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Pablo Soares Machado

RESISTÊNCIA À FADIGA DE UM SISTEMA BICAMADA DE PORCELANA SOBRE ZIRCÔNIA: EFEITO DAS CONDIÇÕES DE SINTERIZAÇÃO E TRATAMENTO DE SUPERFÍCIE

Santa Maria, RS
2019

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Dissertação apresentada ao Curso de Mestrado do Programa de Pós-Graduação em Ciências Odontológicas, área de concentração em Odontologia, ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do grau de **Mestre em Ciências Odontológicas.**

Orientadora: Prof^{a}. Dr^{a}. Marília Pivetta Rippe

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2019

DEDICATÓRIA

Dedico esse trabalho ao meu grande amigo Marcelo Cavalli Lagreca (In Memoriam), que em algum lugar certamente está muito feliz no dia de hoje. Sinto sua falta.

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“Transmita o que aprendeu. Força, maestria. Mas fraqueza, insensatez, fracasso também. Sim, fracasso acima de tudo. O maior professor, o fracasso é”.

(Kennedy, Kathleen; **Star Wars- O Último Jedi**, 2017)

RESUMO

RESISTÊNCIA À FADIGA DE UM SISTEMA BICAMADA DE PORCELANA SOBRE ZIRCÔNIA: EFEITO DAS CONDIÇÕES DE SINTERIZAÇÃO E TRATAMENTO DE SUPERFÍCIE

AUTOR: Pablo Soares Machado
ORIENTADORA: Marília Pivetta Rippe

Este trabalho está formatado em 2 estudos: O estudo 1 teve por objetivo avaliar a influência da quantidade e do posicionamento de espécimes de zircônia recobertos por cerâmica feldspática no forno de sinterização sobre a resistência à fadiga, cor e translucidez. Discos de zircônia In Ceram YZ (VITA) foram confeccionados e sinterizados (espessura final: 0,7mm; diâmetro: 15mm). Foi realizada a aplicação da porcelana VITA VM9 (VITA), e os espécimes foram divididos em dois grupos para a sinterização: G1- grupo de 20 espécimes, sinterizados um por vez no centro do forno; e G5- grupo de 100 espécimes, sinterizados 5 de cada vez, estando 1 deles no centro do forno (G5C) e outros 4 na periferia (G5P). Todos os espécimes foram desgastados e polidos na face da porcelana até que apresentassem uma espessura final de 1,4mm com a zircônia. Os espécimes foram avaliados quanto à diferença de cor e translucidez através de um espectrofotômetro e submetidos ao teste de fadiga (Step-stress, n=20). Foi realizada análise de sobrevida (Kaplan-Meier) e análise de cor e translucidez pelo parâmetro CIEDE 2000. Não houve diferença estatística entre os espécimes posicionados no centro do forno (G5C) e na sua periferia (G5P) para resistência à fadiga (p=0,13) e translucidez (p=0,32), somente para cor (ΔE_{00}>1,8). Em relação ao número de espécimes por ciclo, houve diferença estatística entre o grupo G1 e G5C para resistência à fadiga (p=0,00) e cor (ΔE_{00}>1,8), mas não para translucidez (p=0,26). O estudo 2 teve como objetivo avaliar a influência de quatro tratamentos de superfície da zircônia e termociclagem sobre a resistência à trinca e à delaminação da porcelana durante teste de fadiga. Discos de zircônia IPS e.max Zircad MO (Ivoclar) foram confeccionados, sinterizados (espessura final: 0,7mm; diâmetro: 15mm) e randomizados em 8 grupos (n=15), de acordo com: ‘tratamento de superfície’ (controle - apenas limpeza; desgaste com ponta diamantada; jateamento com óxido de alumínio; e aplicação de um *liner*) e ‘termociclagem’ (presença ou ausência). Todos os espécimes foram submetidos ao teste de rugosidade previamente à aplicação da porcelana IPS e.max Ceram (Ivoclar) e analisados estatisticamente (Kruskal-Wallis). Após a aplicação da porcelana e sinterização, os espécimes foram desgastados e polidos na face da porcelana até a espessura final de 1,4 mm com a zircônia. Após a termociclagem dos devidos grupos, os espécimes foram submetidos ao teste de resistência à fadiga (Step-stress, n=15) e à análise de sobrevida (Kaplan-Meier). O grupo controle se mostrou semelhante aos demais grupos experimentais para os dois desfechos, sendo que o grupo *liner* apresentou piores resultados em relação ao grupo jateado e ao desgastado com ponta diamantada para a “resistência à trinca”. Nenhum dos desfechos foi afetado pela termociclagem.

Palavras-chave: Cor; Porcelana; Resistência à fadiga; Tratamentos de superfície; Zircônia policristalina parcialmente estabilizada por óxido de ítrio.

ABSTRACT

FATIGUE STRENGTH OF A BILAYER SYSTEM OF PORCELAIN OVER ZIRCONIA: EFFECT OF SINTERING CONDITIONS AND SURFACE TREATMENT

AUTHOR: Pablo Soares Machado
ADVISOR: Marília Pivetta Rippe

This study is formatted into two parts: the objective of study 1 was to evaluate the influence of the quantity and positioning of feldspathic ceramic over zirconia infrastructure specimens inside the sintering furnace on fatigue strength, color and translucency. Zirconia discs of In Ceram YZ (VITA) were made and sintered (final thickness: 0.7mm, diameter: 15mm). The VITA VM9 (VITA) porcelain was applied, and the specimens were divided into two groups for sintering: G1 group- 20 specimens, sintered one at a time in the center of the furnace; and G5 group- 100 specimens, sintered 5 at a time, 1 in the center of the furnace (G5C) and another 4 in the periphery (G5P). All specimens were ground and polished on the porcelain face until a final thickness of 1.4 mm with zirconia. The specimens were evaluated for color difference and translucency through a spectrophotometer and submitted to the fatigue test (Step-stress, n = 20). Survival rate analysis was performed for fatigue strength, while color and translucency analysis were performed by the CIEDE 2000 parameter. There was no statistical difference between the specimens positioned at the center of the furnace (G5C) and those at the periphery (G5P) for fatigue strength ($p = 0.13$) and translucency ($p = 0.32$), just for color ($\Delta E_{00} > 1.8$). Regarding the number of specimens per cycle, there was a statistical difference between the G1 group and the central G5 specimens (G5C) for fatigue strength ($p = 0.00$) and color ($\Delta E_{00} > 1.8$), but not for translucency ($p = 0.26$). Study 2 aimed to evaluate the influence of four zirconia surface treatments and thermocycling on the porcelain crack and delamination resistance of a bilayer assembly during fatigue test. Zirconia discs of IPS e.max Zircad MO (Ivoclar) were made, sintered (final thickness: 0.7mm, diameter: 15mm) and randomized into 8 groups (n = 15), according to: ‘surface treatment’ (control - just cleaned; grinding; air-abrasion; and liner application) and ‘thermocycling’ (presence or absence). All specimens were submitted to a roughness test prior to application IPS e.max Ceram (Ivoclar) porcelain and statistically analyzed (Kruskal Wallis). After the porcelain sintering, all specimens were ground and polished on the porcelain face until they had a total thickness of 1.4 mm with zirconia. The thermocycling was performed for the appropriate groups, and the bilayer specimens were then submitted to the fatigue test (Step-stress, n = 15) and survival rate (Kaplen-Meier). The control group was similar to the other treatments for both outcomes, and the liner application treatment presented worse results in relation to the grinding and air-abrasion groups for crack outcome. The outcomes were not affected by thermocycling.

Key words: Color; Cycled loading; Porcelain; Surface treatments; Zirconium oxide partially stabilized by yttrium.

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1. INTRODUÇÃO

O rigor estético e a longevidade são hoje dois dos principais pilares para o sucesso do tratamento restaurador em odontologia. Nesse sentido, embora as coroas metalo-cerâmicas apresentem bons resultados clínicos (WALTON, 1999), o crescente rigor estético levou ao desenvolvimento de sistemas baseados no uso de materiais totalmente cerâmicos (VICHI et al., 2015), desde a infraestrutura até o recobrimento da restauração, ou até por meio de coroas monolíticas, que se mostram vantajosas em termos de preservação da estrutura dental (NAKAMURA et al., 2015). No entanto, os sistemas cerâmicos bicamada ainda se mostram uma opção de escolha, principalmente quando se tem um substrato escurecido, onde um mascaramento da sua cor é necessário (BACCHI et al., 2019).

Dentro desse contexto, a zircônia tetragonal policristalina parcialmente estabilizada por ítrio (Y-TZP) vem sendo cada vez mais utilizada, devido às suas excelentes propriedades, como biocompatibilidade, resistência mecânica e tenacidade à fratura, além de proporcionar melhor interação com a luz e se mostrar esteticamente mais agradável quando comparada ao metal utilizado para infraestrutura em prótese fixa (PICONI; MACCAURO, 1999; OZTURK et al., 2008; STAWARCZYK et al., 2017). Sua apresentação na natureza se dá em 3 diferentes formas cristalinas: monoclínica, a sua fase pura (em temperatura ambiente e abaixo de 1170 C°); tetragonal, quando entre 1170C° e 2370 C°; e cúbica, quando acima de 2370C° (PICONI; MACCAURO, 1999). A Y-TZP, após sinterizada, é praticamente 100% tetragonal, contudo reversões de fase ocorrem em função de diferentes estímulos sobre o material, transformando parcialmente a fase tetragonal em monoclínica (ALKURT et al., 2016). É esse mecanismo, conhecido como tenacificação por transformação, que garante excelente desempenho mecânico da Y-TZP, pois o aumento de volume gerado pelos grãos monoclínicos e a consequente tensão de compressão sobre os defeitos do material supera a tensão de propagação de trincas, gerando um aumento de resistência e tenacidade à fratura (PICONI; MACCAURO, 1999; GARVIE et al., 1975).

No entanto, apesar de se mostrar mais similar à estrutura dental em relação ao metal, o alto conteúdo cristalino da zircônia dificulta a passagem de luz, remetendo à uma aparência opaca (GUESS et al., 2009), o que leva à necessidade de um recobrimento de porcelana para uma maior harmonia visual (OH et al., 2015). As cerâmicas vítreas, como a feldspática, são capazes de acrescentar translucidez à restauração, muito em função da grande presença de conteúdo à base de sílica na sua composição, mascarando a infraestrutura e a tornando mais agradável esteticamente (HO; MATINLINNA, 2011; BAJRAKTAROVA-VALJAKOVA et al., 2018; MI-JIN-KIM et al., 2013). No entanto, mesmo tendo uma pequena presença de conteúdo cristalino em sua composição, a cerâmica feldspática possui uma reduzida capacidade de suportar cargas mecânicas (BAJRAKTAROVA-VALJAKOVA et al., 2018). Sendo assim, do ponto de vista clínico, o sucesso do sistema bicamada, tanto do ponto de vista mecânico quanto estético, depende do bom aproveitamento de ambas as partes, a infraestrutura de zircônia e seu recobrimento de porcelana (STAWARCZYK et al., 2013).

Estudos mostram que vários fatores podem afetar as propriedades finais dos sistemas cerâmicos (GOZNELI et al., 2014), como por exemplo a espessura de material utilizada (OZTURK et al., 2008), regularidade da superfície (YILMAZ; OZKAN, 2010) e número de sinterizações (VICHI et al., 2015). Claus (1989) relata que também a temperatura do forno, bem como a taxa de aumento da mesma, tempo de espera e resfriamento afetam a sinterização das fases vítreas e cristalinas da porcelana, gerando alterações a nível microestrutural. No entanto, a literatura não aborda as variações dentro de um mesmo ciclo de queima, como a organização espacial das coroas protéticas dentro do forno, nem a quantidade de peças sinterizadas ao mesmo tempo em relação a possível influência destes parâmetros sobre as propriedades mecânicas e ópticas dos materiais.

Isso se torna relevante quando se tem em mente o crescimento da utilização de materiais totalmente cerâmicos, o que leva a uma sobrecarga de trabalho para os laboratórios protéticos. Dessa forma, a sinterização de coroas é feita de forma simultânea em um mesmo forno para otimização do tempo, sem a devida atenção para uma possível influência dessa etapa sobre as propriedades finais dos materiais, que poderiam comprometer a longevidade e até mesmo o resultado estético final do tratamento. Mais do que isso, a questão se mostra importante principalmente dentro de um contexto de resistência à fadiga, visto que no meio oral a constante função mastigatória leva a cargas cíclicas sobre esses materiais, e estas são responsáveis pela grande maioria das falhas das restaurações totalmente cerâmicas (KELLY et al., 2017).

Contudo, mesmo um bom aproveitamento das propriedades mecânicas muitas vezes não é capaz de evitar falhas nas restaurações cerâmicas bicamada. Dentre as principais situações de insucesso para esse sistema, destacam-se a ocorrência de delaminação da porcelana e lascamento (RECOW et al., 2011). Estudos relatam que as taxas dessas falhas em porcelanas sobre infraestruturas de Y-TZP chegam a 13% em 3 anos (SAILER et al., 2006) e até 15,2% em 5 anos (SAILER et al., 2007). Por outro lado, a taxa de fratura em restaurações metalo-cerâmicas tem sido relatada como 2,5% após 5 anos (SCURRIA et al., 1998). Isso ocorre pelo fato da resistência de união entre a cobertura de porcelana e a Y-TZP ser menor do que em relação ao metal (MATSUMOTO et al., 2013). De acordo com Guess et al. (2008) isso pode ser explicado pela falta de um consenso na literatura sobre o mecanismo de união entre materiais cerâmicos, o que dificulta a padronização de ações para reduzir a incidência de delaminação. Por essa razão, ainda se considera como padrão ouro a utilização das coroas metalo-cerâmicas para restaurações com prótese fixa (WALTON, 2013).

Na tentativa de resolver este problema, alguns estudos indicam que o aumento da rugosidade superficial da Y-TZP aumentaria o embricamento micromecânico e consequentemente a resistência de união com a porcelana (GUAZZATO et al., 2005), no entanto, outros estudos apontam que a união química também se mostra importante (FISHER et al., 2008). Nesse sentido, diferentes tipos de tratamentos de superfície da zircônia são propostos para melhorar sua união com a porcelana, como o jateamento com óxido de alumínio, desgaste com ponta diamantada, aplicação de uma cerâmica intermediária e introdução de partículas de vidro (HE et al., 2014; ABOUSHELIB et al., 2006; LIU et al., 2015). A fim de ampliar o conhecimento a respeito do comportamento mecânico em fadiga e cor dos sistemas cerâmicos bicamada, a realização de estudos sobre esse assunto é encorajada, para que seja possível aumentar a longevidade dos tratamentos restauradores, através da diminuição da ocorrência de delaminação, que é responsável pela necessidade de substituição dessas restaurações com prótese fixa. Sendo assim, a presente dissertação está apresentada sob a forma de dois artigos científicos, cada um com objetivos distintos, a serem submetidos, respectivamente, nas revistas *Journal of Prosthodontic Research,* de fator de impacto 2.636 e Qualis A1, e *Operative Dentistry,* de fator de impacto 2.027 e Qualis A1.

ARTIGO 1 – “*Fatigue behavior and colorimetric differences of a porcelain-veneered zirconia: effect of quantity and position of specimens during firing.”* Com o objetivo de avaliar a influência da organização de espécimes de cerâmica feldspática sobre uma infraestrutura de zircônia quanto à quantidade e posicionamento no forno de sinterização sobre a resistência à fadiga, diferença de cor e translucidez.

ARTIGO 2 – “*Influence of zirconia surface treatments of a bilayer restorative assembly on the fatigue performance.*” Com o objetivo de avaliar a influência de quatro tratamentos de superfície de uma Y-TZP e envelhecimento sobre a resistência à trinca e delaminação em fadiga da porcelana de cobertura, além de análise de sobrevida do conjunto bicamada.

2. ARTIGO 1: Fatigue behavior and colorimetric differences of a porcelain-veneered zirconia: effect of quantity and position of specimens during firing.

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Original Research Article

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

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Short title: Spatial organization during firing, fatigue and colorimetric properties of veneered-zirconia

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

Abstract

Purpose: To evaluate the influence of quantity and positioning of veneered 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), where both layers presented 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 (ΔE_{00}) and translucency (TP_{00}) were calculated using CIEDE2000 equations. The step-stress fatigue test was performed with 10 Hz frequency, initial load 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 cycles for failure were recorded for statistical analysis (Kaplan-Meier and Mantel-Cox post-hoc tests).

Results: Unacceptable color differences ($\Delta E_{00}>1.8$) were observed comparing G5C vs. G1 (quantity) and G5C vs. G5P (positioning), meanwhile translucency parameters were not affected. Besides, only the factor 'quantity' influenced the fatigue performance (G1>G5C).

Conclusion: 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. Sintering. Mechanical Cycling. Optical Phenomena. Y-TZP.

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 esthetics 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 [2]. In addition to the high crystalline content, the toughening mechanism of Y-TZP through phase transformation (tetragonal to monoclinic) gives it a higher resistance to crack propagation when subjected to the masticatory cyclical loading [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, especially when restoring scenarios with non-favorable substrate color, e.g. darkened teeth or metallic based cores [5].

Among the options of veneering materials for all-ceramic restorations, feldspathic ceramic is highly indicated due to their excellent optical properties [6]. When applied by the layering technique, these ceramics require multiple firing cycles until reaching the desired thickness and color. 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]. Even though they present good mechanical properties, all-ceramic crowns may fail when subjected to fatigue stresses over time [8], which are caused by cyclical intermittent loading with intensity below the monotonic strength of the material. The crack originates from a critical defect and then propagates until catastrophic failure occurrence [9].

The oral environment is challenging for ceramic materials since it generates different stimuli such as masticatory cyclic loading, temperature and pH variations, and humidity [10].

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 and density and decrease its porosity, enhancing its mechanical properties [11]. Also, according to Hasssija et al. [12], repeating firings up to nine times did not affect the color stability 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, can influence their mechanical and color properties.

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. Therefore, it is necessary to investigate the effects of the quantity and positioning of porcelain-veneered zirconia samples inside the furnace on their final mechanical and optical properties, since this could be an important issue concerning the clinical performance of restorations.

Considering these circumstances, the objective of this study was to evaluate the effect of the position (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 [13] 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 inLab 40/19, VITA, 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, Buehler, Lake Bluff, USA) with 600 grit silicon carbide sandpaper until a cylinder was shaped. Next, the cylinders were cut, under water irrigation, with a diamond disc in a precision cutting machine (Isomet 1000, Buehler) in discs 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 Zyrcomat 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. The ceramic powder was handled with the build liquid in standardized proportions (1:1), and condensed with a metal spatula over and around the zirconia discs, which was centralized into a metal template of 18 mm in internal diameter, until filling in the total thickness of the template (2 mm). The veneer was applied around the zirconia to compensate the porcelain contraction during the firing cycle, hence avoiding the discover of the infrastructure. The excess liquid was removed at each increment with absorbent paper (softy's

Elite, Melhoramentos, Sao Paulo, Brazil). All specimens were prepared by the same trained operator. The ceramic set was then placed on refractory trays and taken to the furnace (Vacumat 6000 MP, VITA) for porcelain firing process. The furnace was previously calibrated to guarantee a standard firing cycle for all specimens, thus ensuring that the furnace was not a variable in this study.

The veneering ceramic firings were performed using the “first dentin” mode according to Table 1. Whereas for G1, each specimen (n= 20) were positioned in the center of the furnace refractory base and fired alone (Fig. 1A); and for G5, the specimens (N= 100) were randomly allocated in sets of 5 which were positioned together in the furnace for each firing, where one specimen was positioned in the center (G5C) and the other four specimens positioned equidistant from each other at the periphery of the refractory base (G5P) (Fig. 1B). In order to use the same sample size, only one specimen from the periphery (G5P) was used, always from the same place of the refractory base from each firing cycle of group G5 for the comparisons with the specimen located in the center (G5C) of group G5.

After firing, the feldspathic ceramic surface was ground using a polishing machine (EcoMet/AutoMet 250) with #200, #400, #600 and #1200 grit silicon carbide papers under constant water cooling to standardize the specimens thickness (feldspathic layer of 0.7 mm, summing a final specimen thickness of 1.4 mm and diameter of 15 mm, in accordance to ISO 6872:2015 [13] specifications) and to acquire a shining surface. The porcelain around the zirconia was equally ground, using the same approach, to avoid excess of veneer material on the margins of the specimens, without any interference in the fatigue test results, since the margins were out of the stress concentration area, which is inside the three steel balls diameter [14]. Finally, 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.

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 $C_3H_80_3$, Vetec Química Fina Ltda, Rio de Janeiro, Brazil) was used to minimize light scattering between the product and the background [15]. 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 $L^*a^*b^*$ were used in translucency and color difference calculations. After the CIE $L^*a^*b^*$ coordinates evaluation, the specimens were cleaned with alcohol and dried with absorbent paper.

Color differences (ΔE_{00}) between the central G5 specimens (G5C) and the G1 group specimens (quantity factor), and between the center (G5C) and the periphery (G5P) 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. [16], were considered for evaluating if the color differences (ΔE_{00}) caused by quantity or position of specimens during firing were clinically relevant.

$$\mathit{\Delta} E_{00} = \left[\left(\frac{\Delta L\prime}{K_L S_L}\right)^2 + \left(\frac{\Delta C\prime}{K_C S_C}\right)^2 + \left(\frac{\Delta H\prime}{K_H S_H}\right)^2 + R_T\left(\frac{\Delta C\prime}{K_C S_C}\right)\left(\frac{\Delta H\prime}{K_H S_H}\right)\right]^{\frac{1}{2}} \qquad (1)$$

where $\Delta L'$, $\Delta C'$ and $\Delta H'$ are the differences in luminosity, chroma, and hue, respectively, for a pair of measurements. R_T is a rotation function which accounts for the interaction between chroma and hue differences in the blue region. S_L, S_C, and S_H 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 k_L, k_C, and k_H 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 (ΔTP_{00}) 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 G5C, and G5C and G5P, were considered as 0.62 and 2.62, respectively [17].

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 cyclical intermittent loads using the assembly preconized by ISO 6872:2015 [13] 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 (tension side). Next, a tungsten piston with a 1.6 mm diameter flat cylindrical tip was used to apply the cyclic load on the specimen [13]. An adhesive tape was glued to the specimens on the compression side before the test for better contact between the piston and the specimen [18]. 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 [19]. 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% isopropyl 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) 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-Cox post-hoc tests. Besides the survival rates for each step considered during fatigue test were tabulated.

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 ($\Delta E_{00}> 1.8$). 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 TP_{00} values were not enough to reach clinically perceptible and unacceptable thresholds ($\Delta TP_{00}< 0.62$) (Table 2).

Regarding the mechanical fatigue behavior, it was noticed that the 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 (G5C = G5P; $p= 0.131$) (Table 3). In terms of survival rate, it was observed that the 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 and Fig. 2), 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. 3).

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 ($\Delta E_{00}> 1.8$). Therefore, the second hypothesis was also partially accepted.

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 [20], 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 [21]. 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, therefore in accordance with the findings of Hammad and Stein [22]. Another previous study [23] 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 led to a worst/different firing process among samples in terms of crystalline and vitreous phase formation, and a consequently higher porosity of the porcelain layer, decreasing the mechanical resistance for crack initiation and resulting in a lower fatigue strength [24]. We believe that those were the reasons which explain that the specimens of the G5 group demonstrated a lower survival rate, fatigue strength and number of cycles for failure than the G1 group (Table 3).

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 triggering sub-critical crack growth mechanisms, which lead to defects 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 [10].

In addition, the results have also shown 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 [27] evaluated the increase in firing temperature on the hue, chroma and luminosity of metal-ceramics. The authors 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 [20], at the G1 group the specimens absorbed all the heat inside the furnace, while those from the G5 groups 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 [23,24], 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). 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) [28]. 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

[29,30]. 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 [31]. 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. [32].

It is important to note that the outcomes tested in the present study in regards of the performance of bilayer specimens are directly related to the geometry of the furnace used (chamber size and position of the heating resistance). Also, we emphasize that manufacturers preconize strict recommendations for how fixed bridges should be positioned inside the firing furnace, which should be allocated at the periphery with their long axis following the curvature of the refractory, and not towards the center, which implies that the furnace indeed generates differences between the center and the periphery. Thus, our data clearly encourages that new studies evaluates and characterizes the effect of such parameters at 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. Also, 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 [33-35].

5. Conclusions

- The quantity of bilayer specimens in the furnace for porcelain firing affected the biaxial fatigue strength, i.e. only one specimen may 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.

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Tables and figures

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	ZrO_2, 3 mol% Y_2O_3, HfO_2, Al_2O_3, SiO_3, Na_2	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.

Table 2. Means and standard deviations for color difference and translucency.

Parameters	**Specimens position**		**Specimens quantity**	
	Center G5C	**Periphery G5P**	**Five G5C**	**One G1**
L*	67.92 (1.40)	72.14 (2.44)	67.92 (1.40)	69.47 (1.34)
a*	7.72 (0.80)	6.00 (0.84)	7.72 (0.80)	7.02 (0.52)
b*	40.24 (1.07)	39.15 (0.94)	40.24 (1.07)	39.42 (1.18)
†Color difference (ΔE_{00})	3.57 (1.87)		2.15 (1.23)	
¥Translucency (TP_{00})	8.61 (0.45)^{A}	8.81 (0.77)^{A}	8.61 (0.45)^{A}	8.67 (0.75)^{A}
ΔTP_{00}	0.2		0.06	

Different letters in the same line show significant statistical differences in relation to the position or quantity of specimens in the furnace ($p < 0.05$).

† Perceptibility ($\Delta E_{00} > 0.8$) and unacceptability ($\Delta E_{00} > 1.8$) thresholds for color difference. (Paravina et al., 2015).

¥ Perceptibility ($\Delta TP_{00} > 0.62$) and unacceptability ($\Delta TP_{00} = 2.62$) thresholds for translucency (Salas et al., 2018).

Table 3. Results from Step-stress fatigue testing; mean and 95% confidence interval (CI).

Groups	Fatigue (Step-stress; n=20)	
	Fatigue strength (MPa) Mean (CI)	**Number of cycles for failure Mean (CI)**
G1	67.0 (62.7 - 71.3) ^{a}	52,000 (47,711 - 56,289) ^{a}
G5C	55.5 (53.3 - 57.7) ^{b}	40,500 (38,263 - 42,737) ^{b}
G5P	58.0 (55.7 - 60.3) ^{b}	43,000 (40,707 - 45,293) ^{b}

Distinct letters in the columns show significant statistical differences ($p < 0.05$).
*Different letters in each column indicate significant statistical difference based on the confidence intervals overlapping for Kaplan-Meier and Mantel-Cox (Log rank) test for the survival analysis under fatigue.

Table 4. Survival rates, i.e. specimens' probability to exceed the respective fatigue failure strength and number of cycles without failure, and their respective standard error measurements.

Groups	Stress for failure (MPa) / Number of cycles until failure							
	20 / 5×10^3	**30 / 15×10^3**	**40 / 25×10^3**	**50 / 35×10^3**	**60 / 45×10^3**	**70 / 55×10^3**	**80 / 65×10^3**	**90 / 75×10^3**
G1	1	1	1	0.95 (0.49)	0.50 (0.11)	0.20 (0.89)	0.05 (0.05)	0 (0)
G5C	1	1	1	0.55 (0.11)	0 (0)	-	-	-
G5P	1	1	1	0.75 (0.10)	0.05 (0.05)	0 (0)	-	-

The sign '-' indicates absence of specimen being tested on the respective step.

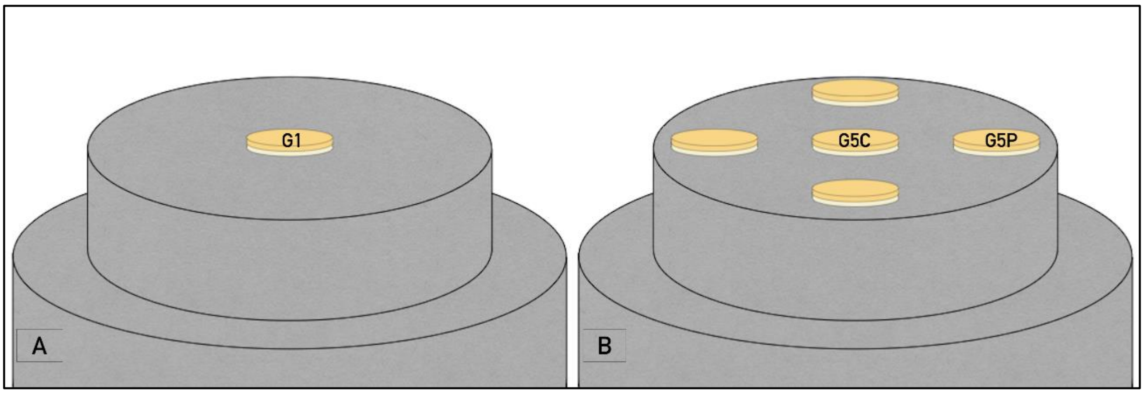
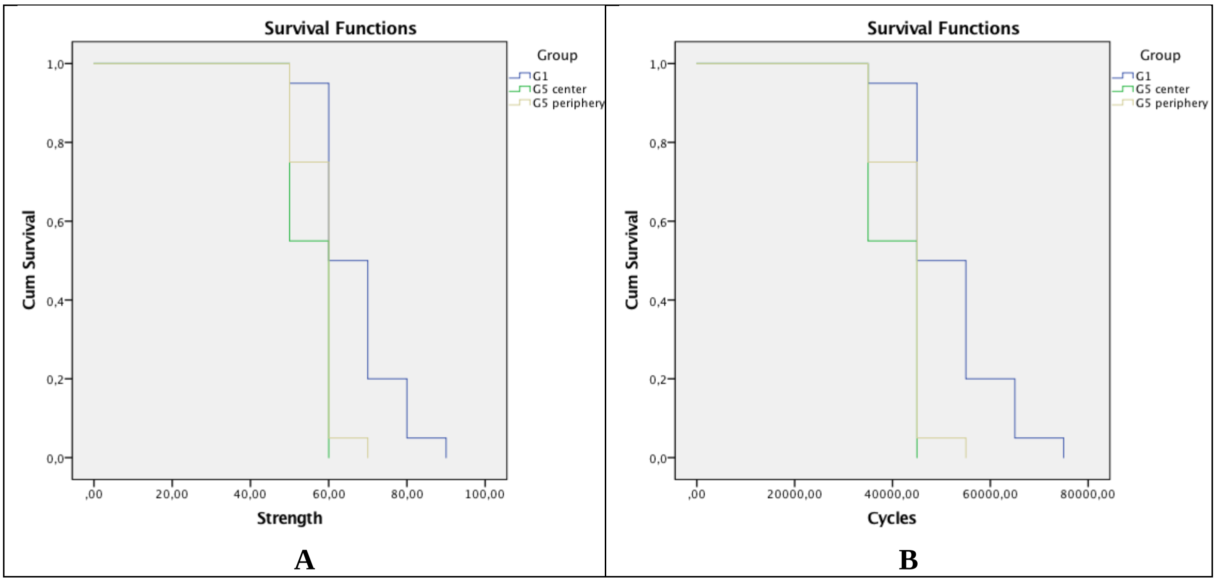


Figure 1. Disposition of the G1 (A) and G5 groups (B) inside the furnace for each firing cycle.



A **B**

Figure 2. Survival graphs obtained by Kaplan-Meier and Log-rank tests for stress to failure (**A**) and number of cycles to failure (**B**).

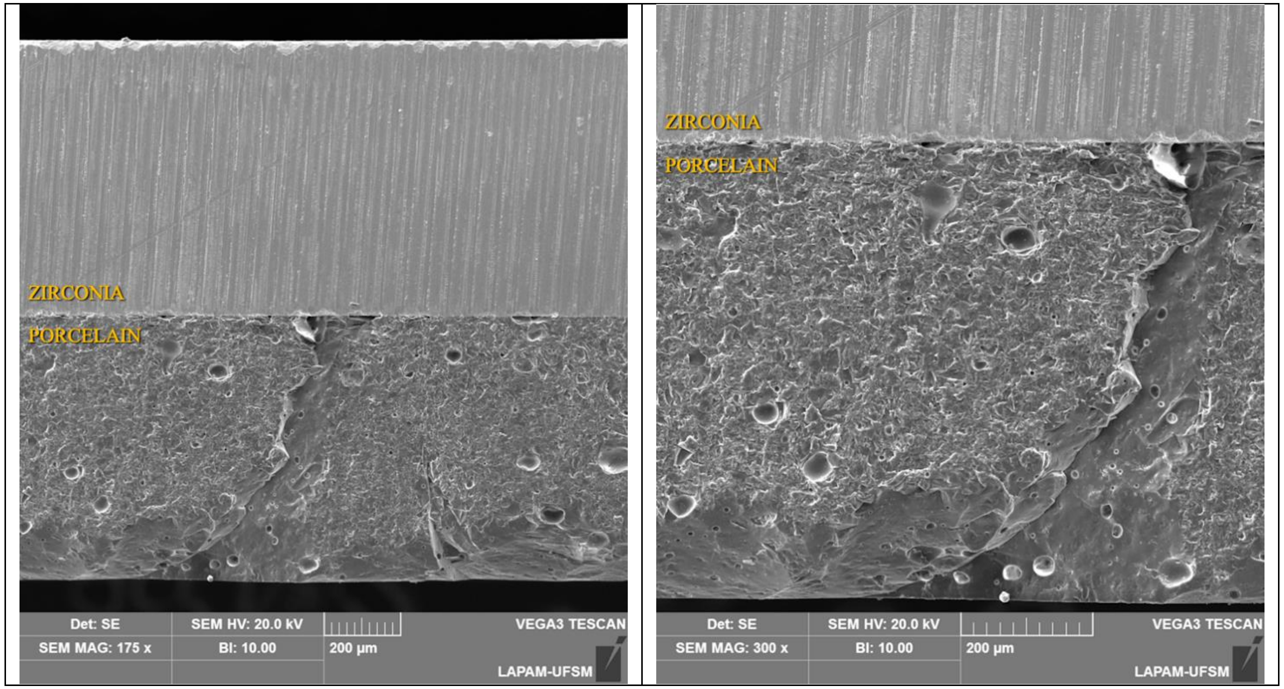


Figure 3. Scanning electron microscopy (SEM) of a representative failure pattern (specimen selected from group G5P), analyzed at 175 and 300× of magnification (from left to right respectively). Radial cracks could be seen during the fatigue test and were cut perpendicularly to this crack. It can be seen that the crack propagates trough the porcelain layer until the interface with Y-TZP, parallel to the load application direction.

3. ARTIGO 2: Influence of zirconia surface treatments of a bilayer restorative assembly on the fatigue performance

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Influence of zirconia surface treatments of a bilayer restorative assembly on the fatigue performance

Short title: Effect of zirconia surface treatments on the fatigue behavior of a bilayer assembly

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ABSTRACT

Objective: This study evaluated the influence of different surface treatments of zirconia, used to enhance bonding with the veneering porcelain, and thermocycling on the resistance to porcelain crack and delamination during fatigue test. **Methodology:** Bilayer ceramic discs were made from zirconia infrastructure blocks (IPS e.max Zircad MO, Ivoclar Vivadent – 0.7 mm thickness). Prior to porcelain application, the zirconia discs were randomized into 8 groups (n= 15), according to two factors: ‘zirconia surface treatment’ in 4 levels: *Control* - cleaning only, *Grinding* - grinding with 46 µm grain size (#4219F) diamond bur, *Air-abrasion* - air-abrasion with 45 µm aluminum oxide particles, and *Liner*- application of a ceramic liner (IPS e.max Zirliner, Ivoclar Vivadent); and ‘thermocycling’ in 2 levels: presence (performed after the veneering porcelain application; 12,000 thermal cycles; 5-55ºC) or absence. The zirconia disks were veneered with porcelain (IPS e.max Ceram, Ivoclar Vivadent – 0.7 mm; 1.4 mm total thickness disks) according to ISO 6872:2015 for biaxial flexure strength testing. Fatigue tests (step-stress approach; 20 MPa - 100 MPa; step of 10 MPa; 10,000 cycles per step; 10 Hz frequency; n= 15) were performed and the strength to failure and number of cycles for failure were recorded for Kaplan-Meier and Mantel-Cox post-hoc tests considering the two outcomes (resistance to porcelain crack and delamination). Complementary analysis of roughness, topography, crystallographic phase arranges (t-m phase content) and fractography were executed. **Results:** The surface treatment and aging by thermocycling factors did not influence the crack resistance nor the porcelain delamination resistance of bilayer specimens (always statistically similar to control group). When comparing only the performance of surface treated conditions for crack resistance outcome, the liner application depicted the worst performance in comparison to grinding and air-abrasion. For delamination, all groups were similar. **Conclusion:** Neither the surface treatment of the zirconia nor the thermocycling influences the porcelain crack resistance and the resistance to delamination of the bilayer porcelain-veneered zirconia specimens.

Keywords: Zirconia-based restoration; Yttria-stabilized tetragonal zirconia polycrystal; Surface modification; Air-abrasion; Ceramic liner; Cyclic loading; Veneer crack; Delamination.

CLINCAL RELEVANCE

Among the types of failures in bilayer ceramic assembly, delamination and chipping are the most frequent. Thus, it is important to achieve mechanisms that could increase the bond between the core and the veneering porcelain, and also the strength of the bilayer ceramic restorations, mainly when considering fatigue behavior, since cyclic loads are the most frequent stimulus in the oral environment.

1. INTRODUCTION

Nowadays, the literature supports that the use of bilayer restorations with a strong core material covered by an esthetic veneering porcelain is still the best approach to restore tooth that present unfavorable substrate color shades, such as: discolored tooth, metal base cores, among others.[1,2] On such scenario, the use of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) as infrastructure veneered with a porcelain is commonly employed.

However, several studies[3-5] have reported high rates of failure using all-ceramic bilayer systems (13% in 3 years[6]; 21% in 5 years[7]). Among the types of failures, delamination and chipping are the most frequent.[8-10] Delamination consists on a fracture that involves the interface between Y-TZP and veneer, while chipping consists on the cohesive failure of the porcelain.[3] The reason for occurrence of both delamination and chipping has been linked to several factors, such as: repeated occlusal loads during chewing,[8] low thermal diffusivity of the zirconia, residual stress generated by the difference in coefficient of thermal expansion between the infrastructure and the veneering material,[11] as also the difficult to enhance bond strength between these materials (zirconia and veneering ceramic).[12]

According to He et al. (2014),[13] some approaches have been applied to enhance the bond strength between the infrastructure and the veneering porcelain. Zirconia surface treatments as the particle air-abrasion and grinding with diamond burs, for instance, can increase the surface roughness which would generate a greater micromechanical interlocking between the core and veneering ceramic.[14] Besides the influence on bonding, it is important to consider that those treatments may leads also to a t-m phase transformation, which results in a volumetric expansion of ~4% around the superficial defects, inducing a compressive stress concentration and difficulting crack propagation, and also the introduction of defects at the surface of the material.[15-18] It may impair the mechanical properties and increase the risk of failure, if they are not completely constrained by the transformation mechanism.[19] Thus, an alternative to mechanical surface treatment is the use of a liner on the interface between veneering and zirconia, which may enhance the bond strength,[20] but it is unknown if it influences the assembly mechanical strength. Furthermore, it must be considered that the literature suggests chemical and mechanical approaches to achieve an optimal bonding and, consequently, a better mechanical performance.[21] Therefore, studies considering such scenario are still encouraged.

Is also important to emphasize that to predict the performance of multilayer ceramic assemblies is very complex and difficult task, and the interaction between the different materials usually affects the mechanical behavior during the assay.[22] So, the comprehension of the adhesion mechanism between both ceramics and its influence over the materials strength are not yet fully elucidated, remaining contradictory. Also, it is essential to evaluate the performance of all-ceramic restorations under cyclic loading, which occurs daily in the oral environment, to better understand the influence of the zirconia surface treatments on longevity of these restorations.[23,24]

So, based on the lack of consensus regarding to the best surface treatment to enhance the mechanical behavior of porcelain veneered zirconia and decrease the susceptibility of the bilayer restorative assembly to crack initiation and delamination, the present study aimed to evaluate the influence of four surface treatments (control, grinding, air-abrasion and application of a liner) in fatigue strength, number of cycles for failure, and survival rates with and without aging by thermocycling. The assumed null hypotheses were that surface treatments (1) and thermocycling (2) will not statistically influence the results of fatigue performance for porcelain crack and delamination occurrence.

2. MATERIALS AND METHODS

2.1 Specimens preparation

The materials used in the study are described in the table 1. Zirconia discs of IPS e.max Zircad MO (Lot W87787; Ivoclar Vivadent, Schaan, Liechtenstein), an yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), were made according to the ISO 6872:2015.[25] Pre-sintered zirconia blocks were shaped into cylinders by grinding with #60, #200, #400 and #600-grit silica carbide papers (SiC) (3M, Sumare, SP, Brazil) in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, IL, EUA) with constant water irrigation. After that, slices of 18 mm diameter × 1 mm thickness were obtained by using a precision cutting machine (ISOMET 1000, Buehler), obtaining the discs. Then, in order to ensure standardization of thickness and surface smoothness, the discs were ground with #600 and #1200 granulation SiC sandpapers on both sides and then cleaned in an ultrasonic bath (1440 D, 50 / 60 Hz, Odontobras, Ind. And Com. Equip. Odonto. LTDA, Ribeirao Preto, Sao Paulo, Brazil) containing 78% isopropyl alcohol for 5 min.

Finally, the zirconia discs were sintered (VITA furnace Zyrcomat 6000 MS, Vita Zahnfabrik, Germany) according to the manufacturer's recommendations (table1). At the end of the firing cycles, specimens of 15 mm diameter × 0.7 mm thickness discs were obtained. The final dimension was guaranteed using a digital micrometer (Mitutoyo absolute 500-196-20 Digital Caliper; Takatsu-Ku, Kawasaki, Kanagawa, Japan), which a variation up to 0.03 mm was considered acceptable, whereas discs with measurements higher were discarded and replaced. After that, all 120 specimens were randomly assigned into 8 groups (n= 15), according to two factors: ‘surface treatment of Y-TZP’ in 4 levels: control, grinding, air-abrasion and liner application; and ‘thermocycling’ in 2 levels: presence or absence of thermocycling, as described on table 2.

2.2 Y-TZP Surface treatments

Control group

The specimens were only cleaned with 78% isopropyl alcohol in ultrasonic bath (1440 D, 50/60 Hz, Odontobras) for 5 min previously to the veneering porcelain application.

Grinding group

Initially, a single operator was calibrated by means of a pilot test, followed by roughness analysis until a reproducible grinding process was achieved. The grinding was executed only in one side of each specimen using a diamond bur (4219F - 46 μm grain size, KG Sorensen, Cotia, SP, Brazil) with a contra-angle coupled to a corresponding low speed motor (Kavo Dental, Biberach, Germany), under constant irrigation (~ 30 mL/min) and with oscillatory movements.[26] It was used one bur for each two discs, afterwards the diamond tip was discarded and replaced with a new one (1 bur/2 specimens). For grinding thickness standardization and to ensure that the entire specimen surface was subjected to grinding, the specimens were previously marked with a permanent marking pen (Pilot, Sao Paulo, SP, Brazil) and fixed with a double-side tape (3 M from Brazil, Sumare, Sao Paulo, Brazil) to a device that assured parallelism between the specimen and diamond bur, then grinding procedure was performed until the marking pen was completely eliminated.

Air-abrasion group

The specimens were submitted to air-abrasion with aluminum oxide particles (45 μm, Polidental) with oscillatory movements at 2.8 bar pressure and 10mm distance from the devices for 10 s.[27] All the specimens were then gentle air-dried to remove any debris.

Liner application group

The IPS e.max Zirliner (Lot V11933; Ivoclar Vivadent, Schaan, Liechtenstein) was manipulated by mixing powder and respective build liquid (Lot W84644) in standardized proportions (1:1) until a creamy consistency mass was achieved, according to the manufacturer's recommendations. Then, the material was applied, with a brush on the zirconia surface until a homogeneous aspect along the surface was achieved. After the application, the specimens were fired in a specific furnace (Vacumat 6000MP; VITA, Zahnfabrik), following the manufacturer's instructions (table 1).

2.3 Roughness and topography analysis

After each treatment, specimens were cleaned with 78% isopropyl alcohol in ultrasonic bath for 5 min (1440 D, 50/60 Hz, Odontobras). Then, a micrometric analysis was performed with a rugosimeter (n= 15, Mitutoyo SJ-410, Mitutoyo), and three measurements for each specimen were carried out, on three different points, considering the parameters Ra and Rz (cut-off of 5; λC of 0.8 mm; λS of 2.5 μm), according to ISO 4287:1997.[28] Ra is the arithmetical mean of the absolute values of peaks and valleys measured from a mean plane (in μm), and Rz is the average distance between the five highest peaks and five major valleys of a surface (in μm).

Additionally, after roughness analysis, representative specimens of the 4 surface treatments were cleaned with 78% isopropyl alcohol in ultrasonic bath for 5 min (1440 D, 50/60 Hz, Odontobras), metallized and observed by scanning electron microscope (SEM; Tescan, Vega 3, Bern, Czech Republic) at 500× and 10,000× of magnification, under a high vacuum with 20.00 kV at a working distance of approximately 13.5 mm for topographic analysis.

2.4 Application of porcelain veneer

After all surface treatments, the zirconia specimens were cleaned, as prior described, and positioned inside metal templates (18 mm internal diameter and 2 mm of thickness) that guided the thickness of the porcelain layer application. The ceramic IPS e.max Ceram (Lot W42786; Ivoclar Vivadent, Schaan, Liechtenstein) was manipulated by mixing ceramic powder and build liquid according to manufacturer`s instructions (Lot W44495; Ivoclar Vivadent, Schaan, Liechtenstein) in standard proportions (1:1), until a slurry solution was obtained. Then, with the aid of a metal spatula, the porcelain was condensed over and around the surface treated zirconia. The veneer was applied around the zirconia to compensate the porcelain contraction during the firing cycle and to avoid the discover of the infrastructure. For each layer of ceramic applied inside the template, the excess liquid was gently removed with smooth absorbent paper (Softy's, Elite, Brazil) with the aid of a flat metal of the same diameter as the internal space of the template. After, the bilayer specimens were removed from the template and putted on the refractory to be fired in a specific furnace (Vacumat 6000MP; VITA, Zahnfabrik), following the manufacturer's instructions (table 1).

After firing, the porcelain layer was polished with SiC sandpapers (#200, #400, #600 and #1200 grit-size) under constant irrigation in a polishing machine (Ecomet 250 Grinder Polisher, Buehler) to achieve a smooth and shining surface. The thickness of the set was constantly inspected to respect the final dimension of the bilayer set, recommended by the manufacturer, as 1.4 mm (Y-TZP = 0.7 mm in thickness + veneer layer = 0.7mm). This dimension is also in accordance with ISO 6872:2015 guidelines for biaxial flexure strength testing.[25] Prior to the fatigue test, specimens were analyzed under optical microscopy (Stereo Discovery V20; Carl Zeiss, Göttingen, Germany) and all specimens showing bubbles or irregularities were discarded and replaced.

2.5 Thermocycling

Half of the bilayer specimens were aged in a thermocycler machine (Model 521-6D, Ethik Technology, Nova Etica Produtos e Equipamentos Cientificos LTDA, Sao Paulo, Brazil) for 12,000 thermal cycles, with temperature ranging from 5 to 55 ºC, with a dwell time of 30 s in each bath, and 2 s of transfer time between baths.

2.6 Fatigue test (piston-on-three-ball biaxial flexure assembly)

The specimens were submitted to a biaxial fatigue strength test in an electrodynamic machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, United States) by the application of a cyclic load. Each bilayer set was positioned in the device on a metal base composed of three equidistant balls. Prior to start the test, an adhesive tape was glued on the compression side of the specimen for a better piston contact and tension distribution. In addition, it prevents the spreading of fragments at the moment of fracture.[29] In addition, a piece of cellophane paper (2.5µm) was positioned on the tensile side between the specimen and the three supporting balls, also to improve the contact pressure. The set was submerged in distilled water with the porcelain side facing down (tensile tension side), and a tungsten piston with a flat cylindrical tip of 1.6mm diameter was used to apply the cyclic load on the material (ISO 6872:2015).[25]

The fatigue strength under biaxial flexure was determined for each specimen using step-stress methodology described by Collins (1993).[30] The method began by subjecting the specimens to 20 MPa at a frequency of 10Hz to 5,000 cycles, followed by increments of 10 MPa for each 10,000 cycles, which means steps of 30 MPa, 40 MPa, 50 MPa, and so on, up to 100 MPa. The specimens were tested until the first crack and then until the delamination of the porcelain or until 85,000 cycles were completed, and the step and number of cycles for both outcomes were registered. Cracks were searched by transillumination following each step of the test.[23] The number of cycles and strength data of the test was collected, both for cracking and for delamination.

2.7 Failure analysis

Failed specimens were analyzed in a stereomicroscope (Stereo Discovery V20, Carl Zeiss, Gottingen, Germany) to identify the failure characteristics and to select representative specimens to be analyzed in the SEM. Representative specimens selected for the scanning electron microscope (Tescan, Vega 3) were previously metallized and then analyzed in 32, 100 and 200× of magnification under high vacuum with 20.00 kV at a working distance of approximately 13.5mm, to detect the crack origin and the direction of crack propagation.

2.8 Phase transformation analysis (X-Ray Diffractometry - XRD)

Additional Y-TZP zirconia discs, without veneering, were made and submitted to the four surface treatments considered herein, as previously descripted, and taken to the furnace for simulation of porcelain firing (without porcelain). Then, a quantitative descriptive analysis of phase transformation using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) was performed (n= 1) to determine the relative amount of monoclinic phase content generated by each surface treatment and any potential alteration of such composition triggered by porcelain firing cycle. The porcelain veneer was not applied due to the possibility of interference in the phase transformation measurement, since the test is restricted to a superficial or near surface region, typically no more than the top few microns.[31,32]

Spectra were collected in the 2θ range of 27 to 37 degrees and 72 to 76 (where θ is the angle of incidence relative to the sample surface), with tension of 40Kv and 40 mA, at a step interval of 2 s, and step size of 0.01 degrees/step. The m-phase fraction (XM) was calculated using the Garvie & Nicholson (1972) method[33]:

$$\mathrm{Xm} = \frac{(-111)\mathrm{M}+(111)\mathrm{M}}{(-111)\mathrm{M}+(111)\mathrm{M}+(101)\mathrm{T}} \qquad \text{Eq. (1)}$$

where (−111)M and (111)M represent the intensity of the monoclinic peaks (2θ = 28° and 2θ = 31.2°, respectively) and (101)T indicates the intensity of the respective tetragonal peak (2θ = 30°). The volumetric fraction of the m-phase was calculated according to Toraya et al. (1984)[34]:

$$\mathrm{Fm} = \frac{1.311\mathrm{Xm}}{1+0.311\mathrm{Xm}} \qquad \text{Eq. (2)}$$

2.9 Statistical analysis

A statistical software was used to evaluate the roughness (Ra and Rz parameters) and the fatigue data (SPSS version 21, IBM, Chicago, IL, USA) using an $\alpha = 0.05$. As the roughness data assumed a non-parametric distribution (Shapiro-Wilk test), the data was submitted to the Kruskal-Wallis and Dunn’s post-hoc tests. The fatigue data was submitted to a survival analysis (Kaplan-Meier and Mantel-Cox post-hoc tests) considering both the crack initiation and the delamination as failure outcomes. Moreover, the survival probability on each step of the test considering each outcome was calculated.

3. RESULTS

3.1 Roughness and surface topography

The roughness analysis showed that control and air-abrasion groups were statistically similar depicting the smoother surfaces, meanwhile grinding and liner groups were statistically similar to each other and showed the rougher surfaces (Table 3). Regarding the surface topography, SEM analysis shown completely different surface patterns. At control group it clearly depicts the presence of the grains of zirconia, meanwhile all surface treatments alter such surface pattern. Despite having a similar roughness to control condition, air-abrasion group depicts a completely deformed surface, with the presence of a more irregular surface introduced by the impact of the abrading particle. A deformed surface is also observed after grinding, but with scratches being introduced in relation to the movement of the grinding tool. Liner application clearly leads to the presence of inconstant layers of material, whereas it seems that the liner concentrates in some areas and depletes in others, creating an aspect of presence of pores and high surface irregularity (Fig. 1).

3.2 Fatigue results

The survival analysis (Kaplan-Meier and Mantel-Cox post-hoc tests) pointed out that both factors considered in this study (surface treatment and aging by thermocycling) did not statistically influence the strength and number of cycles for crack and for delamination of bilayer zirconia veneered specimens, since all conditions were statistically similar to the control group (Table 3). When comparing only the surface treated conditions, it is observed that the liner application showed the worst performance for the crack initiation outcome. The survival rates generally corroborate the observations of the survival analysis, although some slight differences can be noticed in some steps. For instance, at 50 MPa or 35,000 cycles, for the crack initiation outcome, the liner group present 20% probability of survival, while the grinding group 73% and the air-abrasion group 47% (Tables 4 and 5; Fig. 2).

3.3 Fractography analysis

The fractography analysis evidenced that for all conditions considered, the failure has originated from surface or sub-surface defects in the outer surface of the porcelain layer (tensile side during fatigue test). After initiation, the crack propagated up to the zirconia core until the delamination occurrence (Fig. 3).

3.4 XRD analysis

The XRD analysis showed a presence of monoclinic phase in the grinding (7.74%) and air-abrasion (8.91%) groups. No m-phase was observed for the control and liner groups (Table 3).

4. DISCUSSION

The study findings showed no influence of different surface treatments and thermocycling on fatigue strength, number of cycles for failure, and survival rates for crack and delamination occurrence of the porcelain-veneered zirconia when the surface treated groups were compared with the control group, by that, the null hypotheses were accepted.

Even with the differences in topography, roughness and presence of monoclinic phase between groups, no one was superior to the absence of treatment (control group), when considering the crack outcome (fig. 1, table 3). It may be due the test assembly used in this study. It has been reported that the bilayer ceramic behavior under flexural strength test depends of the material on the bottom surface, so if the porcelain is under tensile stress, the set strength is similar as if it was a monolithic veneering ceramic.[22,35] Thus, the ultimate strength and mode of failure is dictate by the properties of the material at bottom, which is the porcelain in this case, and the contribution of the YTZP core results in just a modest increase of the strength.[36] For this reason, further more studies of flexural fatigue tests with different test assemblies for bilayer ceramic systems are encouraged.

Regarding the delamination outcome, all Y-TZP surface treatments groups, with or without thermocycling, were statistically similar. The chipping and delamination of veneering ceramic are some of the most frequent reasons for the failure of metal-free restorations.[8-10] The literature points that different zirconia surface treatments lead to different bond strengths and chipping/delamination occurrence on the veneering ceramics.[37,38] However, the findings of the present study show that such bonding provided by the grinding, air-abrasion and liner application surface treatments were not sufficient to generate a better fatigue performance of the bilayer ceramic system then control group, and even the survival rate for all the groups were similar (table 5).

Perhaps for a better performance in regards of crack and delamination resistance under repeated load cycles, which is the most frequent mechanical stimulus in a clinic scenario,[23,24,39] the adhesion between porcelain and framework layers should be even greater, since the achieved bonding between the zirconia core and ceramic veneer is still limited and controversial.[12,40] Even so, literature shows that, from a clinical viewpoint, maximum masticatory forces may easily achieve 300–400 N with lower average chewing forces of approx. 220 N in the molar region.[41,42] Assigning those forces to a contact area of 7–8 mm^2 (single molar tooth) results in an average chewing pressure of 27–31 MPa.[43] Thus, all the groups of the present study achieve a strength superior to 30 MPa, which is also in accordance to the ISO 6872:2015 recommendations for use as a veneer of bilayer crowns.[25]

Besides, thermocycling also did not significantly influenced the tested outcomes, which could be an indicative that it did not influenced neither the adhesion between layers, nor the defects present in the bilayer set. It may have occurred due to the aging protocol used in the present study not to be sufficiently deleterious to cause thermal expansion of the ceramics, which have different thermal coefficients.[11] Thus, in future studies maybe it is interesting to use other temperatures or the combination of mechanical and thermal stimulus at the same time.

However, when comparing the surface treatment groups with each other, a slightly worse performance was observed for the liner application in relation to the grinding and air-abrasion groups when considering the porcelain crack outcome. It can be explained due to the tetragonal to monoclinic phase transformation of the Y-TZP in the grinding (7.74%) and air-abrasion (8.91%) groups, showed by the XRD analysis (Table 3). The mechanism of zirconia phase transformation, which occurs in the presence of a mechanical stimulus,[15] is responsible for the increase of the material toughness,[44] through the volume increase of the monoclinic grains in 3-4%, generating a compression stress concentration that slows/avoids the crack propagation into the zirconia.[16]

Guazzato et al. (2004) related two peaks of stress concentration during flexural test for bilayer systems, both on the bottom surface of the core and the porcelain, when the specimens were tested with the porcelain turning down (tensile side).[36] Thus, the transformation toughening mechanism may have generated a slight decrease the zirconia’s deflection during the flexural fatigue test, and hence generating a lower stress concentration at the bottom surface of the porcelain,[45] which did not happen for the liner application group. Besides, surface topography analysis shown that the liner application leads to the presence of pores and high surface irregularities near the interface that could contribute on the mechanism of crack propagation, as the pores are small in dimension and may have been difficult to be filled by the porcelain layer (fig. 1). Thus, it could have facilitated the crack propagation towards to the interface (table 4). Grinding clearly presents a more regular topography to be filled as the scratches are less complex (fig. 1).

In addition, there is a lack of consensus in the literature, but the ceramic liner does not seem to enhance the adhesion of the Y-TZP with porcelain. The manufacturers' guides show that for pre-pigmented zirconia infrastructures, the use of liner is dispensed. So, the liner is applied basically to masking the opacity of the infrastructure, for a better esthetic condition.[46] Besides that, Fisher et al. (2010) reported that the application of a liner on Y-TZP had no significant effect on its shear bond strength with veneering ceramics,[47] and Wang et al. (2014) reported that the use of a liner reduced the bond strength of the zirconia-veneer interface, so a liner should be applied with caution.[48]

Regarding to the failure pattern, the fractography analysis showed that for all groups the crack initiation was at the bottom surface of the ceramic assembly (porcelain), which was under tensile stress during the flexural fatigue test. The crack towards to the interface, was hindered by the tougher core material and propagates within the interface until the delamination of the porcelain veneer (fig. 3), which is in accordance with previously studies using such test assembly.[22,36]

Despite the relevance of the present study findings, it is important to consider its limitations. In spite of the standardization of powder and build liquid proportions and uniform layer application, the impossibility of precise control of the liner thickness may have interfered in the results of the fatigue test, such as the test assembly used, with the porcelain on the bottom surface. However, if the porcelain was at the compressive side, the incidence of Hertzian cone cracks could happen before the delamination.[36] The study of the fatigue behavior of a ceramic bilayer assembly is still insipient in literature. The findings show that none of the zirconia surface treatments had a better performance when compared to the absence of treatment (control group), for both outcomes of the present study. Thus, for this restorative assembly, there would be no need to perform additional zirconia surface treatment prior to the application of the veneering porcelain for a better fatigue behavior. However, it is important to considering the simplify geometry of the present *in vitro* study, and in the oral environment the forces applied to the teeth are much more complex, which demands another approaches and *in vivo* studies to validate these findings.

5. CONCLUSIONS

- No zirconia surface treatment (grinding, air-abrasion and liner application) was superior to the absence of treatment (control) when the final crack resistance and delamination was considered.
- Aging by thermocycling did not influence resistance to crack and delamination of bilayer zirconia veneered specimens.

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TABLES AND FIGURES

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

Material (manufacturer)	Composition	Firing cycle
IPS e. max Zircad MO **(IVOCLAR)** Lot. W87787	ZrO_2, 3 mol% Y_2O_3, HfO_2, Al_2O_3, SiO_3, Na_2	temperature increases 5°C/min until 700°C (±130 min); remains at 700ºC for 5 min/ temperature increases 5°C/min until 1,000°C (±60 min); remains at 1,000°C for 5 min/ temperature increases 5°C/min until 1,600°C (±120 min)/ remains at 1,600°C for 5 min; cooling until 1,000; 500 and 200°C.
IPS e. max Ceram (A2) **(IVOCLAR)** Lot. W42786	SiO2, Al2O3, ZnO2, Na2O, K2O, ZrO, CaO, P2O5, fluoride and pigments	Pre-drying 403°C for 4 min; temperature increases 40°C/min (±9 min); remains at 750°C for 1 min; cooling until 200°C; vacuum for ±7 min.
IPS e. max Zirliner **(IVOCLAR)** Lot. V11933	SiO2, Al2O3, ZnO2, Na2O, K2O, ZrO, CaO, P2O5, fluoride and pigments	Pre-drying 403°C for 4 min; temperature increases 40°C/min (±14 min); remains at 960°C for 1 min; cooling until 200°C; vacuum for ±14 min.

Table 2. Experimental design.

Group Code	Surface Treatment	Thermocycling 5-55°C, 12,000 cycles	Analysis
Control	just cleaned with isopropyl alcohol	Without	- Roughness analysis (n=15) - Fatigue test for crack and delamination (n=15) - Survival rate (n=15) - X-Ray Diffractometry (n=1)
Control Aged		With	
Grinding	grinding with 4219F diamond bur	Without	
Grinding Aged		With	
Air- abrasion	blasting with 45 µm aluminum oxide particles for 10s	Without	
Air-abrasion Aged		With	
Liner	treated with IPS e.max Zirliner (Ivoclar Vivadent)	Without	
Liner Aged		With	

Table 3. Results obtained at the fatigue test for crack and for delamination occurrence (mean and 95% confidence intervals); and results from roughness analysis (mean and Standard Deviation) and XRD analysis (content of monoclinic phase).

Groups	**Crack occurrence***		**Delamination occurrence***		**Roughness analysis****		**XRD analysis**
	Strength Mean (95%CI)	**Cycles Mean (95%CI)**	**Strength Mean (95%CI)**	**Cycles Mean (95%CI)**	**Ra**	**Rz**	***m*-phase (%)**
Control	53.33 (48.78 – 57.89)^{ABC}	38,333 (33,780 – 42,886)^{ABC}	74.00 (70.27 – 77.73)^{A}	57,345 (53,852 – 60,838)^{A}	0.26 (0.06)^{A}	2.37 (0.79)^{A}	0
Control Aged	52.67 (48.19 – 57.14)^{ABC}	37,666 (33,194 – 42,138)^{ABC}	75.33 (72.10 – 78.57)^{A}	59,676 (56,441 – 62,911)^{A}	0.27 (1.12)^{A}	2.20 (0.49)^{A}	-
Grinding	58.67 (53.30 – 64.03)^{A}	42,484 (37,907 – 47,061)^{A}	74.67 (70.03 – 79.30)^{A}	58,369 (53,098 – 63,641)^{A}	1.22 (0.26)^{B}	7.03 (1.72)^{B}	7.74
Grinding Aged	54.00 (50.80 – 57.20)^{AB}	39,000 (35,799 – 42,200)^{AB}	74.00 (69.39 – 78.61)^{A}	59,000 (54,393 – 63,606)^{A}	1.26 (1.14)^{B}	7.00 (1.07)^{B}	-
Air-abrasion	54.67 (50.91 – 58.43)^{AB}	39,021 (35,809 – 42,233)^{AB}	72.00 (68.58 – 75.42)^{A}	55,769 (51,796 – 59,741)^{A}	0.27 (0.04)^{A}	2.14 (0.39)^{A}	8.91
Air-abrasion Aged	56.67 (51.73 – 61.61)^{AB}	40,443 (35,826 – 45,061)^{AB}	72.67 (67.80 – 77.53)^{A}	57,139 (52,090 – 62,189)^{A}	0.30 (0.03)^{A}	2.27 (0.22)^{A}	-
Liner	48.00 (44.08 – 51.92)^{C}	32,344 (28,306 – 36,381)^{C}	71.33 (66.70 – 75.97)^{A}	55,773 (51,300 – 60,246)^{A}	1.58 (1.85)^{B}	7.61 (2.07)^{B}	0
Liner Aged	52.67 (50.35 – 54.98)^{BC}	37,666 (35,350 – 39,983)^{BC}	73.33 (69.20 – 77.47)^{A}	57,036 (53,602 – 60,470)^{A}	1.23 (0.38)^{B}	7.09 (1.08)^{B}	-

- Distinct letters show significant statistical differences ($p < 0.05$) depicted by *Kaplan-Meier and Mantel-Cox (Log-rank) tests and by **Kruskal-Wallis and Dunn's post-hoc tests.

Table 4. Survival rates, i.e. specimens' probability to exceed the respective cycles and fatigue strength for crack occurrence, and their respective standard error measurements.

Groups	Number of cycles / Fatigue strength (MPa) for crack occurrence							
	5,000 / 20	15,000 / 30	25,000 / 40	35,000 / 50	45,000 / 60	55,000 / 70	65,000 / 80	75,000 / 90
Control	1	1	0.80 (0.10)	0.47 (0.13)	0.07 (0.06)	0	-	-
Control Aged	1	1	0.80 (0.10)	0.40 (0.13)	0.07 (0.06)	0	-	-
Grinding	1	1	0.87 (0.09)	0.73 (0.11)	0.20 (0.10)	0.13 (0.09)	0	-
Grinding Aged	1	1	0.93 (0.06)	0.47 (0.13)	0	-	-	-
Air-abrasion	1	1	0.93 (0.06)	0.47 (0.13)	0.07 (0.06)	0	-	-
Air-abrasion Aged	1	1	0.87 (0.09)	0.60 (0.13)	0.20 (0.10)	0	-	-
Liner	1	1	0.60 (0.13)	0.20 (0.10)	0	-	-	-
Liner Aged	1	1	1	0.27 (0.11)	0	-	-	-

* the sign '-' indicates absence of specimen tested on the respective step.

Table 5. Survival rates, i.e. the specimens' probability to exceed the respective cycles and fatigue strength for delamination, and their respective standard error measurements.

Groups	Number of cycles / Fatigue strength (MPa) for delamination								
	5,000 / 20	15,000 / 30	25,000 / 40	35,000 / 50	45,000 / 60	55,000 / 70	65,000 / 80	75,000 / 90	85,000 / 100
Control	1	1	1	1	0.93 (0.06)	0.40 (0.13)	0.07 (0.06)	0	-
Control Aged	1	1	1	1	1	0.47 (0.13)	0.07 (0.06)	0	-
Grinding	1	1	1	1	0.87 (0.09)	0.47 (0.13)	0.13 (0.09)	0	-
Grinding Aged	1	1	1	1	0.93 (0.06)	0.33 (0.12)	0.07 (0.06)	0.07 (0.06)	0
Air-abrasion	1	1	1	1	0.87 (0.09)	0.33 (0.12)	0	-	-
Air-abrasion Aged	1	1	1	1	0.80 (0.10)	0.33 (0.12)	0.13 (0.09)	0	-
Liner	1	1	1	0.93 (0.06)	0.87 (0.09)	0.27 (0.11)	0.07 (0.06)	0	-
Liner Aged	1	1	1	1	0.87 (0.09)	0.40 (0.13)	0.07 (0.06)	0	-

* the sign '-' indicates absence of specimen tested on the respective step.

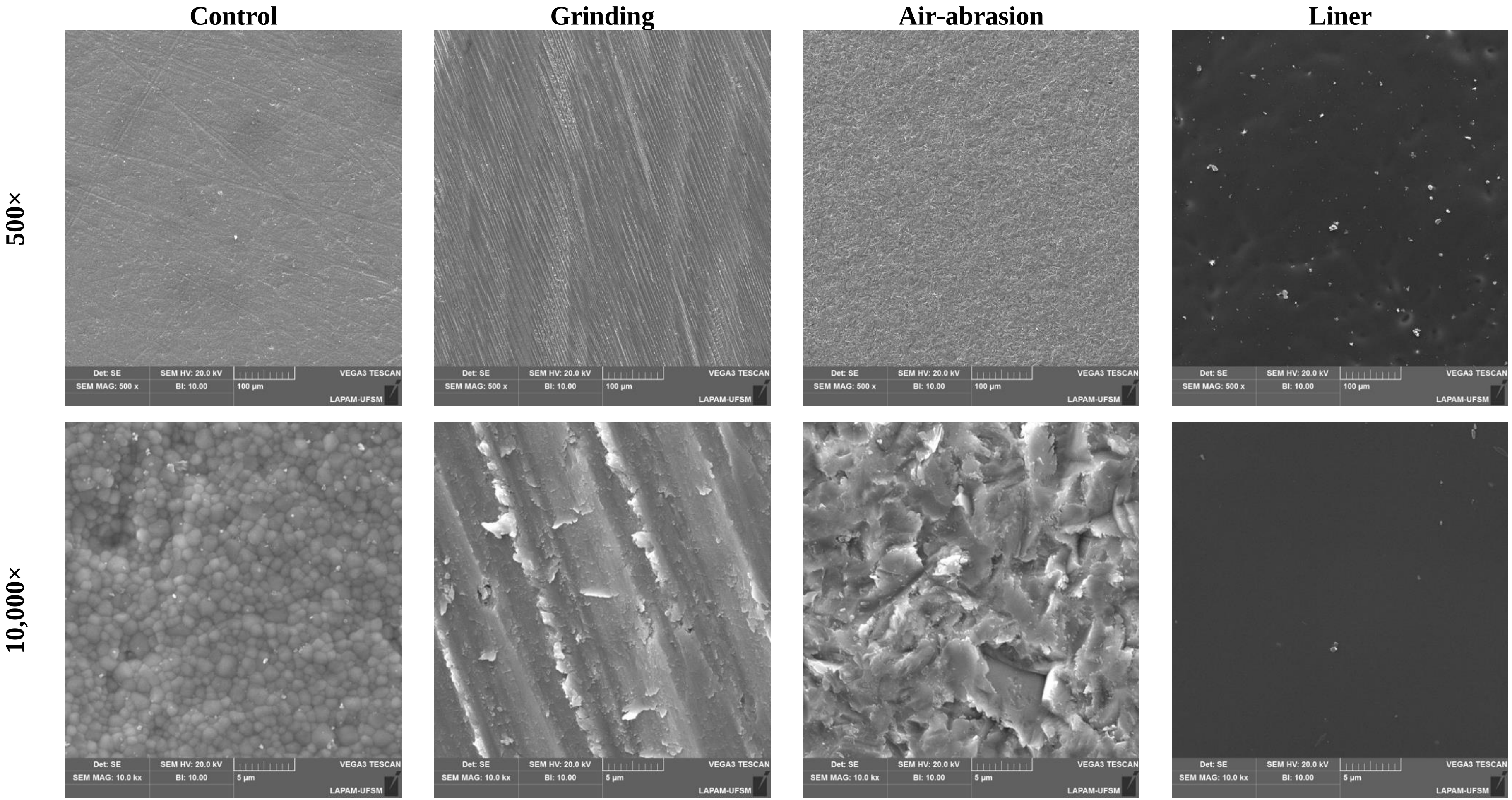

Figure 1. Scanning electron microscopy (SEM) of the zirconia surface after the 4 treatments, analyzed at 500 and 10,000× of magnification. Grinding, Air-abrasion and Liner application treatments created a more irregular topography compared to the control group. Also, the liner group showed a presence of some porosities after firing.

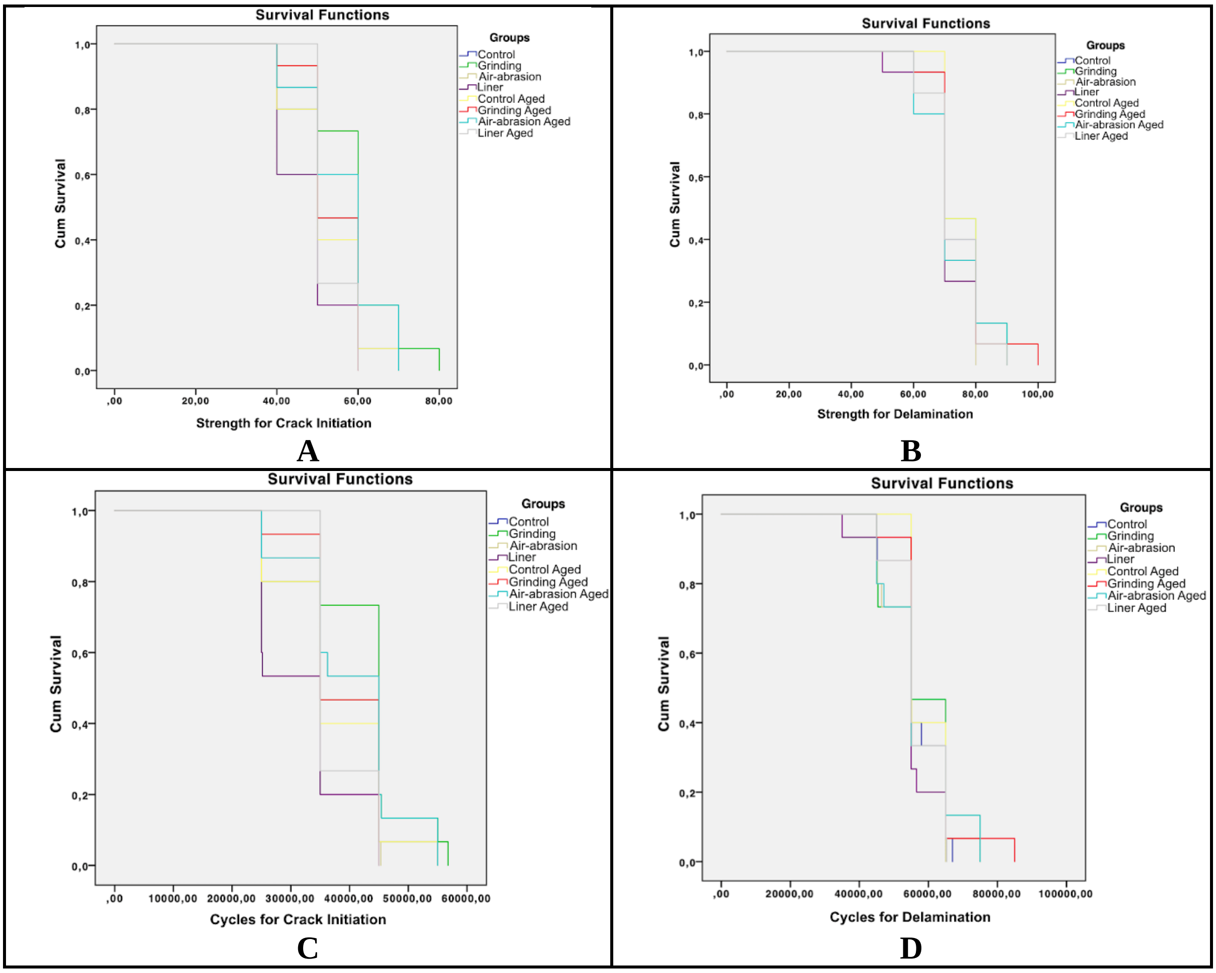

Figure 2. Survival graphs obtained by Kaplan-Meier test for fatigue strength (**A** and **B**) and number of cycles for failure (**C** and **D**) considering the two outcomes: crack (left) and delamination occurrence (right).

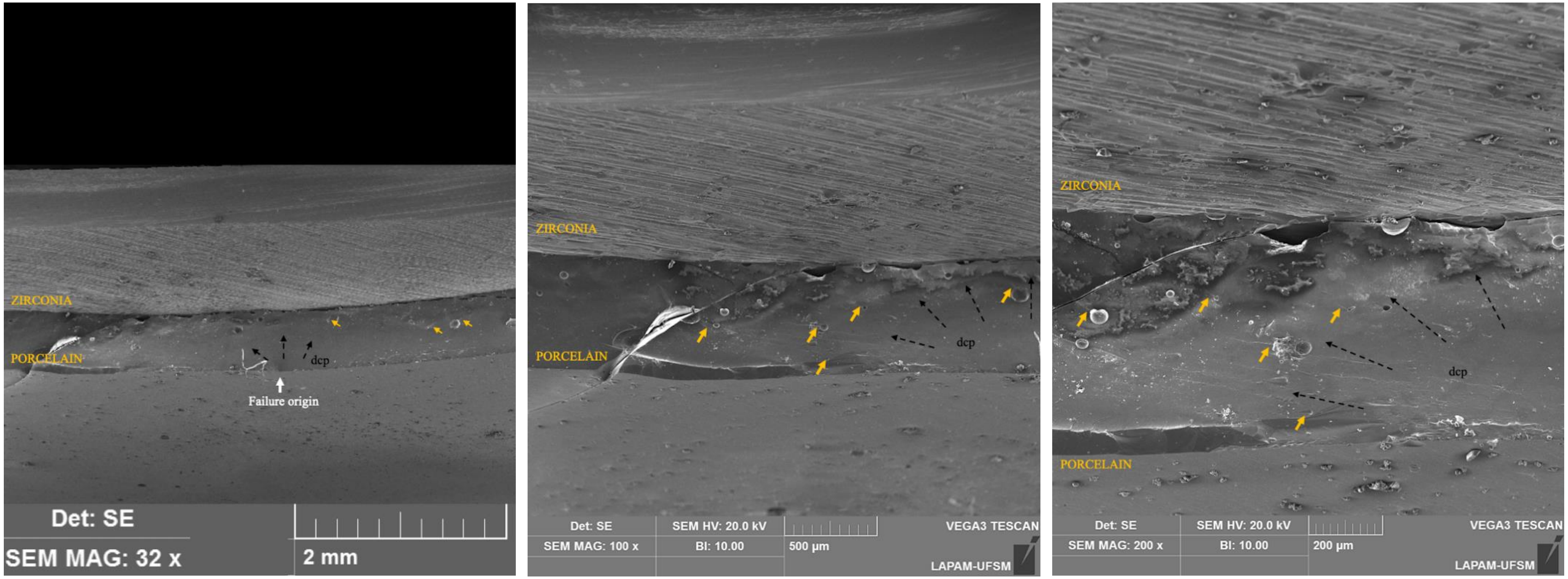

Figure 3. Scanning electron microscopies of a representative delaminated specimen selected from the grinding group and analyzed at 32 (left), 100 (middle) and 200× (right) magnification. The crack propagated from the outer surface of the porcelain which was on the bottom/tensile side during the fatigue test. The filled arrows inside the porcelain point to the wake hackles, which show the direction of crack propagation (dcp; dashed arrows).

4. DISCUSSÃO

Os sistemas bicamada cerâmica se mostram como boas opções restauradoras com prótese fixa por combinarem as propriedades mecânicas da infraestrutura com as características estéticas do material de cobertura (STAWARCZYK et al., 2013). A zircônia tetragonal policristalina estabilizada por ítrio (Y-TZP) tem tido amplo uso como material de infraestrutura devido à sua biocompatibilidade e ao seu conteúdo cristalino que garante grande resistência mecânica (KELLY, 2008), enquanto que a cerâmica feldspática se mostra como opção para material de cobertura em virtude de seu conteúdo vítreo, que garante propriedades ópticas excelentes (OH et al., 2018). No entanto, esse conjunto restaurador, como qualquer material, está sujeito a falhas, de maneira que as mais comuns para os sistemas bicamada cerâmica são o lascamento e a delaminação (RECOW et al., 2011).

A delaminação da porcelana de cobertura decorre de diferentes causas, que incluem espessura da porcelana, estímulos mecânicos contínuos aplicados e propriedades mecânicas do material utilizado (GUESS et al., 2009; REKOW et al., 2011). Variações no processamento dos materiais também tem apresentado influências sobre a resistência mecânica das cerâmicas, onde se incluem número de sinterizações, modos de resfriamento e temperatura de sinterização (CLAUS, 1989). Além disso, a resistência de união do material de cobertura com a Y-TZP tem se mostrado menor do que em relação ao metal (MATSUMOTO et al., 2013). Sendo assim, a presente dissertação buscou avaliar a influência de diferentes condições de queima da porcelana no forno (quantidade e posicionamento dos espécimes para cada ciclo), bem como diferentes tratamentos de superfície da zircônia de infraestrutura, sobre a resistência à fadiga, cor e translucidez de um sistema bicamada cerâmica.

Os resultados do artigo 1 da presente dissertação (*Fatigue behavior and colorimetric differences of a porcelain-veneered zirconia: effect of quantity and position of specimens during firing)* indicaram que a quantidade de espécimes por fornada (1 ou 5) influenciou na diferença de cor e nos resultados do teste de fadiga, de maneira que os espécimes queimados sozinhos apresentaram os melhores resultados frente aos estímulos mecânicos, enquanto que o posicionamento (centro ou periferia do refratário) não afetou a resistência à fadiga, mas gerou diferenças de cor entre os grupos. Já a translucidez não foi afetada por nenhuma das variáveis estudas.

A otimização da resistência mecânica dos materiais cerâmicos está intimamente conectada aos cuidados no manejo e processamento dos mesmos, na tentativa de reduzir a presença de defeitos internos no material (SEGHI, 1995). A literatura relata que diferentes condições de queima das cerâmicas odontológicas, como número de queimas e temperatura, podem alterar seu comportamento mecânico, visto que diferenças na energia gerada pelo forno e aplicada sobre as cerâmicas geram alterações microestruturais, em termos de sinterização das fases vítreas e cristalinas do material, impactando na resistência do material. (LENZ et al., 2002; CLAUS, 1989).

Seguindo essa linha, o estudo 1 da presente dissertação corrobora com o impacto das condições de queima sobre as propriedades finais das cerâmicas odontológicas, acrescentando que mesmo a organização espacial dos espécimes dentro do refratário pode gerar diferenças na absorção de energia e condição final de queima da porcelana. Um maior número de espécimes por ciclo de queima parece exigir mais energia do forno para que todos alcancem uma maior densidade e menor porosidade após a queima, reduzindo defeitos e melhorando a performance mecânica, o que corrobora com os achados de Tang et al. (2012). Da mesma forma, diferenças de cor foram observadas variando-se o número e também posicionamento dos espécimes no forno (centro ou periferia). A presença de mais espécimes, bem como a diferença de proximidade dos mesmos em relação à resistência do forno (acoplada nas laterais do forno em questão) pode ter levado a diferenças na liberação de óxidos metálicos presentes na porcelana, e consequentemente levando à diferenças de cor, o que está de acordo com os achados de Pires-de-Souza et al., (2009).

Os resultados do artigo 2 da presente dissertação (*Influence of zirconia surface treatments of a bilayer restorative assembly on the fatigue performance)* indicaram que nenhum dos tratamentos de superfície estudados (desgaste por ponta diamantada, jateamento com óxido de alumínio e aplicação de um *liner)* se mostraram superiores à ausência de tratamento (grupo controle) em relação ao comportamento em fadiga, tanto para o desfecho trinca quanto para delaminação da camada de porcelana. Além disso, o envelhecimento por termociclagem realizado nesse estudo também não foi capaz de agredir a interface a ponto de gerar mudanças de desempenho em fadiga entre os grupos.

Os mecanismos de união entre infraestruturas de zircônia e cerâmicas de cobertura ainda são controversos e demandam mais estudos para que se possa determinar os meios de atingir um melhor desempenho mecânico dos conjuntos bicamada (PEREIRA et al., 2019). Contudo, uma comparação entre cada tratamento mostrou que a aplicação de um *liner* apresentou os piores resultados quando comparado com o jateamento com óxido de alumínio e desgaste com ponta diamantada, considerando o desfecho “trinca da porcelana”, provavelmente devido a maior presença de porosidades e defeitos próximo da interface, e também pela ausência de fase monoclínica da zircônia, o que demonstra que não ocorreu o mecanismo de tenacificação por transformação, responsável pelo aumento da tenacidade do material (PICONI; MACCAURO, 1999; HANNINK et al., 2000). Porém, nenhum dos tratamentos demonstrou uma melhor performance em fadiga quando comparados à ausência de tratamento (grupo controle). Estudos prévios relatam que a resistência flexural de sistemas cerâmicos bicamada é determinada pelo teste utilizado (BORBA et al., 2011; GUAZZATO et al., 2004; MARCHIONATTI et al., 2019). Dessa forma, quando o teste é realizado com a porcelana voltada para baixo (lado de tração), em termos de resistência mecânica, o sistema se comporta como um material monolítico de porcelana, demonstrando assim um pior desempenho (BAJRAKTAROVA-VALJAKOVA et al., 2018; MARCHIONATTI et al., 2019). Quando o teste é realizado com a infraestrutura voltada para baixo, o sistema se comporta como um material monolítico de zircônia, o que consequentemente leva a maiores níveis de resistência flexural (KELLY, 2008; MARCHIONATTI et al., 2019). Sendo assim, no presente estudo, a resistência flexural à fadiga foi ditada principalmente pela camada de cobertura (posicionada para o lado de tração), e apesar do pequeno reforço gerado pela zircônia, esta não foi capaz de gerar diferenças significativas entre os grupos comparados ao controle (GUAZZATO et al., 2004).

Os sistemas cerâmicos bicamada ainda seguirão sendo muito utilizados na prática clínica. Dessa forma, cuidados que vão desde a confecção dos materiais, bem como com mecanismos de união entre ambos, são necessários para que se chegue a uma maior resistência mecânica da infraestrutura e estética da porcelana de cobertura. Além disso, a resistência mecânica da porcelana também sempre será importante, visto que a mesma é que sempre está exposta ao meio oral, e receberá diretamente os estímulos químicos, físicos e mecânicos (TANG et al., 2015; PREIS et al., 2011). Portanto, cada vez mais serão necessários estudos que acrescentem informações para determinação dos melhores mecanismos de confecção e união entre os materiais cerâmicos, afim de potencializar os índices de sucesso e longevidade das restaurações cerâmicas bicamada.

5. CONCLUSÃO

Com base nos achados científicos apresentados nessa dissertação, os resultados preliminares sugerem que:

- As condições de sinterização, como a quantidade de espécimes bicamada de porcelana sobre zircônia dentro do forno durante o ciclo de queima da porcelana, podem afetar a sua resistência à fadiga e cor. Além disso, o posicionamento dos espécimes também afetou a cor dos espécimes. Já a translucidez parece não ser afetada por nenhuma das condições.

- Nenhum tratamento de superfície da zircônia (desgaste com ponta diamantada, jateamento com óxido de alumínio e aplicação de um liner) se mostrou superior à ausência de tratamento (controle) quando os desfechos de resistência à trinca e delaminação em fadiga do sistema bicamada foram considerados. O envelhecimento por termociclagem também não influenciou a resistência do conjunto para os dois desfechos.

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ANEXO A - AUTHORS GUIDE FOR PUBLICATION ON DENTAL JOURNAL OF PROSTHODONTIC RESEARCH

GUIDE FOR AUTHORS

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Manuscripts should be written clearly in English. The order of the manuscript should be: Title page, abstract, key words, text, references, tables, legends, and figures. The manuscript should be typed with double spacing (not to exceed 70 characters, including spaces, on a line) about 800 words per page correspond to one page of finished makeup. All manuscripts must be covered with a title page including the title (within 25 words), type of article, an abbreviated title (within 10 words) for use as a running head and three to five key words. The authors' full and complete names, degrees, and institutions should be given on the title page, as well as full postal address, telephone/fax numbers, and e-mail address for correspondence. Define abbreviations at their first occurrence in the article: in the abstract but also in the main text after it. Ensure consistency of abbreviations throughout the article. The number of pages in the text, number of tables and figures, and the quantity of reprints desired should be stated on the bottom of the title page. The pledge statement attached to this journal must be accompanied with manuscript.

Key features of articles

JPOR_GFA_Table_20160914.jpg

Essential title page information

All manuscripts must be covered with a title page including the title (within 25 words), type of article, an abbreviated title (within 10 words) for use as a running head and three to five key words. The authors' full and complete names, degrees, and institutions should be given on the title page, as well as full postal address, telephone/fax numbers, and e-mail address for correspondence.

Acknowledgements

All contributors who do not meet the criteria for authorship as defined above should be listed in an acknowledgements section. Examples of those who might be acknowledged include persons who provided purely technical help or writing assistance or a department chair who provided only general support. Authors should disclose whether they had any writing assistance and identify the entity that funded for this assistance.

Article Type

FUNDAMENTAL RULE FOR STRUCTURE OF TEXT AND METHOD OF DESCRIPTION

Review

The length shall be no more than 8 printed pages. Reviews shall introduce and summarize a specific theme useful for the reader. It shall correctly introduce the background subject area and the outcomes of past research, and special attention shall be paid to the selection of reference literature. The presentation of strongly biased views should be avoided. It is desirable to describe the methods used to search, select, and summarize the information.

Original article

Original articles shall have high novelty leading to objective conclusions and contribute to the development of prosthodontics. The length shall be no more than 10 printed pages.

<Structure of original article>

Introduction: The background, purpose, and significance of research shall be described in understandable manner.

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Method of research (Materials and methods): The material and apparatus or method used for the research shall be clearly and concisely described so that additional tests may be performed by other persons using the same method. Also, the setup of experimental conditions, number of samples, sampling method, and statistical processing shall conform to the purpose of study.

Results (Performance): Only the objective observations shall be described; the subjective views of the authors shall be avoided. The observed results shall be indicated in tables, in principle, and values such as average and standard deviation shown jointly. Refer to "Measuring data and its treatment" described hereunder for verification of significant difference and multiple comparison.

Discussion: Adequate elaboration shall be made on the methods and results referring to the relevant literature, and arguments and opinions should follow a logical procedure. Furthermore, the discussion shall be focused on the purpose of the research; comprehensive discussion of irrelevant material shall be avoided. In addition, discussion shall be made not only of the results obtained but also on their significance for prosthodontics.

Conclusion (Summary): Only obtained results shall be described accurately and concisely.

Case report

Proposals for the modification of diagnostic methods, treatment methods, and treatment skill that are considered established in the field of prosthodontics as well as reports of rare case examples, unexpected complications, or unexpected development of disease may fall in this category. Cases shall be concretely and concisely described so as to inform readers in their treatment of patients. The length shall not exceed 6 printed pages, in principle.

<Structure of case report>

Introduction: The introduction shall state the positioning of the case in dental clinics and its characteristics; describe the problems identified and clearly explain why the case is worthy of reporting.

Outline of the case: Concrete and concise description shall be made on the outline of the case such as examination, findings of inspection, diagnosis, and therapeutic policy, treatment, and progress. Subtitles may be used to help the understanding of readers.

Discussion: Refer to the related and important literature and discuss the case to be reported. Discuss the characteristics of the case, treatment, and progress, and refer to the prosthodontic positioning of the case.

Conclusion: The conclusion shall include helpful points for readers in their own clinical practice.

Technical procedure

Introduction of new clinical operation method, research method, and use method of materials may be submitted, and the length shall not exceed 6 printed pages, in principle. Acceptable articles shall not introduce new products or mere technical information but shall describe novel effectiveness of treatment, long-term stability, or performance of equipment enhanced due to improvement proposed by the author.

<Structure of technical introduction procedure>

Introduction: Clearly describe the purpose of technology (operation method, research method, use method, etc.) to be introduced.

Materials and methods: Describe clearly, systematically, and understandably the materials, equipment, use method, methodology, and operational method.

Difference from conventional methods: Summarize and describe concisely the main points of the new contrivance and novelty that are different from conventional methods. Especially, clear description shall be made on the development or contrivances made by the author.

Effect or performance: Clearly describe the improvement in effectiveness and safety resulting from the improvement introduced. Also, description shall be made on the merits and demerits of the operation method to be introduced.

Conclusion: Description shall be made only of the obtained conclusions about the new contrivance and novelty different from conventional methods as well as the points improved thereby and its effectiveness.

Letter to the Editor

A Letter to the Editor should be in one of the following forms of presentation:

1. A brief report of research findings appropriate for the scope of Journal of Prosthodontic Research and of special interest to the readers.
2. An article that may not cover standard research but that is of general interest to the broad readership of Journal of Prosthodontic Research (e.g., technical tips and brief procedures for prosthodontic treatments).
3. A discussion that comment on a recent Journal of Prosthodontic Research article.

As with other articles, a Letter to the Editor may be subject to peer review. Typically, it will contain about 1,000 words of text, figure legends, and references. It will have no abstract, and the references are limited to 10. It need not follow the usual classification of sections, such as materials and methods.

A Letter to the Editor usually contains 1 or 2 figures or tables.

FULL-LENGTH PAPERS

In the case of full-length papers, the following format is recommended:

Abstract

Briefly state a summary of the text, within 250 words, as a structured abstract. Review: Purpose, Study selection, Results, Conclusions Original article: Purpose, Methods, Results, Conclusions Case Report: Patients, Discussion, Conclusions Technical Procedure: Purpose, Methods, Conclusions

Introduction

Clearly and briefly describe the background and the rational objective of the study, with a review of earlier publications. It is recommended that previous studies described should be the most relevant. Avoid exhaustive review of the literature.

Materials and methods

Clearly describe the subjects and sample size, the experimental procedures, and apparatus (manufacturer's name and address) used in the study. In the case of experiments on human and animal subjects, give an account that the methods are regarded as ethically sound. In the event of an original design, the details should be provided. Otherwise, references accompanied by sufficient information for interdisciplinary evaluation will suffice. The type of statistical analysis used, as well as commercial software, must be stated in this section. Do not include discussion in this section. Describe precisely all drugs and chemicals used, including generic names, doses, and routes of administration.

Results

Present the essential results in the text, in a clear and concise manner. Use tables and figures to compare and contrast the findings. Do not repeat in the text all the detailed data in the tables and figures. Do not include discussion in this section. In the statistical analysis, please define the probability values and show that the differences reported were found to be statistically significant.

Discussion

Demonstrate the objective reliability of the results, as well as the property and limitation of the experimental procedures and subjects used. Point out the significance and the limitation of the study, including implications for future research. Describe and evaluate the results with a scientifically critical view, and discuss your findings in the context of other publications,

including opposing views. The introduction or details of the results should not be repeated in this section. Subjective comments can
only be made in this section; however, speculation must be identified as such. Link the conclusions with the objectives of the study, as stated in the introduction.

Acknowledgements

Acknowledgments, a scientific meeting at which the data were presented, the sources of funding for the study, and/or any other special mention, may be stated before the references section.

References

Journal of Prosthodontic Research uses Vancouver reference style.

Citation in text

Please ensure that every reference cited in the text is also present in the reference list (and vice versa). Any references cited in the abstract must be given in full. Unpublished results and personal communications are not recommended in the reference list, but may be mentioned in the text. If these references are included in the reference list they should follow the standard reference style of the journal and should include a substitution of the publication date with either 'Unpublished results' or 'Personal communication'. Citation of a reference as 'in press' implies that the item has been accepted for publication.

Reference links

Increased discoverability of research and high quality peer review are ensured by online links to the sources cited. In order to allow us to create links to abstracting and indexing services, such as Scopus, CrossRef and PubMed, please ensure that data provided in the references are correct. Please note that incorrect surnames, journal/book titles, publication year and pagination may prevent link
creation. When copying references, please be careful as they may already contain errors. Use of the DOI is highly encouraged.

A DOI is guaranteed never to change, so you can use it as a permanent link to any electronic article. An example of a citation using DOI for an article not yet in an issue is: VanDecar J.C., Russo R.M., James D.E., Ambeh W.B., Franke M. (2003). Aseismic continuation of the Lesser Antilles slab beneath northeastern Venezuela. Journal of Geophysical Research, https://doi.org/10.1029/2001JB000884. Please note the format of such citations should be in the same style as all other references in the paper.

Web references

As a minimum, the full URL should be given and the date when the reference was last accessed. Any further information, if known (DOI, author names, dates, reference to a source publication, etc.), should also be given. Web references can be listed separately (e.g., after the reference list) under a different heading if desired, or can be included in the reference list.

Data references

This journal encourages you to cite underlying or relevant datasets in your manuscript by citing them in your text and including a data reference in your Reference List. Data references should include the following elements: author name(s), dataset title, data repository, version (where available), year, and global persistent identifier. Add [dataset] immediately before the reference so we can properly identify it as a data reference. The [dataset] identifier will not appear in your published article.

References in a special issue

Please ensure that the words 'this issue' are added to any references in the list (and any citations in the text) to other articles in the same Special Issue.

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If EndNote is used, this journal has standard templates available in key reference management packages (http://endnote.com/downloads/style/journal-prosthodontic-research).

Reference style

Text: Indicate references by number(s) in square brackets in line with the text. The actual authors can be referred to, but the reference number(s) must always be given.

List: Number the references (numbers in square brackets) in the list in the order in which they appear in the text.

Examples:

Reference to a journal publication:

[1] Van der Geer J, Hanraads JAJ, Lupton RA. The art of writing a scientific article. J Sci Commun 2010;163:51–9. https://doi.org/10.1016/j.Sc.2010.00372.

Reference to a journal publication with an article number:

[2] Van der Geer J, Hanraads JAJ, Lupton RA. The art of writing a scientific article. Heliyon. 2018;19:e00205. https://doi.org/10.1016/j.heliyon.2018.e00205

Reference to a book:

[3] Strunk Jr W, White EB. The elements of style. 4th ed. New York: Longman; 2000.

Reference to a chapter in an edited book:

[4] Mettam GR, Adams LB. How to prepare an electronic version of your article. In: Jones BS, Smith RZ, editors. Introduction to the electronic age, New York: E-Publishing Inc; 2009, p. 281–304.

Reference to a website:

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[5] Cancer Research UK. Cancer statistics reports for the UK, http://www.cancerresearchuk.org/ aboutcancer/statistics/cancerstatsreport/; 2003 [accessed 13 March 2003].

Reference to a dataset:

[dataset] [6] Oguro M, Imahiro S, Saito S, Nakashizuka T. Mortality data for Japanese oak wilt disease and surrounding forest compositions, Mendeley Data, v1; 2015. https://doi.org/10.17632/ xwj98nb39r.1.

Note shortened form for last page number. e.g., 51–9, and that for more than 6 authors the first 6 should be listed followed by 'et al.' For further details you are referred to 'Uniform Requirements for Manuscripts submitted to Biomedical Journals' (J Am Med Assoc 1997;277:927–34) (see also Samples of Formatted References).

Journal abbreviations source

Journal names should be abbreviated according to the List of Title Word Abbreviations.

Research data

This journal encourages and enables you to share data that supports your research publication where appropriate, and enables you to interlink the data with your published articles. Research data refers to the results of observations or experimentation that validate research findings. To facilitate reproducibility and data reuse, this journal also encourages you to share your software, code, models, algorithms, protocols, methods and other useful materials related to the project. Below are a number of ways in which you can associate data with your article or make a statement about the availability of your data when submitting your manuscript. If you are sharing data in one of these ways, you are encouraged to cite the data in your manuscript and reference list. Please refer to the "References" section for more information about data citation. For more information on depositing, sharing and using research data and other relevant research materials, visit the research data page.

Data linking

If you have made your research data available in a data repository, you can link your article directly to the dataset. Elsevier collaborates with a number of repositories to link articles on ScienceDirect with relevant repositories, giving readers access to underlying data that gives them a better understanding of the research described.

There are different ways to link your datasets to your article. When available, you can directly link your dataset to your article by providing the relevant information in the submission system. For more information, visit the database linking page.

For supported data repositories a repository banner will automatically appear next to your published article on ScienceDirect.

In addition, you can link to relevant data or entities through identifiers within the text of your manuscript, using the following format: Database: xxxx (e.g., TAIR: AT1G01020; CCDC: 734053; PDB: 1XFN).

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Tables

Tables should be submitted online as a separate file, bear a concise title, and be numbered with Arabic numerals. Tables should be cited in the text. Column headings should be brief, but sufficiently explanatory. Standard abbreviations of units of measurement should be added between parentheses. Vertical lines should not be used to separate columns.

Figures

The number of figures used to present data essential to illustrate or prove a point should be kept to a minimum. Reference should be made in the text to each illustration. Figures will be reduced to fit to the size of one column (7.5 cm) or two columns (16 cm), and any lettering should be large enough to allow this reduction without becoming illegible. Each figure should be accompanied by a title and an explanatory legend on a separate page called Legends to Figures. There should be sufficient experimental details in the legend to make the figure intelligible without reference to the text. Legends to Figures should be typed double-spaced, in numerical order, on a separate page. Photographs should be as high in contrast as possible. Indicate the magnification of photomicrographs in bar scales on the illustration itself instead of numerical magnification factors. Make sure you use uniform lettering and sizing of your original artwork. Save text in illustrations as "graphics" or enclose the font. Only use the following fonts in your illustrations: Arial, Courier, Helvetica, Times, Symbol. Number the illustrations according to their sequence in the text. Use a logical naming convention for your artwork files. Provide all illustrations as separate files. Provide captions to illustrations separately. Produce images near to the desired size of the printed version.

AFTER ACCEPTANCE

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Corresponding authors will receive an e-mail with a link to our online proofing system, allowing annotation and correction of proofs online. The environment is similar to MS Word: in addition to editing text, you can also comment on figures/tables and answer questions from the Copy Editor. Web-based proofing provides a faster and less error-prone process by allowing you to directly type your corrections, eliminating the potential introduction of errors. If preferred, you can still choose to annotate and upload your edits on the PDF version. All instructions for proofing will be given in the e-mail we send to authors, including alternative methods to the online version and PDF.

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ANEXO B - AUTHORS GUIDE FOR PUBLICATION ON OPERATIVE DENTISTRY

Manuscript Submission (Author Guidelines)

General Requirements

Operative Dentistry requires electronic submission of all manuscripts. All submissions must be sent to Operative Dentistry using the Allen Track upload site. A mandatory and nonrefundable $50.00 fee is required at submission.

Your manuscript will only be considered officially submitted after it has been approved through our initial quality control check, and any quality problems have been resolved. You will have 6 days from when you start the process to submit and approve the manuscript. After the 6 days limit, if you have not finished the submission, your submission may be removed from the server. You are still able to submit the manuscript, but you must start from the beginning. Be prepared to submit the following manuscript files in your upload:

- A Laboratory or Clinical Research Manuscript file must include:
  - a title
  - a running (short) title
  - a clinical relevance statement
  - a concise summary (abstract)
  - introduction, methods & materials, results, discussion and conclusion
  - references (see Below)
- The manuscript body MUST NOT include any:
  - Author identifying information such as:
    - Authors names or titles
    - Acknowledgements
    - Correspondence information
    - Response to reviewer files should also NOT include any author identifying information, such as a signature at the end, etc.
  - Figures
  - Graphs
  - Tables
- An acknowledgement, disclaimer and/or recognition of support (if applicable) must be uploaded as a separate file and uploaded as miscellaneous material.
- Appendix material that you would like us to publish electronically with your article, but not as part of your printed manuscript (such as indices, supplemental tables, etc.), should be submitted as supplemental material. It will not be typeset, and will appear exactly as you provide to Operative Dentistry. References submitted as part of supplemental material should appear in our preferred reference format. Supplemental material is viewable by the reviewers, and so SHOULD NOT contain any author identifiable information.
- All figures, illustrations, graphs and tables must also be provided as individual files. Figures should be submitted without figure letters or numbers within the image itself, these designations will be added by the journal staff as needed. All Figures should be high-resolution images, which are used by the editor in the actual typesetting of your manuscript. Please refer to the instructions below for acceptable formats and sizes.
- All other manuscript types use this template, with the appropriate changes as listed below.
- When figures of identifiable individuals are submitted, the author must verify that they have received releases from the individual or guardian to use said figure. Eye blocks are no longer sufficient to anonymize an individual. Eye blocks may still be used, but a release will still be required.

- ALL studies using human tissue must have an accompanying Institutional Review Board (IRB) statement – it must indicate that either the board has approved the study, or that the study is exempted from approval. There are no exceptions to this policy.
- All studies using animal tissue must have an accompanying approval from the appropriate ethics board.
- All manuscripts reporting on a Clinical Trial must indicate that the trial information was submitted to a public Clinical Trial Registry. A URL of where the trial appears in a registry is required to be submitted with the manuscript.

Complete the online form (which includes complete author information, copyright release and conflict of interest), and select the files you would like to send to Operative Dentistry. Manuscripts that do not meet our formatting and data requirements listed below will be sent back to the corresponding author for correction.

Important Information

- All materials submitted for publication must be submitted exclusively to Operative Dentistry.
- The editor reserves the right to make literary corrections.
- Currently, color will be provided at no cost to the author if the editor deems it essential to the manuscript. However, we reserve the right to convert to gray scale if color does not contribute significantly to the quality and/or information content of the paper.
- The author(s) retain(s) the right to formally withdraw the paper from consideration and/or publication for any reason up to the submission of the final paper to our press vendor for publication.
- International authors whose native language is not English must have their work reviewed by a native English speaker prior to submission.
    - o Manuscripts that are rejected before peer-review for English correction should be entered as a new manuscript upon resubmission. In the manuscript comments box the comment, “this is a resubmission of manuscript number XX-XXX” should be noted.
    - o Manuscripts that are rejected after peer-review are not eligible for resubmission.
    - o Manuscripts that have major revisions requested (i.e. For English correction) are entered as a resubmission of the original article.
- Spelling must conform to the American Heritage Dictionary of the English Language, and SI units for scientific measurement are preferred.
- While we do not currently have limitations on the length of manuscripts, we expect papers to be concise; authors are also encouraged to be selective in their use of figures and tables, using only those that contribute significantly to the understanding of the research.
- Acknowledgement of receipt is sent automatically upon acceptance through quality control. This may take up to 7 days. If you do not receive such an acknowledgement, please check your author homepage at http://jopdent.allentrack.net if the paper does not appear there please resend your paper.

IMPORTANT: Please add our e-mail address to your address book on your server to prevent transmission problems from spam and other filters. Also make sure that your server will accept larger file sizes. This is particularly important since we send page-proofs for review and correction as .pdf and/or .doc(x) files.

Manuscript Type Requirements

All Manuscripts

CORRESPONDING AUTHOR must provide a WORKING / VALID e-mail address which will be used for all communication with the journal. *NOTE: Corresponding authors MUST update their profile if their e-mail or postal address changes. If we cannot contact authors within seven days, their manuscript will be removed from our publication queue.

AUTHOR INFORMATION must include:

- full name of all authors
- complete mailing address for each author
- valid email address for each author
- degrees (e.g. DDS, DMD, PhD)
- affiliation (e.g. Department of Dental Materials, School of Dentistry, University of Michigan)

MENTION OF COMMERCIAL PRODUCTS/EQUIPMENT must include:

- full name of product
- full name of manufacturer
- city, state and country of manufacturer

MANUSCRIPTS must be provided as Word for Windows files. Files with the .doc and .docx extensions are accepted. TABLES may be submitted as either Word (.doc and .docx) or Excel (.xls and .xlsx) files. All tables must be legible, with fonts being no smaller than 7 points. Tables have the following size limitations: In profile view a table must be no larger than 7 x 9 inches; landscape tables should be no wider than 7 inches. It is the Editor's preference that tables not need to be rotated in order to be printed, as it interrupts the reader's flow.

ILLUSTRATIONS, GRAPHS AND FIGURES must be provided as TIFF or high resolution JPEG files with the following parameters:

- line art (and tables that are submitted as a graphic) must be sized with the short edge being no shorter than 5 inches. It should have a minimum resolution of 600 dpi and a maximum resolution of 1200 dpi. This means the shortest side should be no smaller than 3000 pixels.
- gray scale/black & white figures must be sized with the short edge being no shorter than 5 inches. It should have a minimum resolution of 300 dpi and a maximum of 400 dpi. This means the shortest side should be no smaller than 1500 pixels.
- color figures and photographs must be sized with the short edge being no shorter than 3.5 inches. It should have a minimum resolution of 300 dpi and a maximum of 400 dpi. This means that the shortest side should be no smaller than 1050 pixels.

Other Manuscript Type – Additional Requirements

CLINICAL TECHNIQUE/CASE STUDY MANUSCRIPTS must include as part of the narrative:

- a running (short) title
- purpose
- description of technique
- list of materials used
- potential problems
- summary of advantages and disadvantages
- references (see below)

LITERATURE AND BOOK REVIEW MANUSCRIPTS must include as part of the narrative:

- a running (short) title
- a clinical relevance statement based on the conclusions of the review
- conclusions based on the literature review…without this, the review is just an exercise and will not be published
- references (see below)

References

REFERENCES must be numbered (superscripted numbers) consecutively as they appear in the text and, where applicable, they should appear after punctuation.

The reference list should be arranged in numeric sequence at the end of the manuscript and should include:

1. Author(s) last name(s) and initial (ALL AUTHORS must be listed) followed by the date of publication in parentheses.
2. Full article title.
3. Full journal name in italics (no abbreviations), volume and issue numbers and first and last page numbers complete (i.e. 163-168 NOT attenuated 163-68).
4. Abstracts should be avoided when possible but, if used, must include the above plus the abstract number and page number.
5. Book chapters must include chapter title, book title in italics, editors' names (if appropriate), name of publisher and publishing address.
6. Websites may be used as references, but must include the date (day, month and year) accessed for the information.
7. Papers in the course of publication should only be entered in the references if they have been accepted for publication by a journal and then given in the standard manner with "In press" following the journal name.
8. DO NOT include unpublished data or personal communications in the reference list. Cite such references parenthetically in the text and include a date.
9. References that contain Crossref.org's DOIs (Digital Object Identifiers) should always be displayed at the end of the reference as permanent URLs. The prefix http://dx.doi.org/ can be appended to the listed DOI to create this URL. i.e. http://dx.doi.org/10.1006/jmbi.1995.0238

Reference Style Guide

- Journal article-two authors: Evans DB & Neme AM (1999) Shear bond strength of composite resin and amalgam adhesive systems to dentin American Journal of Dentistry 12(1) 19-25.
- Journal article-multiple authors: Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin Critical Review of Oral and Biological Medicine 8(3) 306-335.
- Journal article: special issue/supplement: Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry Operative Dentistry (Supplement 6) 119-144.
- Abstract: Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003) Comparative study on adhesive performance of functional monomers Journal of Dental Research 82(Special Issue B) Abstract #0051 p B-19.
- Corporate publication: ISO-Standards (1997) ISO 4287 Geometrical Product Specifications Surface texture: Profile method – Terms, definitions and surface texture parameters Geneve: International Organization for Standardization 1st edition 1-25.
- Book-single author: Mount GJ (1990) An Atlas of Glass-ionomer Cements Martin Duntz Ltd, London.
- Book-two authors: Nakabayashi N & Pashley DH (1998) Hybridization of Dental Hard Tissues Quintessence Publishing, Tokyo.
- Book-chapter: Hilton TJ (1996) Direct posterior composite restorations In:
Schwarts RS, Summitt JB, Robbins JW (eds) Fundamentals of Operative Dentistry Quintessence, Chicago 207-228.
- Website-single author: Carlson L (2003) Web site evolution; Retrieved online July 23, 2003 from: http://www.d.umn.edu/~lcarlson/cms/evolution.html
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