectively.

2.3.2. Light irradiance and transmittance

The specimens were evaluated to determine the amount of light irradiance (i.e. amount of light received by the specimen), and total irradiant energy through each considered condition (ceramic under different surface treatments). A personalized colorimeter was assembled at ACTA (Academic Centre for Dentistry Amsterdam) within a black-box (avoiding any influence of day-light on the measurements). A light source that emitted pulses with 3 light shades in specific wavelengths (blue - wavelength ranging from 440 –

485 nm; green – 500 – 565 nm; and red 625 – 740 nm) was used. A sensor located on the opposite side of the system inside a base for placement of the ceramic sample measured the amount of transmitted light which was displayed on a computer attached to the device.

Two measurements were performed for each specimen, one with the surface treated side of the ceramic facing up, close to the light emission source, and the other under the opposite direction (facing down), close to the sensor. In addition, measurements without samples were performed before and after each analysis to assure the amount of light emitted from the source. The overall light transmittance value of each condition was divided by the overall light transmittance value to analyze the light transmittance, with no specimen in the spectrophotometer to determine overall-light transmittance as a %-value. The transmittance was given as a value between 100% (transparent) and 0% (opaque).

2.4. Surface characteristics assessments

2.4.1. Topography

Two specimens per group were initially cleaned in an ultrasonic bath (1440D – Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min, and then they were gold sputtered and analyzed by Scanning Electron Microscopy (SEM - Vega3, Tescan) under 150×and 5000× magnification to observe the superficial topographical pattern. An additional transversal analysis was also performed on SEM to elucidate the shape of superficial defects and also to enable accessing the glaze layer integrity.

2.4.2. Roughness

All specimens from each evaluated condition were submitted to a roughness analysis on a specific profilometer (Mitutoyo SJ-410, Mitutoyo Corporation) considering the parameters recommended by ISO:4287-1997 [33] (*Ra* and *Rz*). Three readings were executed per specimen on the opposite direction of that used during grinding, polishing and/or glazing with a cut-off (n = 5), λC 0.8 mm e λS 2.5 μm. Average values were obtained for each specimen after reading. The topographical profiles were extracted using the specific manufacturer's computer software.

2.5. Statistical Analysis

A descriptive analysis of final thickness, roughness (*Ra* and *Rz* parameter), optical measurements at the three coordinate dimensions of L, a* and b*, TP_{00} and ΔE_{00} was performed to obtain mean and standard deviation (SD) values for each condition. Next, the Shapiro Wilk and Levene tests were performed to assess data distribution and homoscedasticity. A one-way ANOVA with a post-hoc Tukey test ($\alpha = 0.05$) were subsequently used since all considered parameters assumed parametric homoscedastic distribution.

3. Results

Grinding deleteriously impacted the translucency parameter (TP_{00}) of lithium disilicate ceramic, especially when the treated surface was faced bottom down (in contact with the background). Despite this, polishing was able to reestablish the translucency observed at baseline conditions (mirror-polished and/or as-cut). When glaze was applied alone, it was not able to reestablish such parameter, but it was reached when associated with polishing (Figure 1).

With regards to color change (ΔE_{00}), grinding also deleteriously impacted this parameter, triggering variations which were perceptible (above 0.81) but clinically acceptable (below 1.77). The same performance was noticed in all backgrounds (black, white, gray and shaded A2). When the treated surface was faced bottom down (in contact with the background), the influence of the surface treatment on ΔE_{00} decreased, where grinding only led to perceptible alteration over the black background. It is also emphasized that all post-processing protocols were able to reestablish ΔE_{00} parameters similar to the baseline (mirror-polished), as they were below the perceptibility threshold 0.81 (Figure 2) in all conditions considered. Table 2 describes the statistical analysis of all color scale parameters (L*a*b*) in all backgrounds and under both explored conditions (treated surface on top or on bottom).

When considering the light transmittance through the specimen, different performance was noticed when different light spectra were applied (Table 3). Grinding deleteriously impacted (both the treated surface facing up or down) under a blue wavelength (440-485 nm), and the most enhanced light transmittance ability was noticed with polished and/or glazed conditions. Lower differences were observed under a green wavelength (500-565 nm), but still followed the same trend (grinding deleteriously impacting and polishing and/or glazed conditions enhancing). Finally, no statistical influences were noticed on the amount of light transmitted through the ceramic under a red wavelength (625-740 nm) (Table 3).

Regarding topography, grinding greatly altered the surface pattern, introducing scratches and defects throughout the surface, following the movement pattern of the grinding tool. The mirror-polished condition showed a completely homogeneous and smooth surface which was only comparable to the ones observed when glaze was applied (ground glazed and ground polished glazed groups). The As-cut group showed an altered surface pattern, with some similarities to the ground surface condition, but much less intense. Finally, when polishing was performed alone, it was not able to remove all defects introduced by grinding (Figure 3). Roughness analysis (Figure 4) corroborated the topographical assumptions, both statistically and also considering the profilometric surface pattern generated by such analysis.

4. Discussion

The present data corroborate that grinding harmfully affects the optical (translucency – TP_{00}; color change - ΔE_{00}; and light transmittance/irradiance) and surface characteristics (topography and roughness) of a lithium disilicate ceramic, regardless of the background color considered. It was also found that only glaze application is not able to completely reestablish such deleterious impact regarding optical characteristics. Therefore, the necessity of polishing protocols after grinding is mandatory for reverting such deleterious influence. It was additionally noticed that glaze application was the only procedure that completely turned back topographical characteristics comparable to the baseline (mirror-polished). The present data also proves that the region where the surface roughness is introduced interferes in both outcomes: different performances were observed when the treated surface was faced up or bottom in relation to the background. Based on this, the assumed null hypothesis was rejected.

It is already known that an increase in roughness and topographical alterations may inherently affect the light transmittance through a restorative material [22,25,26]. The influencing mechanism is explained by the fact that when light transmits through a roughened surface, the light direction and incidence is altered, and the material consequently shows opacity, among other optical characteristics (e.g., light transmittance and color changes) which may also be altered. We believe that this is the main reason for the alterations depicted by the present data. Therefore, after clinical adjustment with diamond burs, the restorative surface should be submitted to posterior processing treatments to reestablish adequate surface and optical properties. Our results corroborate that polishing is mandatory to revert optical impairments, and glaze is the best protocol to reestablish surface roughness and smoothness.

When only glaze is applied over an extremely rough surface, it is not guaranteed that it will completely fill-in all the defects present on this surface, and the presence of bubbles and unfilled areas by doing so may be responsible for the inferior optical performance observed herein. This assumption is not new; some studies have already shown such difficulty in obtaining a homogeneous layer free of bubbles of the glaze material [34,35]. Kilinc and Turgut [20] also recently noticed the best optical behavior (color changes - ΔE) for polishing in comparison to glaze. Despite this, only the application of a glaze material enabled reestablishing the surface roughness and topographical features which were seen at the baseline condition (mirror-polished) in the present study, as seen in Figures 3 and 4. Thus, our data shows that the best approach should be to associate polishing and glaze, however more studies are necessary to corroborate such an assumption.

With regards to the differences observed when the treated surface was faced top or bottom, mimicking the occlusal or intaglio surface of a monolithic restoration, respectively, it has to be emphasized that the surface post-processing protocols are only viable options for reverting deleterious impacts on the external surface. The internal surface is much more difficult to be treated since the glaze application or even a polishing protocol may alter the restoration setting onto the tooth substrate, thus promoting a marginal misfit [36] which could compromise periodontal health and lead to biological failures [37,38]. In this sense, our data encourages that perhaps manufacturers may consider such a mechanism and introduce milling instruments with a subsequential grit size decrease, which would then enable a smoother surface without altering the restorations' dimensions. More studies are also necessary to explore such an alternative. Finally, it is also important to consider that the intaglio surface will interact with the luting agents, and perhaps no deleterious impact would be present if the luting agent is capable of completely filling in the defects and topographical changes present on the restoration, but this hypothesis need to be explored in future studies.

Another potential deleterious influence taking into consideration our findings is regarding light transmittance; deleterious impacts on light transmittance were noticed when surface treatments were executed on both external or internal surfaces. These results are important as the light should be transmitted through the restoration for adequate curing of a luting agent during cementation, otherwise the conversion degree of the resin cement may be decreased, and in turn the properties of such material will be also deleteriously impacted, affecting the longevity of the restorations [39,40].

Thus, based on the aforementioned presuppositions, our results clearly show the mandatory necessity of polishing the surface after grinding to enhance the optical characteristics, and to apply a glaze material on such a surface in order to reestablish the surface smoothness.

5. Conclusion

The following conclusions could be drawn:

- Grinding harmfully affects the optical (translucency – TP_{00}; color change - ΔE_{00}; and light transmittance/irradiance) and surface characteristics (topography and roughness) of a lithium disilicate ceramic, regardless of the background color considered.
- Polishing after grinding is mandatory to revert the deleterious influence triggered by grinding, especially considering optical characteristics.
- Glaze application was the only procedure able to completely revert topographical characteristics comparable to the baseline condition (mirror-polished).

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Figures and Tables

Figures – removed from this report based on restrictions of file size.

Tables

Table 1. Experimental design and final thickness of specimens (Mean and standard deviation after statistical analysis under one-way ANOVA and post-hoc Tukey tests).

Group (n= 5)	Surface Treatment	Final thickness (mm)
		Mean (SD)
Mirror-Polished	Polished in a polishing machine with 600-and 2000-grit size SiC papers	1.52 (0.03)^{A}
As-cut*	Polished with a diamond grinding disc (Dia-Grid Diamond Discs #120, Allied High-Tech Products) in a polishing machine, mimicking a CAD/CAM milled surface.	1.53 (0.02)^{A}
Ground*	Grinding with medium-grit diamond bur (#4219 grit size 90-120 µm, KG Sorensen)	1.53 (0.02)^{A}
Ground Polished*	Ground + Finishing with fine (#4219F grit size 46 µm, KG Sorensen) and extra-fine (#4219FF grit size 30 µm, KG Sorensen) diamond burs followed by the 3-step sequential diamond ceramic polishing kit (Optrafine System – Ivoclar Vivadent)	1.50 (0.02)^{A}
Ground Glazed*	Ground + Glaze application (IPS Ivocolor Glaze Paste, Ivoclar Vivadent)	1.53 (0.01)^{A}
Ground Polished Glazed*	Association of Ground + Polished + Glaze	1.53 (0.03)^{A}

* The opposite side of the specimen was mirror-polished as described for the control group.

Table 2. Description of data and statistical analysis of all color scale parameters (L*a*b*) in all backgrounds and under both explored conditions (treated surface on top or on bottom).

Group		Spectrophotometer Analysis											
		L				a				b			
		B	W	G	S (A2)	B	W	G	S (A2)	B	W	G	S (A2)
Treated surface facing up	Mirror-Polished	76.2 $(0.3)^{A}$	88.5 $(0.5)^{A}$	81.7 $(0.4)^{A}$	82.8 $(0.3)^{A}$	-1.9 $(0.1)^{D}$	-1.2 $(0.1)^{BC}$	-0.9 $(0.1)^{B}$	-0.9 $(0.1)^{B}$	8.7 $(0.2)^{B}$	15.4 $(0.4)^{A}$	12.7 $(0.2)^{AB}$	14.1 $(0.3)^{A}$
	As-cut	75.8 $(0.1)^{A}$	88.1 $(0.4)^{A}$	81.2 $(0.1)^{A}$	82.3 $(0.3)^{A}$	-1.7 $(0.0)^{BC}$	-1.0 $(0.1)^{B}$	-0.8 $(0.1)^{B}$	-0.8 $(0.1)^{B}$	8.7 $(0.1)^{B}$	15.4 $(0.1)^{A}$	12.6 $(0.1)^{B}$	14.0 $(0.2)^{AB}$
	Ground	74.9 $(0.9)^{B}$	86.9 $(0.8)^{B}$	80.1 $(0.7)^{B}$	81.1 $(0.5)^{B}$	-1.5 $(0.1)^{A}$	-0.8 $(0.1)^{A}$	-0.5 $(0.1)^{A}$	-0.6 $(0.1)^{A}$	9.2 $(0.2)^{A}$	15.6 $(0.2)^{A}$	13.0 $(0.1)^{A}$	14.3 $(0.3)^{A}$
	Ground Polished	76.0 $(0.4)^{A}$	88.8 $(0.4)^{A}$	81.6 $(0.4)^{A}$	82.9 $(0.4)^{A}$	-1.8 $(0.1)^{DC}$	-1.1 $(0.1)^{BC}$	-0.9 $(0.1)^{B}$	-0.9 $(0.1)^{B}$	8.8 $(0.1)^{AB}$	15.4 $(0.2)^{A}$	12.7 $(0.2)^{AB}$	14.2 $(0.2)^{A}$
	Ground Glazed	76.3 $(0.4)^{A}$	88.3 $(0.3)^{A}$	81.6 $(0.4)^{A}$	82.8 $(0.4)^{A}$	-1.6 $(0.0)^{AB}$	-1.1 $(0.1)^{BC}$	-0.8 $(0.1)^{B}$	-0.9 $(0.1)^{B}$	8.7 $(0.2)^{B}$	14.8 $(0.2)^{B}$	12.4 $(0.1)^{B}$	13.6 $(0.2)^{BC}$
	Ground Polished Glazed	76.1 $(0.3)^{A}$	88.5 $(0.5)^{A}$	81.7 $(0.2)^{A}$	83.0 $(0.6)^{A}$	-1.6 $(0.1)^{ABC}$	-1.2 $(0.1)^{C}$	-0.8 $(0.0)^{B}$	-0.9 $(0.0)^{B}$	8.2 $(0.4)^{C}$	14.4 $(0.3)^{B}$	11.9 $(0.2)^{C}$	13.4 $(0.2)^{C}$
Treated surface facing down	Mirror-Polished	76.2 $(0.3)^{B}$	88.5 $(0.5)^{ABC}$	81.7 $(0.4)^{BC}$	82.8 $(0.3)^{B}$	-1.9 $(0.1)^{D}$	-1.2 $(0.1)^{B}$	-0.9 $(0.1)^{B}$	-1.0 $(0.1)^{C}$	8.7 $(0.2)^{B}$	15.4 $(0.4)^{A}$	12.7 $(0.2)^{B}$	14.1 $(0.3)^{ABC}$
	As-cut	76.2 $(0.2)^{B}$	88.3 $(0.3)^{BC}$	81.5 $(0.2)^{C}$	82.6 $(0.2)^{BC}$	-1.7 $(0.0)^{BC}$	-1.0 $(0.1)^{AB}$	-0.8 $(0.1)^{AB}$	-0.8 $(0.1)^{AB}$	8.9 $(0.1)^{B}$	15.3 $(0.1)^{A}$	12.7 $(0.1)^{B}$	14.0 $(0.2)^{BC}$
	Ground	77.5 $(0.3)^{A}$	88.9 $(0.5)^{AB}$	82.5 $(0.5)^{A}$	83.5 $(0.4)^{A}$	-1.6 $(0.1)^{AB}$	-0.9 $(0.1)^{A}$	-0.7 $(0.1)^{A}$	-0.8 $(0.0)^{A}$	9.5 $(0.1)^{A}$	15.6 $(0.2)^{A}$	13.1 $(0.2)^{A}$	14.3 $(0.1)^{AB}$
	Ground Polished	76.0 $(0.4)^{B}$	88.7 $(0.4)^{ABC}$	81.6 $(0.4)^{C}$	82.8 $(0.3)^{B}$	-1.8 $(0.1)^{CD}$	-1.1 $(0.1)^{B}$	-0.8 $(0.1)^{AB}$	-0.9 $(0.1)^{BC}$	8.9 $(0.2)^{B}$	15.5 $(0.2)^{A}$	12.7 $(0.2)^{B}$	14.2 $(0.2)^{ABC}$
	Ground Glazed	77.0 $(0.4)^{A}$	89.1 $(0.3)^{A}$	82.3 $(0.3)^{AB}$	83.4 $(0.3)^{A}$	-1.5 $(0.1)^{A}$	-1.1 $(0.1)^{B}$	-0.7 $(0.1)^{A}$	-0.8 $(0.1)^{A}$	9.5 $(0.2)^{A}$	15.6 $(0.2)^{A}$	13.2 $(0.2)^{A}$	14.4 $(0.1)^{A}$
	Ground Polished Glazed	75.7 $(0.2)^{B}$	88.0 $(0.3)^{C}$	81.2 $(0.1)^{C}$	82.2 $(0.1)^{C}$	-1.6 $(0.1)^{AB}$	-1.1 $(0.1)^{B}$	-0.8 $(0.0)^{AB}$	-0.9 $(0.1)^{BC}$	8.8 $(0.1)^{B}$	14.8 $(0.2)^{B}$	12.5 $(0.1)^{B}$	13.9 $(0.2)^{C}$

Table 3. Results (mean and standard deviation – SD) from light transmittance analysis in colorimeter (in %).

Group	Treated surface facing up			Treated surface facing down		
	Light transmission (colorimeter)			Light transmission (colorimeter)		
	Blue Mean (SD)	Green Mean (SD)	Red Mean (SD)	Blue Mean (SD)	Green Mean (SD)	Red Mean (SD)
Mirror-Polished	5.1 $(0.1)^{BC}$	4.0 $(0.0)^{AB}$	5.0 $(0.2)^{A}$	5.1 $(0.1)^{B}$	4.0 $(0.0)^{A}$	5.0 $(0.1)^{A}$
As-cut	5.1 $(0.1)^{C}$	3.9 $(0.0)^{B}$	4.7 $(0.0)^{B}$	5.1 $(0.1)^{B}$	3.9 $(0.0)^{AB}$	4.7 $(0.0)^{B}$
Ground	4.9 $(0.0)^{D}$	3.8 $(0.0)^{C}$	4.9 $(0.1)^{AB}$	4.9 $(0.1)^{C}$	3.8 $(0.1)^{B}$	4.8 $(0.1)^{AB}$
Ground Polished	5.2 $(0.1)^{AB}$	4.0 $(0.0)^{A}$	4.9 $(0.1)^{AB}$	5.2 $(0.0)^{AB}$	4.0 $(0.0)^{A}$	4.9 $(0.1)^{AB}$
Ground Glazed	5.1 $(0.0)^{ABC}$	3.9 $(0.0)^{B}$	4.8 $(0.1)^{AB}$	5.0 $(0.0)^{B}$	3.9 $(0.0)^{AB}$	4.8 $(0.1)^{AB}$
Ground Polished Glazed	5.2 $(0.1)^{A}$	4.0 $(0.0)^{AB}$	4.9 $(0.1)^{AB}$	5.3 $(0.1)^{A}$	4.0 $(0.1)^{A}$	4.9 $(0.1)^{AB}$

*Different letters in each column indicates statistical differences depicted by one-way ANOVA and Tukey post-hoc test.

2- Artigo 2

Artigo submetido para o periódico *Journal of the Mechanical Behavior of Biomedical Materials* (Qualis A1; FI 3.485).

Influence of testing environment at the fatigue performance of lithium disilicate ceramics and yttria stabilized zirconia

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Short title: Test environment influence on the fatigue performance of ceramics

Abstract
The present study aimed to characterize the influence of the test environment on the static fatigue performance of lithium disilicate-based (LD) glass ceramic and yttrium stabilized zirconia (YSZ) ceramics. Ceramic specimens of LD (IPS e.max CAD, Ivoclar Vivadent) and YSZ (IPS e.max ZirCAD, Ivoclar Vivadent) were obtained at 14×12×1.2 mm, and randomly allocated into three experimental groups, according to the environment condition considered during static fatigue test (n=15): air (ambient condition), inert (paraffin oil) and distilled water. The test was performed using a piston-on-three ball assembly, according to the ISO 6872:2015, under the following static loading protocol: starting stress 100 N for LD and 300 N for YSZ groups; loading application time set to 1 hour for each loading step; step size of 50 N for LD and 100 N for YSZ, applied successively until the specimen fracture. Data from static fatigue strength (MPa) and time to fracture (hours) were recorded with statistical purposes. Fractographic analysis was executed under optical microscopy and scanning electron microscopy. Kaplan Meier analysis corroborate absence of influence of environment on static fatigue outcomes (fatigue strength, time to fracture and survival rates) for the YSZ ceramic. However, for LD glass ceramic, specimens tested in air presented survival rate and static fatigue strength statistically superior than the ones tested in distilled water (p=0.025). Besides, in regards of time to fracture, LD discs tested in air were significantly superior than the ones tested in distilled water (p=0.019) or inert (p=0.017) environments. No statistical differences for Weibull modulus were observed. As for fractography, all failures started on the side where tensile stress concentrates (downside) during testing. Based on that, it can be concluded that the test environment did not affect slow crack growth mechanisms during static fatigue test of YSZ ceramics, but it plays a significant role for lithium disilicate-based glass ceramics.

Keywords: Dental Ceramics. Fatigue. Slow-crack growth. Sub-critical crack growth. Testing medium.

1. Introduction

The fracture of solids in response to load application is associated with little/no plastic deformation, or may be preceded by considerable plastic deformation, depending on the nature of the material (Cesar et al., 2017). Ceramics and glasses are included in the first category being covered by the fields of Linear Elastic Fracture Mechanics (LEFM), and hence are considered as presenting a brittle nature (Barsoum, 1997; Cesar et al., 2017). Another consolidated presuppose in the literature is that the fracture strength of those brittle materials is known to be inversely proportional to the largest or critical flaw present in its volume, as described by Griffith's law (Griffith, 1921).

Being so, any ceramic component when submitted to function will have to withstand mechanical stimuli without triggering rupture to present longevity. However, under function, even in scenarios where they are loaded far below its critical load, continuously or under repetitive conditions, ceramic materials are known to surpass a phenomenon called "fatigue", which is often defined as the degradation (weakening) of a structural component under the influence of mechanical, chemical or biological stress – and in most cases – a combination of them (Kelly et al., 2017).

As described by Cesar et al., 2017, the definition of fatigue at ambient temperatures mostly involves two relevant mechanisms, arising either from stress corrosion, sub-critical crack growth mechanisms (SCG), chemically-assisted by water, and/or from additional cyclic effects. In brief, while SCG deals with crack initiation and its slow growth under the influence of the environment, cyclic effects arise from friction and hydrolytic pressure during crack closing. Today, there is a common understanding that cyclic effects contribute to over-all degradation of brittle ceramics, although to lesser extent compared to SCG (Danzer et al., 2008; Evans, 1974).

In response to variations on ceramic microstructural arrange we may observe different toughening mechanisms that will difficult crack growth (e.g. crack bridging, crack deflection, or transformation toughening - Kruzic et al., 2018; Kelly et al., 2017; Chevalier et al., 1999). The environment may also influence on the fatigue phenomena, specifically at SCG mechanisms, as a humid environment may trigger a corrosive process, where a diffusion-controlled attack of water molecules at the tip of a crack takes place, hydrolyzing siloxane bonds (Si–O–Si). Under mechanical loads the Si–O–Si bonds are strained, which further accelerates the hydrolytic reaction (Joshi et al., 2013; Freiman et al., 2009; Wiederhorn & Bolz, 1970; Wiederhorn, 1967; Charles, 1958).

Despite that, literature in regards of the effect of the environment on SCG mechanism of YSZ (yttrium stabilized zirconia) ceramics, to the best of authors knowledge, are scarce. YSZs are mainly polycrystalline ceramics that are almost absent of glassy composition (Kelly et al., 2008), by that the degradation mechanism aforementioned where humid environment would trigger hydrolyzation of siloxane bonds and facilitate SCG may be altered, or even absent. In fact, YSZ ceramics when submitted to stimuli may surpass a process named transformation toughening where crystals at tetragonal arrange convert into a monoclinic arrange expanding approximately 3-4% in volume and triggering residual stress concentration around surface defects and actuating on closure/decrease on size of any existing crack (Hannink et al., 2000; Piconi & Maccauro, 1999; Chevalier et al., 1999). Thus, these presupposes, justifies an evaluation of how environment conditions (humid or not) may influence the fatigue performance of YSZ ceramics. Static fatigue test would potentiate to access such influence, isolating SCG mechanisms, as it eliminates the effect of cyclic mechanical loads triggering friction between walls and the deleterious consequences linked to such cyclic stimuli.

Based on that, the present study aimed to characterize the influence of environment (distilled water, inert/oil, air) on the static fatigue performance of YSZ ceramics. As a comparison to access environmental influence, a control condition considering lithium disilicate-based (LD) glass ceramics were adopted. The assumed hypotheses were that (1) the environment would not influence the static fatigue performance of YSZ ceramics, and (2) that the environment would influence the performance of LD ceramics.

2. Material and Methods

2.1. Specimen preparation

First, pre-crystallized LD glass ceramic blocks (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) and partially-sintered YSZ blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Liechtenstein) were cut into rectangular specimens with a diamond disc in a saw machine (Isomet 1000; Buehler, USA) under water cooling and then subsequentially manually polished with 400-, 600-, and 1200- grit silicon carbide papers.

Secondly, the LD and YSZ specimens were submitted to thermal treatment to promote full crystallization and sinterization, respectively, according to the manufacturer's instructions. The final dimensions of the specimens were 14×12×1.2 mm and discrepancies on such dimensions lead to specimen replacement. Finally, the specimens of each material were randomly allocated into three experimental groups, according to the environment condition considered during static fatigue test (n=15): air (ambient condition), inert (paraffin oil) and distilled water, as seen on Table 1.

2.2. Static fatigue test

The test was performed using a piston-on-three ball assembly, according to the ISO 6872:2015, in a universal testing machine, and considering the three different test environments described a priori. The bottom surface of the rectangular specimen was positioned on the top of the three steel spheres (2.5 mm diameter, 120º apart, and forming a circle of 10 mm diameter), with a load applied perpendicular to the center of the top surface of the specimen, by a circular cylinder steel piston with a 1.4 mm diameter flat tip.

The static loading protocol started with 100 N for LD and 300 N for YSZ groups. The loading application time was set into 1 hour for each loading step. If the specimen survived, the stress was increased 50 N for LD and 100 N for YSZ, and then, successively until the fracture of the specimen. The software was adjusted to register the static fatigue strength and the time to failure of each specimen. The static fatigue strength, in MPa, of each specimen was calculated using Eqs. (1, 2 and 3) (ISO 6872:2015).

$$\sigma_m = (-0.2387P(X-Y))/b^2 \quad \text{Eq. (1)}$$

$$X=(1+\nu)\ [\ln(B/C)]^2+[((1-\nu))/2]\ [(B/C)]^2 \quad \text{Eq. (2)}$$

$$Y=(1+\nu)[1+\ln[[A/C]^2]]+(1-\nu)([A/(C)])^2 \quad \text{Eq. (3)}$$

where P is the static load at fracture (N), b is the thickness of the specimen (mm), A is the support ball radius (5 mm), B is the radius of the tip of the piston (0.7 mm), and C is the specimen radius (6.5 mm, corresponding to one-half of the edge length of the square plate specimens (Ramos et al., 2018)). A specific Poisson ratio (ν) was used for each ceramic material (0.21 for LD; 0.26 for YSZ), based on the mean values obtained in the resonant ultrasound spectroscopy (RUS) methodology (Belli et al., 2017).

2.3. Statistical analysis

The static fatigue strength (in MPa) and the time to fracture (in hours) were submitted to descriptive analysis. Weibull modulus (m) was calculated to evaluate the influence of the test condition on the reliability of these variables (static fatigue strength and the time to fracture). Survival analysis (Kaplan Meier and Mantel-Cox post-hoc tests), at a significance level of 5%, was also used to investigate

the effect of the test environment (air, inert, and distilled water) on static fatigue strength and the time to fracture of the YSZ and LD ceramics. The survival rates on each respective testing step were tabulated.

2.4. Fractography analysis

After fatigue testing, the specimens were evaluated in an optical microscope (Stereo Discovery V20, Carl-Zeiss; Göttingen, Germany) at 100× of magnification to determine their crack characteristics and assure that the failure started at the side where tensile stress concentrates (ISO 6872:2015). Representative specimens of each group (n=1) were ultrasonically cleaned (1440 D, 50/60 Hz, Odontobras) in isopropyl alcohol for 10 minutes, entirely air-spray dried, sputtered with a gold-palladium alloy and subjected to scanning electron microscopy analysis (VEGA3 Tescan; Brno-Kohoutovice, Czech Republic) at 200× of magnification.

3. Results

Kaplan Meier analysis (Table 2 and 3, Figure 1) showed that the survival rate of the YSZ ceramic was not influenced by the test environment during the static fatigue test (p=0.345 for static fatigue strength; p=0.698 for time to fracture).

However, for LD glass ceramic, the group tested in air presented a significantly high survival rate and static fatigue strength than the group tested in distilled water (p=0.025). In regards of time to fracture, LD discs tested in air were significantly superior than the ones tested under distilled water (p=0.019) or inert (p=0.017) environments (Table 2 and 3, Figure 1).

Weibull modulus of LD and YSZ ceramics were not influenced by the test environment (Table 2).

Fractographic analysis, in all groups, shows the failure origin located on the side where tensile stress concentrates (downside) during testing. Whereas, the crack propagated towards the opposite side, where compression stress concentrates and the load was applied (Figure 2).

4. Discussion

The results of the present study showed that the test environment did not affect the static fatigue behavior of the YSZ ceramic. Regarding the LD glass ceramic, the static fatigue strength and the time to fracture were significantly reduced by the presence of distilled water. Therefore, the hypotheses of the present study were accepted.

As mentioned at the introduction section, the material's microstructure plays an important role in the mechanical behavior of dental ceramics. It is known that polycrystalline ceramics are less susceptible to slow crack growth phenomenon (Cesar et al., 2017; Amaral et al., 2018) than glassy materials, since they present almost absent presence of glassy composition, that optimizes toughening mechanisms and difficult crack growth (e.g. crack bridging, crack deflection, and transformation toughening - Kruzic et al., 2018; Kelly et al., 2017; Chevalier et al., 1999).

Water, either in liquid or as a vapor, is the most common medium that stimulates slow crack growth on materials that present hydrolyzing siloxane bonds (Si–O–Si) (Quinn, 2007; Joshi et al., 2013; Freiman et al., 2009; Wiederhorn & Bolz, 1970; Wiederhorn, 1967; Charles, 1958). The molecules of water chemically react with the strained atomic bonds at the crack tip, inducing a stress corrosion, and weakening the material. To control the effect of the water in this process, an inert medium may be used, being possible to evaluate the influence of the test environment in the fatigue behavior of dental glass-based ceramics. This inert environment may be provided by using some substances, as oils. In the present study the use of a paraffin oil might not completely eliminate the influence of the SCG for lithium disilicate specimens, in concordance with Anusavice and Lee (1989), suggesting a high susceptibility of moisture assisted crack propagation for glassy dental ceramics.

Despite that, based on the present data, we again emphasize that when considering polycrystalline ceramics, as YSZ, the environment did not play a significant role on SCG mechanisms. In fact, it should be considered that the presence of moisture during testing may trigger phase transformation mechanisms in YSZ materials that may lead to even optimized performance, as shown in many existing papers (Piconi & Maccauro, 1999; Pereira et al., 2016; Guilardi et al., 2017; Silvestri et al., 2018). The presence of increased m-phase content may be deleterious only when the amount of m-phase becomes extremely high, where some corroborates such outcome only when the quantity surpasses at least 50% of content (Pereira et al., 2015; Chevalier et al., 2007).

Therefore, the present data corroborate that fatigue tests of YSZ ceramics may be adequately run under any environment to stimulate fatigue without bias, meanwhile for LD, and potentially for other glassy materials, the influence of the environment should be considered to set the adequate testing method.

5. Conclusion

Test environment did not affect SCG mechanisms during static fatigue test of YSZ ceramics. Meanwhile, it plays a significant role for lithium disilicate-based glass ceramics.

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TABLES

Table 1. Experimental design.

Testing environment	Ceramic material Commercial Name	Manufacturer	Analysis
Air	Lithium disilicate-base glass ceramic IPS e.max CAD	Ivoclar Vivadent, Liechtenstein	Static fatigue test (n= 15); Fractography (representative samples for each group)
	Yttrium stabilized zirconia ceramic IPS e.max ZirCAD		
Inert (Paraffin oil)	Lithium disilicate-base glass ceramic IPS e.max CAD		
	Yttrium stabilized zirconia ceramic IPS e.max ZirCAD		
Distilled Water	Lithium disilicate-base glass ceramic IPS e.max CAD		
	Yttrium stabilized zirconia ceramic IPS e.max ZirCAD		

Table 2. Fatigue test results considering static fatigue strength and time to fracture variables - Mean and Standard deviation; Weibull modulus with its respective 95% confidence interval.

Ceramic	Testing environment	Static fatigue strength		Time to fracture	
		Mean (SD) - MPa*	Weibull Modulus**	Mean (SD) - h*	Weibull Modulus**
YSZ	**Air**	961 (163)^{A}	6.22 (3.59-8.69)^{A}	3.34 (1.13)^{A}	2.89 (1.67-4.04)^{A}
	Inert	1,011 (113)^{A}	10.45 (6.03-14.59)^{A}	3.58 (0.7)^{A}	5.81 (3.36-8.12)^{A}
	Distilled Water	951 (134)^{A}	8.52 (4.92-11.90)^{A}	3.28 (0.87)^{A}	4.26 (2.46-5.95)^{A}
LD	**Air**	388 (72)^{A}	5.85 (3.38-8.17)^{A}	3.51 (1.0)^{A}	4.32 (2.49-6.03)^{A}
	Inert	350 (62)^{AB}	6.03 (3.48-8.42)^{A}	2.88 (0.94)^{B}	2.79 (1.61-3.90)^{A}
	Distilled Water	340 (69)^{B}	5.57 (3.22-7.78)^{A}	2.87 (0.76)^{B}	4.32 (2.49-6.03)^{A}

*different letters indicate statistical differences depicted by Kaplan Meier and Mantel-Cox post-hoc tests (significance level = 5%).

** similar letters indicate absence of statistical differences depicted by the overlapping of confidence intervals for Weibull parameters (maximum-likelihood estimation).

Table 3. Survival rates – probability of specimens to exceed the respective static fatigue strength (MPa) and time to fracture (hours) step without fracturing, and its respective standard error values.

	Testing environment	Static fatigue strength (MPa) / Time to fracture (hours)										
		300/1h	*400/2h*	*500/3h*	*600/4h*	*700/5h*	*800/6h*	*900/7h*	*1000/8h*	*1100/9h*	*1200/10h*	*1300/11h*
YSZ	**Air**	1	1	1	1	1	0.87 (0.09)	0.80 (0.10)	0.27 (0.11)	0.13 (0.09)	0.13 (0.09)	0.0
	Inert	1	1	1	1	1	0.93 (0.06)	0.87 (0.09)	0.47 (0.13)	0.20 (0.10)	0.07 (0.06)	0.0
	Distilled Water	1	1	1	1	1	0.80 (0.10)	0.67 (0.12)	0.33 (0.12)	0.13 (0.09)	0.07 (0.06)	0.0
	Testing environment	**Static fatigue strength (MPa) / Time to fracture (hours)**										
		100/1h	*150/2h*	*200/3h*	*250/4h*	*300/5h*	*350/6h*	*400/7h*	*450/8h*	*500/9h*	*550/10h*	*600/11h*
LD	**Air**	1	1	1	0.93 (0.06)	0.93 (0.06)	0.73 (0.11)	0.40 (0.13)	0.40 (0.13)	0.0	-	-
	Inert	1	1	1	0.87 (0.09)	0.80 (0.10)	0.60 (0.13)	0.07 (0.06)	0.07 (0.06)	0.0	-	-
	Distilled Water	1	1	1	0.87 (0.09)	0.73 (0.11)	0.53 (0.13)	0.13 (0.09)	0.07 (0.06)	0.0	-	-

* The symbol '-' indicates absence of specimen being tested in this respective step

FIGURES

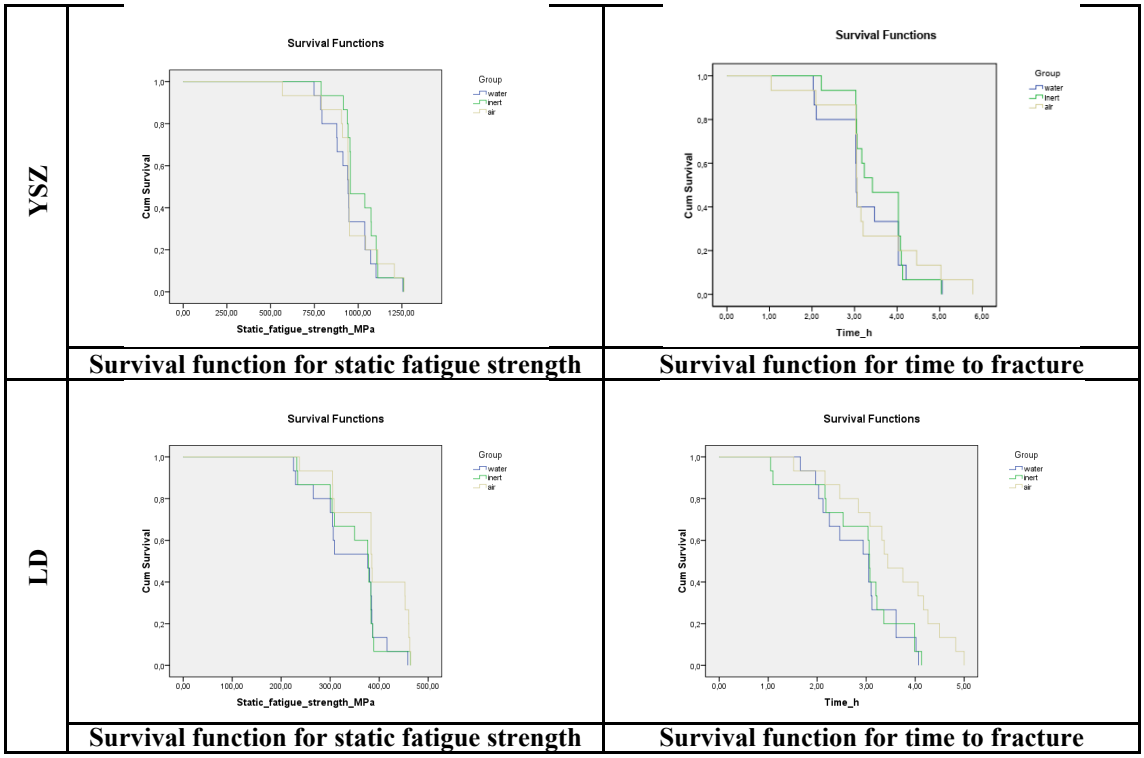


Figure 1. Survival graphs obtained through Kaplan Meier analysis of fatigue data for YSZ and LD ceramics on the three test environments.

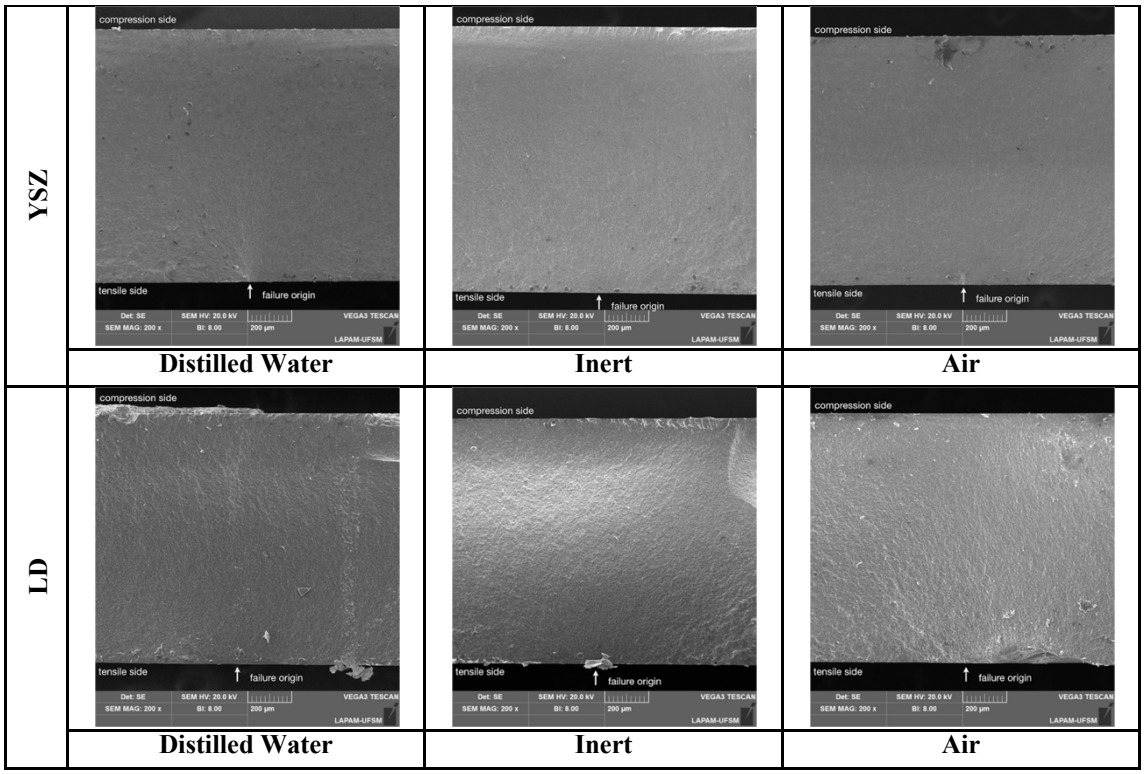
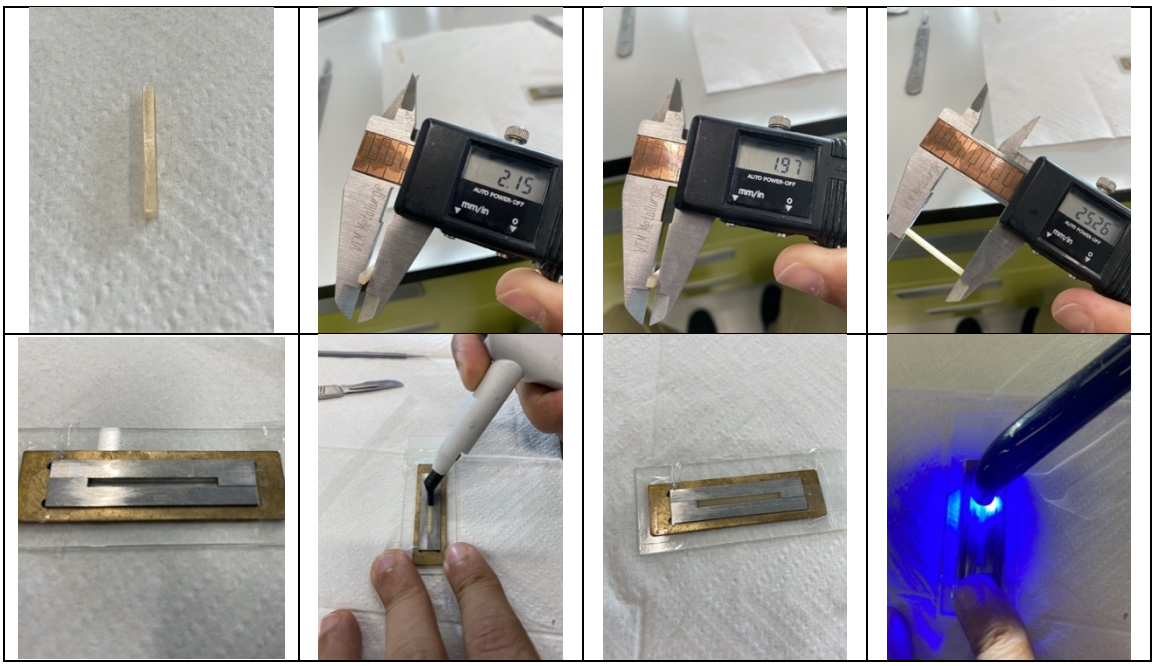


Figure 2. Fractographic images (200× of magn) for YSZ and LD ceramics under the three test environments. The white arrow points to the failure origin on the side where it concentrates tensile stresses during testing, which than propagated to the opposite side.

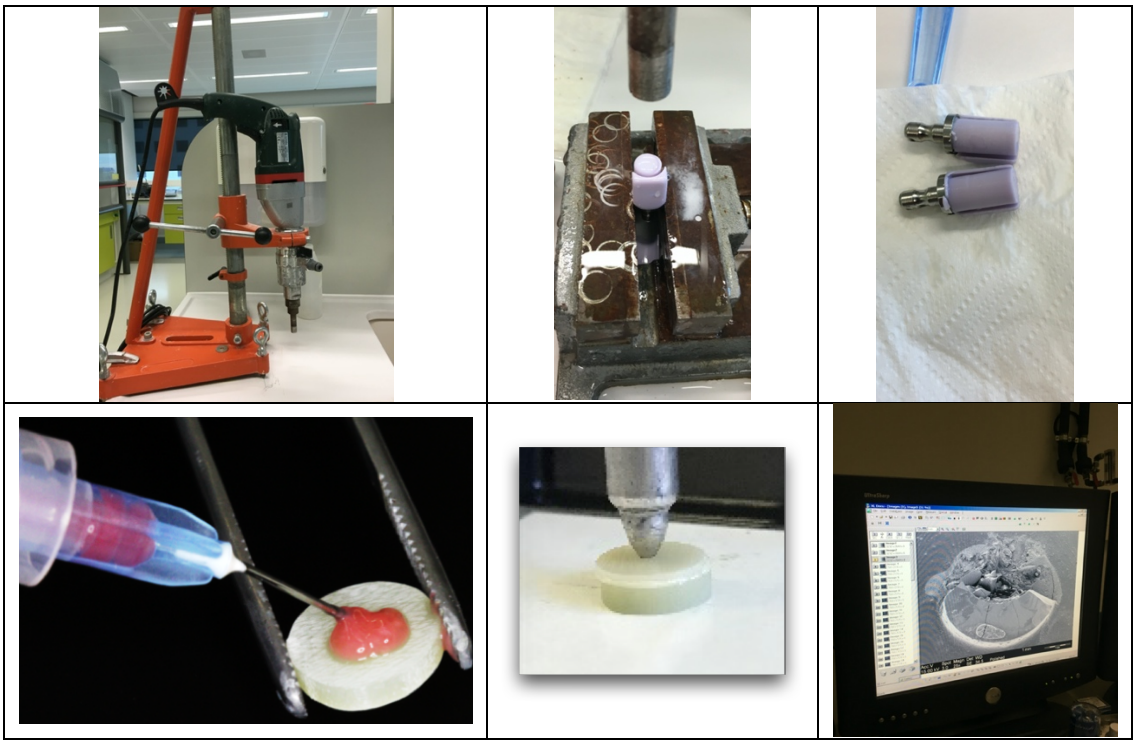
APÊNDICE TÉCNICO

- Treinamento na obtenção de amostras para ensaios em fadiga, em ensaios, e nas análises topográficas e fractográficas posteriores.

Preparo de amostras para fadiga sob flexão em 3 pontos.

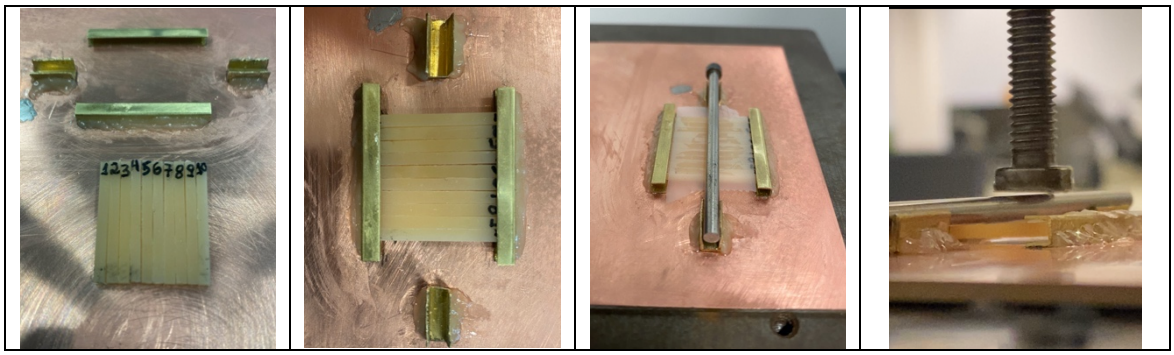


Preparo de amostras para fadiga sob compressão de geometrias cimentadas (g10 – cimento – material restaurador).



- ensaios de fadiga objetivando no desenvolvimento de uma nova metodologia de ensaio para acelerar caracterização de materiais em flexão de 3 pontos.

O professor Cornelis Johannes Kleverlaan propôs uma assembleia de ensaio em flexão 3 pontos que permitiria o ensaio de 10 amostras simultâneas para acelerar a obtenção de dados e a caracterização de comportamento de materiais restauradores utilizados no contexto odontológico. Estudos pilotos foram executados e resultados promissores encontrados, a metodologia encontra-se em otimização para posterior escrita do artigo para publicação. Imagens ilustrativas da assembleia de ensaio encontram-se abaixo.



- Ensaio em flexão 3 pontos, obtenção de dados, análise estatística e discussão dos dados obtidos para escrita de um artigo.

O foco desse estudo é a análise da influência da região do bloco utilizado na usinagem do sistema CAD/CAM de restaurações indiretas. Basicamente o técnico de laboratório na confecção da restauração seleciona o posicionamento ideal desta para a obtenção de características óticas desejadas no caso em tela. Entretanto, inexistem dados na literatura que corroborem que a compactação alcançada pelos fabricantes destes blocos propiciem materiais que apresentam performance mecânica similar nas diferentes regiões consideradas. Como grupos de comparação materiais diretos, onde a compactação fora executada pelos próprios pesquisadores, foram utilizados. Desta forma, diversas barras para ensaio de flexão em 3 pontos foram confeccionadas, e ensaiadas conforme preconizado na ISSO 6872:2015, e então submetidas a análise descritiva, análise de variância 1 via considerando os diferentes materiais em cada região do bloco (interna ou externa), testes de comparação t de student foram utilizados para comparar diferentes regiões dentro de cada material, e uma análise de variância 1 via também foi executada para comparar os materiais com os dados associados das diferentes regiões. Adicionalmente análise de Weibull para obtenção de resistência característica e modulo de weibull em cada um desses cenários também foi calculado. Os dados estão expostos nas tabelas abaixo:

Table 1 – Mean flexural strength (Standard deviation) of the three point bending test considering the different materials and regions (inside, outside or merged).

Material	Flexural strength		
	Inside	Outside	Merged
KAV	248.08^{Ba} (36.36)	249.01^{Ba} (17.24)	248.49^{B} (29.08)
NICE	196.97^{Ca} (34.56)	200.10^{Ca} (27.96)	198.32^{C} (31.56)
APX	189.09^{Ca} (31.20)	196.18^{Ca} (30.74)	191.60^{C} (30.72)
FSUP	112.81^{Ea} (39.41)	130.37^{Da} (31.76)	120.92^{E} (36.81)
LULT	232.51^{Ba} (18.51)	219.31^{BCb} (19.63)	226.23^{B} (19.97)
ENAM	142.02^{DEa} (14.13)	142.76^{Da} (15.75)	142.42^{DE} (14.87)
EMP	144.40^{Da} (25.92)	144.51^{Da} (38.84)	144.45^{D} (32.36)
VMII	122.35^{DEa} (12.58)	112.53^{Da} (8.85)	119.54^{DE} (12.29)
EMAX	347.49^{Ab} (55.08)	392.10^{Aa} (78.28)	369.79^{A} (70.52)

* Different uppercase letters indicate statistical differences on each column.
* Different lowercase letters indicate statistical differences on comparing inside and outside regions for each material.

Table 2 – Weibull analysis (Characteristic strength – σ_c; and Weibull modulus - m) for flexural strength considering inside, outside regions and merged measurements for each material.

Material	σ_c (95% CI)		m (95% CI)		σ_c (95% CI)	m (95% CI)
	Inside	Outside	Inside	Outside	Merged	
KAV	264.00^{Ba} (247.50 – 281.50)	256.70^{Ba} (248.70 – 265.00)	7.32^{ABb} (4.97 – 10.78)	16.41^{Aa} (10.86 – 24.81)	261.30^{B} (251.90 – 271.00)	9.59^{ABC} (7.27 – 12.65)
NICE	209.20^{Ca} (197.50 – 221.60)	209.20^{Ca} (199.00 – 219.80)	7.42^{Ba} (6.09 – 9.05)	10.33^{ABa} (8.68 – 12.30)	209.20^{C} (201.40 – 217.40)	8.60^{BC} (7.54 – 9.81)
APX	201.60^{Ca} (188.80 – 215.20)	209.60^{Ca} (190.60 – 230.50)	7.03^{Ba} (4.96 – 9.97)	6.67^{ABCa} (3.97 – 11.20)	204.20^{C} (194.00 – 215.10)	7.17^{BCD} (5.37 – 9.57)
FSUP	126.10^{DEa} (111.20 – 143.00)	143.20^{Da} (129.80 – 158.10)	3.10^{Ca} (2.31 – 4.18)	4.30^{Ca} (3.07 – 6.02)	134.50^{E} (123.90 – 145.90)	3.51^{E} (2.80 – 4.39)
LULT	240.60^{Ba} (233.50 – 248.00)	227.70^{Ca} (219.70 – 235.90)	14.62^{Aa} (10.71 – 19.97)	13.08^{ABa} (9.29 – 18.44)	234.90^{C} (229.30 – 240.50)	13.43^{A} (10.74 – 16.79)
ENAM	148.10^{Da} (142.50 – 153.90)	149.60^{Da} (143.80 – 155.60)	11.67^{ABa} (8.19 – 16.64)	10.43^{ABa} (7.61 – 14.30)	148.80^{DE} (144.80 – 152.80)	11.40^{AB} (9.05 – 14.36)
EMP	155.30^{Da} (144.80 – 166.50)	158.60^{Da} (142.80 – 176.10)	6.07^{Ba} (4.34 – 8.49)	4.21^{Ca} (3.06 – 5.79)	156.80^{D} (147.80 – 166.50)	5.13^{DE} (4.13 – 6.35)
VMII	127.70^{Ea} (121.80 – 134.00)	116.40^{Ea} (109.30 – 124.00)	11.13^{ABa} (7.45 – 16.61)	13.66^{ABa} (6.66 – 28.01)	124.70^{E} (119.90 – 129.70)	11.51^{AB} (8.39 – 15.79)
EMAX	369.80^{Aa} (347.10 – 393.90)	420.50^{Aa} (389.80 – 453.50)	7.31^{Ba} (5.28 – 10.12)	6.15^{BCa} (4.57 – 8.27)	395.30^{A} (376.00 – 415.60)	6.64^{CD} (5.50 – 8.02)

* Different uppercase letters indicate statistical differences on each column.

* Different lowercase letters indicate statistical differences on comparing inside and outside regions for each material at the same measurement (σ_c or m).