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LUCAS DO NASCIMENTO TAVARES

Análise estrutural e mecânica de cerâmicas vítreas reforçadas por dissilicato de lítio

Tese apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia, como requisito parcial para obtenção do Título de Doutor em Odontologia na Área de Concentração de Clínica Odontológica Integrada

Uberlândia, Julho de 2021

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Iniciando os trabalhos o presidente da mesa, Dr Luís Henrique Araújo Raposo, apresentou a Comissão Examinadora e o candidato(a), agradeceu a presença do público, e concedeu ao Discente a palavra para a exposição do seu trabalho. A duração da apresentação do Discente e o tempo de arguição e resposta foram conforme as normas do Programa.

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“Gente que não tem dúvida só é capaz de repetir”

Mario Sérgio Cortella

SUMÁRIO

RESUMO

RESUMO

As cerâmicas vítreas reforçadas por dissilicato de lítio, consistem em um dos materiais mais importantes para as reabilitações odontológicas, devido a sua excelente estabilidade química e física, sendo uma das cerâmicas odontológicas mais utilizadas em diversos tipos de reabilitações orais. Após o fim patente que a empresa desenvolvedora do material possuía, outras empresas passaram a fornecer novas versões deste material, sendo que esses fabricantes sugerem que estas novas cerâmicas apresentam propriedades estruturais, morfológicas e mecânicas adequadas e compatíveis para o uso em sistemas CAD/CAM. Sendo assim, esta tese de doutorado possuí 4 objetivos específicos: **Objetivo específico 1:** O objetivo do presente estudo foi comparar duas marcas comerciais distintas de cerâmicas vítreas reforçadas por dissilicato de lítio para uso em sistema CAD/CAM (IPS e.max CAD e Rosetta SM), avaliando suas propriedades estruturais, morfológicas e mecânicas. **Objetivo específico 2:** O objetivo do presente estudo foi desenvolver e validar uma metodologia para medição do tamanho médio dos cristais presentes em cerâmicas odontológicas e sua porcentagem utilizando imagens obtidas por meio da microscopia eletrônica de varredura (MEV). **Objetivo específico 3:** Este estudo objetivou comparar 4 marcas comerciais de cerâmicas reforçadas por dissilicato de lítio para CAD/CAM, IPS E.max (Ivoclar Vivadent), Rosetta SM (Rosetta SM, Hass), T-lítio (Talmax) e IRIS (Mainland), avaliando suas propriedades estruturais, morfológicas e mecânicas. **Objetivo específico 4:** Nesta descrição de caso clínico foi feito o relato de situação limítrofe na qual foi realizada a a substituição de coroas metalo-cerâmicas insatisfatórias dos elementos 12, 11, 21 e 22, por coroas cerâmicas reforçadas por dissilicato de lítio em paciente com mordida aberta anterior esquelética.

Palavras chaves: Cerâmicas odontológicas; dissilicato de lítio; propriedades físicas

ABSTRACT

ABSTRACT

Lithium disilicate reinforced ceramics are one of the most important materials for dental rehabilitation, due to its excellent physical and chemical stability. Nowadays, lithium disilicate glass-reinforced ceramics are frequently selected as indirect restorative material for all-ceramic restorations. After the end of the patent, other companies started to produce new versions of this material, and the manufacturers suggest that these ceramics have adequate and compatible structural, morphological, and mechanical properties for CAD/CAM applications. Therefore, this study was divided into 4 specific objectives: **Objective 1:** The aim of this study was to compare two commercial brands of lithium disilicate glass-reinforced ceramics for CAD/CAM system (IPS e.max CAD and Rosetta SM), evaluating their structural, morphological and mechanical properties; **Objective 2:** The objective of this study was to present and validate a methodology for measuring the average size of ceramic crystals and their percentage using images obtained by scanning electron microscopy (SEM). **Objective 3:** To compare 4 distinct commercial lithium disilicate glass-reinforced ceramics brands, IPS E.max (Ivoclar Vivadent), Rosetta SM (Rosetta SM, Hass), T-lithium (Talmax) and IRIS (Mainland), by evaluating their structural, morphological and mechanical properties. **Objective 4:** In this clinical case report, a limit situation is shown with the replacement of unsatisfactory metal-ceramic crowns in the elements 12, 11, 21 and 22 by all-ceramic lithium disilicate glass-reinforced crowns, in a patient with skeletal anterior open bite.

Keywords: Ceramics; Lithium disilicate; Physical Properties.

INTRODUÇÃO E REFERENCIAL TEÓRICO

1. INTRODUÇÃO E REFERENCIAL TEÓRICO

A manutenção dos dentes naturais e a reabilitação de dentes comprometidos tornou-se uma grande preocupação na maioria dos países desenvolvidos. Junto a este contexto, houve grande avanço dos materiais restauradores empregados na odontologia, tanto os de uso direto quanto de uso indireto (Souza J et al., 2021; Kreidler et al., 2008). As cerâmicas surgiram a partir da mistura de caulim (argila), quartzo (sílica em forma cristalina) e feldspato (uma mistura de potássio com silicatos de alumínio e sódio), que por meio da queima em altas temperaturas, se fundem incompletamente (Zarone F et al., 2016). Por definição, as cerâmicas odontológicas são consideradas como “qualquer produto feito essencialmente de material inorgânico não metálico preparado pela queima em alta temperatura para alcançar as propriedades desejáveis” (Sakaguchi RL et., 2012). E mais recentemente, a American Dental Association atualizou esta definição para os termos cerâmica/porcelana: “materiais prensados, sinterizados, polidos ou usinados contendo compostos refratários predominantemente inorgânicos, incluindo porcelanas, vidros, cerâmicas e vitrocerâmicas.”

As restaurações metalocerâmicas apresentam excelente estabilidade com o passar do tempo, com taxas de sobrevida de 94,4% em 5 anos (Piddock & Qualtrough,1990), sendo uma ótima possibilidade de reabilitação utilizada para fabricar coroas e próteses parciais fixas (PPF) por décadas. Sua aplicação clínica é muito importante e acontece até os dias de hoje, no entanto, com o desenvolvimento de materiais cerâmicos reforçados, as restaurações totalmente cerâmicas tem ganhado espaço. Em 2009, um estudo realizado nos Estados Unidos, concluiu que 46% de todas as coroas confeccionadas, já eram totalmente cerâmicas (Holland et al., 2006).

A cerâmica como material restaurador, é de interesse da odontologia há mais de duzentos anos, e este tipo de material vem sendo muito utilizado devido ao aumento da demanda por procedimentos restauradores estéticos, concomitante ao seu grande desenvolvimento (Bustamante-Hernández N et al., 2020). Dentre os materiais restauradores disponíveis, o potencial estético e a biocompatibilidade das cerâmicas odontológicas podem ser considerados

únicos. (Pagani et al., 2003). Nesse sentido, as cerâmicas apresentam boas propriedades estruturais, morfológicas e mecânicas, além do elevado potencial para biomimetizar as características dentais, tais como translucidez, fluorescência, estabilidade química, biocompatibilidade, alta resistência compressiva, coeficiente de expansão térmico linear similar ao da estrutura dental, condutibilidade térmica semelhante aos tecidos dentais e estabilidade de cor (Kreidler et al., 2008).

A busca se tornou intensa pelo desenvolvimento de cerâmicas que atendam às exigências estéticas almejadas sem apresentar limitações significativas em relação a sua durabilidade e resistência mecânica (Conrad et al., 2007). Muitas pesquisas sobre materiais odontológicos, tem sido dirigidas no sentido de produzir restaurações cerâmicas reforçadas que não necessitem de infraestruturas metálicas desde meados da década de sessenta, o que resultou numa tendência considerável em diminuir a utilização de restaurações metalocerâmicas (Barbosa GAS et al., 2016).

As cerâmicas vítreas feldspáticas, são consideradas um material ternário, uma vez que são compostas originalmente por três elementos principais: caulim/argila (alumínio-silicato hidratado), quartzo (sílica em sua forma cristalina) e feldspato natural (mistura de potássio com silicatos de alumínio e sódio). Esse composto é capaz de escoar em elevadas temperaturas, sendo o responsável pela coesão do material (Sakaguchi RL et., 2012). A fase vítrea, presente em 80% do material, é composta pelo feldspato (Al_2O_3+SiO_2) em combinação com óxidos de potássio (K_2O), sódio (Na_2O) ou cálcio (CaO). A fase cristalina, presente em 20%, confere resistência ao material, e é formada por quartzo (SiO_2), alumina (Al_2O_3) ou leucita ($K_2A1_2Si_6O_{16}$) A leucita é um silicato usado para formar feldspatos, dando origem a minerais feldspatóides, com fórmula química semelhante aos feldspatos. O caulim (Al_2O_3. $mSiO_2$. nH_2O) é um composto aglutinante, presente em apenas 4% do material, para unir os componentes da massa antes da sinterização (Van Noort R., 2010)

As cerâmicas feldspáticas eram as únicas empregadas na odontologia para confecção de restaurações indiretas até a década de 1960, tendo as mesmas proporcionado um excelente avanço estético. No entanto, sua baixa resistência mecânica limitava sua indicação inicialmente praticamente à

região anterior, com resistência à flexão variando entre 60 a 70 MPa (Gomes EA et al., 2008). Sendo assim, era necessário que esta cerâmica fosse utilizada como material de cobertura sobre uma infraestrutura metálica, e isto comprometia em a mimetização da estrutura dental, principalmente em espaços protéticos reduzidos, perfil gengival fino e por ser extremamente dependente da habilidade do técnico em prótese dental (Santos MJMC et al., 2015).

A partir do final da década de 1980 e início dos anos 1990, foram introduzidos blocos de cerâmica feldspática para a técnica de fresagem, utilizados na confecção de coroas anteriores, facetas e restaurações do tipo inlay e onlay (Bustamante-Hernández N et al., 2020). Em 1991, por exemplo, foi desenvolvido o sistema VITA TM Mark II (VITA Zahnfabrik, H. Rauter GmbH & Co., Alemanha), um bloco cerâmico monocromático para fresagem disponível em diferentes matizes (Guilardi LF et al 2020). As pastilhas para a técnica de processamento por prensagem também foram desenvolvidas nessa mesma época (Azar B et al., 2018). Esses materiais apresentam resistência a flexão pouco superior à porcelana feldspática aplicada por estratificação, em torno de 120 MPa para os blocos e 100 MPa para as pastilhas prensadas (Bustamante-Hernández N et al., 2020).

As cerâmicas reforçadas por cristais de leucita, surgiram no início dos anos 90 e possuíam conteúdos de leucita diferentes a depender da aplicação clínica e técnica de processamento: a cerâmica em pastilhas para a técnica de prensagem pelo calor, por exemplo, apresenta cerca de 35% a 55% de leucita na matriz vítrea (IPS Empress I, Ivoclar Vivadent). Já as cerâmicas para recobrimento, que são estratificadas, apresentam mais de 45% de leucita na composição (Optec HSP, Jeneric/Pentron Inc., Estados Unidos). A adição de leucita proporcionou um alto CET (coeficiente de expansão térmica), o que contribuiu para aumento da resistência da cerâmica à propagação de trincas. O baixo índice de refração dos cristais de leucita resulta em um material cerâmico translucente (Gracis S et al., 2015)

Em 1998, foi introduzida uma cerâmica reforçada por cristais de dissilicato de lítio (IPS Empress 2, Ivoclar Vivadent), a qual tem permitido a realização de próteses fixas envolvendo até 3 elementos com extensão até a região de 2º pré-molar. Este produto gerou uma patente sendo por muitos

anos produto exclusivo da empresa (Heintze, SD etal., 1998). Em 2001, foi introduzida comercialmente uma nova geração desse material (e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein), melhorando as suas propriedades mecânicas e ópticas (Kang SH et al., 2013; Lien W et al, 2015). As cerâmicas reforçadas por disilicato de lítio estão entre as mais utilizadas para confecção de coroas totalmente cerâmicas (Zarone F et al., 2016). Sua resistência à flexão (±380 MPa) (Kang SH et al., 2013), boa estética, resistência de união (±18,00 MPa) (Barato SS et al., 2015) e a possibilidade de ser fresada por meio da tecnologia CAD/CAM (Kassardjian V et al., 2016), como realizado para as cerâmicas feldspáticas e as reforçadas por leucita, simplificou seu processamento, se mostrando um material bastante versátil.

Vários fatores são importantes para sobrevida das restaurações totalmente cerâmicas, como a adaptação marginal, estética satisfatória, biocompatibilidade com os tecidos da cavidade oral e resistência às forças da mastigação (Gracis et al., 2015). Apesar dos avanços recentes, as cerâmicas são materiais friáveis, com baixa tolerância a tensões de tração, tensões cisalhantes e reduzida resistência ao impacto, sendo portanto, suscetíveis à formação e propagação de trincas (Raposo et al., 2015).

Diante disso, existem no mercado, além da cerâmica feldspática convencional, outras cerâmicas que possuem um reforço na sua fase cristalina, aumentando a resistência destes materiais (Sakaguchi RL et., 2012). Há uma diversidade de materiais para confecção dos diversos tipos de restaurações como: cerâmica reforçada com leucita, reforçada por dissilicato de lítio, alumina e/ou zircônia (Pagani et al., 2003; DellaBona & Kelly, 2008; Fasbinder et al., 2010), e mais recentemente os silicatos de lítio e as resinas nanocerâmicas (Hallmann L et al., 2019). Entre elas, considerando a praticidade de trabalho, a estética obtida, a resistência e a comprovada longevidade, as cerâmicas reforçadas com disilicato de lítio, tem sido indicada como boa opção de material quando da necessidade de resistência e estética, trabalhando sem infraestruturas.

A composição química básica das cerâmicas reforçadas por dissilicato de lítio (Li_2O-$2SiO_2$) é associada a diversos elementos gerando as diferentes apresentações comerciais, relacionadas a cor, translucidez e opacidade desses materiais (Zarone F et al., 2016). Sua microestrura consiste em uma

matriz vítrea circundada por uma fase cristalina que forma inicialmente um vidro homogêneo; após o tratamento térmico, ocorre uma expansão dos cristais, proporcionando uma melhora nas propriedades físicas e mecânicas da mesma (McLaren & Figueira, 2015). O reforço da cerâmica vítrea com os cristais de disilicato de litio, tem como maior vantagem, baixas taxas de fratura, pois sua resistência é aumentada em até cinco vezes quando comparada com a cerâmica feldspática convencional (Ritter et al., 2010).

Hoje, as cerâmicas reforçadas por disilicato de litio, consistem em um dos materiais mais importantes para as reabilitações odontológicas, devido a sua excelente estabilidade física e química. Além disso, sua cristalização ocorre de uma maneira homogênea, pois ela cristaliza-se com uma facilidade maior, quando comparada com os outros vidros alcalinos, servindo como modelo para estudos mais complexos (Braum, 2008). Dois trabalhos relatam altas taxas de sobrevivência de coroas cerâmicas reforçadas por dissilicato de lítio em região posterior: 96,3% em 4 anos de acompanhamento (Reicth & Schierz, 2013), 97,8% em 5 anos e 96,7% em 10 anos (Pieger et al., 2014).

O sistema IPS e.max, tem sido amplamente utilizado nos últimos anos, devido à possibilidade de se mimetizar a naturalidade da estrutura dentária (Gracis S et al., 2015). Este sistema cerâmico apresenta duas tecnologias disponíveis no mercado, prensagem térmica e fresagem por sistemas CAD/CAM. Constitui-se em um sistema versátil que vai das cerâmicas vítreas reforçadas por disilicato de lítio prensadas ou fresadas, respectivamente e.Max Press e e.Max CAD, até o óxido de zircônia prensado ou fresado, e.Max ZirPress e e.Max ZirCAD, respectivamente.

No entanto, recentemente, a patente que a empresa desenvolvedora possuía expirou e outras empresas passaram a fabricar e comercializar o novas versões deste material. Vários fabricantes estão produzindo cerâmicas dentais vítreas reforçadas por disilicato de lítio, tais como T-lítio (Talmax, Curitiba, Paraná, Brasil), Rosetta SM (Hass, Gangneung, Coréia), AIDITE (Shenzehen, Hong Kong, Pequim) e IRIS (Mainland, Tianjin, China). Os fabricantes propõem que estas novas cerâmicas tenham apresentem propriedades estruturais, morfológicas e mecânicas compatíveis com o primeiro sistema IPS e.max (Ivoclar Vivadent, Schaan, Liechtenstein) lançado previamente (Kang SH et al., 2013). Faz-se necessário assim, uma análise

das propriedades destes novos materiais.

OBJETIVOS

2. OBJETIVOS

Objetivo Geral

As cerâmicas vítreas reforçadas por dissilicato de lítio, consistem em um dos materiais mais importantes para as reabilitações odontológicas atualmente, devido a sua excelente estabilidade química e física. Após o fim da patente que a empresa desenvolvedora do material possuía, outras empresas passaram produzir novas versões destas cerâmicas, com compatibilidade para uso em sistemas CAD/CAM.

Objetivos específicos

Objetivo específico 1

Capítulo 1 - **Microstructural and mechanical analysis of two CAD-CAM lithium disilicate glass-reinforced ceramics**

O objetivo do presente estudo foi comparar duas marcas comerciais distintas de cerâmicas vítreas reforçadas por dissilicato de lítio para uso em sistema CAD/CAM (IPS e.max CAD e Rosetta SM), avaliando suas propriedades estruturais, morfológicas e mecânicas.

Objetivo específico 2

Capítulo 2 - **A new method for analyzing dental ceramic crystals size and content using SEM image processing software.**

O objetivo do presente estudo foi desenvolver e validar uma metodologia para

medição do tamanho médio dos cristais presentes em cerâmicas odontológicas e sua porcentagem utilizando imagens obtidas por meio da microscopia eletrônica de varredura (MEV).

Objetivo específico 3

Capítulo 3 – **Microstructural and mechanical analysis of four CAD-CAM lithium disilicate glass-reinforced ceramics**

Este estudo objetivou comparar 4 marcas comerciais de cerâmicas reforçadas por dissilicato de lítio para CAD/CAM, IPS E.max (Ivoclar Vivadent), Rosetta SM (Rosetta SM, Hass), T-lítio (Talmax) e IRIS (Mainland), avaliando suas propriedades estruturais, morfológicas e mecânicas.

Objetivo específico 4

Capítulo 4 – **Asthetic and functional rehabilitation in a patient with anterior skeletal open bite using lithium disilicte glass-reinforced ceramics: a case report.**

Nesta descrição de caso clínico foi feito o relato de situação limítrofe na qual foi realizada a a substituição de coroas metalo-cerâmicas insatisfatórias dos elementos 12, 11, 21 e 22, por coroas cerâmicas reforçadas por dissilicato de lítio em paciente com mordida aberta anterior esquelética.

CAPÍTULOS

3. CAPÍTULOS

3.1 CAPÍTULO 1

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Microstructural and mechanical analysis of two CAD-CAM lithium disilicate glass-reinforced ceramics

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Abstract

The aim of this study was to analyze the structural, morphological, and mechanical properties of two different lithium disilicate glass-reinforced ceramics for CAD-CAM systems (IPS e.max CAD and Rosetta SM). Five methodologies were used for both ceramics: microstructure (n=2) was analyzed using x-ray diffraction (XRD); morphological properties (n=2) were analyzed by scanning electron microscopy (SEM), with and without hydrofluoric etching; porosity (n=3) was assessed using 3D micro-computed tomography (micro-CT); flexural strength was measured (n=10) using the three-point bending test; and bond strength was verified using self-adhesive resin cement (n=10) under a microshear bond test. After performing all the tests, the data were analyzed using *t*-Student test and two-way ANOVA. All tests used a significance level of α=0.05. High peak positions corresponding to standard lithium metasilicate and lithium disilicate with similar intensities were observed for both ceramics in the XRD analysis. Morphological analysis showed no different patterns of crystalline structure after acid etching for either ceramic. Additionally, no significant differences were verified in the number or size of pores for the ceramics evaluated. Moreover, no differences in flexural strength were found for the ceramic materials tested, or in the bond strength to ceramic substrates for the resin cements. Based on the study results, no significant differences were found between the two CAD-CAM lithium disilicate glass-reinforced ceramics tested, since they presented similar crystalline structures with comparable intensities, and similar total porosity, flexural strength and bond strength.

Keywords: Ceramics. Lithium compounds. Physical Properties.

Introduction

The clinical use of ceramic restorations has grown substantially in the past years, owing to the increase in demand for aesthetic restorative procedures, and to the improvements in dental ceramic materials. Today, lithium disilicate glass-reinforced ceramic is one of the most frequently selected indirect restorative materials used to produce all-ceramic restorations.[1] The excellent resistance to fracture (±380MPa),[2] good esthetics, and satisfactory bond strength to resin cements, when adequate surface treatment is performed (±18.00MPa),[3] are factors favoring the growing acceptance of these ceramic materials. Additionally, the possibility of milling this ceramic using simplified fabrication methods, such as laboratory and chairside computer-aided design/computer-aided manufacturing (CAD-CAM) systems,[4-6] is another important advantage contributing to its excellent clinical approval.

Lithium disilicate glass-reinforced ceramics were introduced in 1998 as IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein), an exclusive product released by Ivoclar. The e.max Press system (Ivoclar Vivadent, Schaan, Liechtenstein) emerged in 2006 as the new generation of heat-pressed ceramics, featuring improved mechanical and optical properties over the first-generation material.[7] However, the patent of this product recently expired, and other companies can now fabricate and market similar ceramic materials. Several manufacturers are now producing other lithium disilicate-based ceramics, including Rosetta (Rosetta, Hass, Gangneung, Korea), T-lithium (Talmax, Curitiba, PR, Brazil), AIDITE (Shenzhen, Guangdong, China) and IRIS (Tianjin, Mainland, China). The manufacturers suggest that these new ceramics have mechanical, structural and morphological properties similar to the primary IPS e.max system (Ivoclar Vivadent, Schaan, Liechtenstein), but few studies comparing these materials are available.[2]

The crystalline structure of ceramics influences the mechanical and morphological properties of these materials.[8] Structural properties are commonly investigated using x-ray diffraction analysis (XRD), which identifies the peaks of the crystals present in the ceramic and its crystalline phase, and determines the degree of crystallinity and the size of the crystals formed.[8-11] The morphological characteristics of dental ceramics can also be evaluated by scanning electron microscopy (SEM),[12] since high resolution emission field protocols enable the shape and size of the crystal grains to be observed. The percentage of pores and the ceramic characterization can also be determined by micro-computed tomography (μCT), which allows analysis without destroying the specimens.[13] Moreover, the flexural strength test is important to evaluate the maximum force to fracture and the flexural modulus of dental ceramics, a property that can help characterize the load capacity of the material.[15] Additionally, the interaction of glass ceramics with resin cements and their bonding capability to these cements are important clinical parameters, modulated by the composition and susceptibility of these ceramics to surface treatment with hydrofluoric acid etching associated to silane coupling agents. The microshear bond strength test has been commonly used to measure the bond strength of resin cements to ceramic materials, because it is an easy operation to perform.[14]

Thus, the aim of this study was to compare two CAD-CAM lithium disilicate glass-reinforced ceramics using different methodologies, specifically, XRD, SEM, porosity test (μ-CT), microshear bond strength and three-point bending test. The null hypothesis tested was that no differences would be detected in the microstructural and mechanical properties of either glass ceramic material evaluated.

Methodology

Two CAD-CAM lithium disilicate glass-reinforced ceramics, IPS e.max CAD (Ivoclar-Vivadent, Schaan, Liechtenstein) and Rosetta SM (Hass, Gangneung, Korea), were evaluated using HT-A2 C14 blocks. The specimens for each ceramic material were prepared according to the respective methodologies, as follows:

X-ray diffraction (XRD)

X-ray diffractogram patterns were performed (n=2) at room temperature (25 °C) using a diffractometer (XRD-6000, Shimadzu Corp., Tokyo, Japan) with monochromatic Cu-K_1 (λ=1.54056Å) radiation. XRD scanning was carried out using the Cu-K1 emission (λ = 1.54056Å), generating a current of 15mA, 30kV, and a wavelength equal to 1.5406Å, with a continuous scanning interval of 2θ (20-80), with a step of 0.02s. The XRD patterns were compared with the JCPDS (Joint Committee on Powder Diffraction Standard) to identify the type of crystal and crystalline phase of both ceramic materials.[2-8] Two specimens were selected from each group (pre-crystallized and crystallized) to perform the structural analysis. The specimens crystallized using a special furnace (Programat P300, Ivoclar Vivadent, Schaan, Liechtenstein), together with the P91 program. This furnace reaches the maximum temperature of 845°C, then stabilizes for a period of 7 minutes, after which it starts to cool slowly to prevent thermal shock.

Traditionally, the structural analysis test requires that the specimen be reduced to powder, but the ceramic block was too rigid. Alternatively, the specimens were cut to a size of approximately 1 cm^3 using a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Then, they were placed on a metal device filled with aluminum particles to start the test, which lasts an average of 1 hour and 40 minutes, for each specimen. Two graphs (diffractograms) were obtained for each group and interpreted qualitatively.

Scanning Electron Microscopy (SEM)

The morphological structures of both ceramics were analyzed after crystallization. Initial preparation consisted of obtaining specimens (n=2) with dimensions of approximately 3.0 mm^3 using a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Next, they were sectioned from the ceramic blocks at the pre-crystallized stage, and submitted to the crystallization process as described above. All the ceramic surfaces were polished sequentially using silicon carbide paper (#600, 800, 1200, Norton, Guarulhos, SP, Brazil) for 20 s, and then etched with 10% hydrofluoric acid (HF) (Condac Porcelana, FGM, Joinville, SC, Brazil) for 20 s. The specimens were washed using 70% alcohol and distilled water, followed by an ultrasonic bath with distilled water for 10 min, to remove the HF etching residues. Afterwards, the specimens were mounted on coded brass stubs and sputter-coated with gold palladium for 60 s at 45 mA (QR 150ES, Quorum Technologies, Laughton, Lewes, UK) to obtain images of better quality that allowed visualization of the crystals. Three images of each ceramic material with and without HF etching were obtained using SEM (VEGA 3, TESCAN, LMU, Kohoutovice, Czech Republic), with an accelerating voltage of 20.0 kV and original magnification of 20,000×. The images were obtained and analyzed by a skilled operator.

Total porosity

Three specimens were obtained from each ceramic material, with a cubic format (±4.0mm^3) for pore evaluation, and 3D reconstruction using μCT scanner (Skyscan 1272, Bruker, Kontich, Belgium). The specimens were also sectioned from the ceramic blocks at

the pre-crystallized stage, and then submitted to crystallization as described earlier. The following scanning parameters were used: filter Al 0.5 & Cu 0.038; source voltage 90 kV; source current 111A; image pixel size 10.0m; 81 slices; lower gray threshold 60; upper gray threshold 190.[13] The total porosity percentage was measured and 3D images contrasting the dense mass of the ceramic and the void spaces of the pores were collected. The reconstructed 3D data sets were quantified using a CTAn automated image analysis system (Brunker, Kontich, Belgium). The block images were obtained virtually, without destroying the specimens. The values were analyzed using the *t*-Student test (α=.05).

Three-point bending test

The three-point bending test was performed as recommended by ISO standard 6872.[14,15] Both ceramic materials were sectioned into rectangular bars using a diamond saw mounted on a low speed precision cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). The bar dimensions were approximately 20.0mm long, 4.0mm wide and 1.2mm thick, and all the edges were chamfered, leaving a 0.1-mm-wide chamfer, following ISO 6872:2008. The dimensions were checked with a Digimatic caliper (Mitutoyo Absolute Digimatic Caliper, Tokyo, Japan) and recorded. The bars from each group (n=10) were crystallized in a special furnace (Programat P310, Ivoclar Vivadent, Schaan, Liechtenstein). Afterwards, the bar surfaces were polished with a polishing machine (EXAKT 400 CS, EXAKT Technologies, Oklahoma City, OK, USA), using silicon carbide papers (#600, 800, 1000, and 1200-grit; EXAKT Technologies, Oklahoma City, OK, USA) under running water at 300 rpm. Lastly, the specimens were washed and stored dry until testing.

In the testing process, the specimens were placed in a mechanical testing machine (DL2000, EMIC, São José dos Pinhais, PR, Brazil) over two rods approximately 2.0 mm in

diameter with a 16.0-mm span length. The three-point bending test consisted of applying a compressive force with a 50.0 kgf load cell over the center of the ceramic bar, using a piston approximately 2.0 mm in diameter at a 0.5 mm/min crosshead speed all the way to fracture. The values were analyzed using the *t*-Student test (α=.05). The three-point bending test consisted of calculating flexural strength ($\sigma_{3\text{-pt}}$), where σ is the distance between the supports (16.0 mm), and w and b are the width and thickness of the specimen, respectively, measured immediately prior to testing. The following formula was used:

$$\sigma_f = \frac{3Pl}{2wb^2}$$

where P is the fracture load (N), l is the span size (16 mm), w is the specimen width (mm) and b is the thickness of the specimen (mm).

Bond strength test

Ten CAD-CAM blocks from each ceramic material were selected for the microshear bond strength test. One 3.0 mm slice was removed from each block using a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), and crystallized using the same protocol described earlier. The ceramic slices were embedded in polystyrene resin (Aerojet, Santo Amaro, SP, Brazil). Next, all the ceramic surfaces were polished sequentially using silicon carbide paper of (#600, 800, 1200; Norton, Guarulhos, SP, Brazil) for 20 s.

Afterwards, the test surface of each ceramic slice was etched with hydrofluoric acid 10% (Condac Porcelana, FGM, Joinville, SC, Brazil) for 20 s, followed by rinsing with water for 30 s and air-drying for another 30 s. One layer of a silane-coupling agent (Prosil, FGM,

Joinville, SC, Brazil) was actively applied to the ceramic surface for 20 s, and left to react for 60 s. A self-adhesive resin cement (RelyX U200, 3M ESPE, St. Paul, MN, USA) was prepared according to the manufacturer's directions, and inserted into silicon molds of Tygon® bore tubbing (1.0 mm in diameter and height) on the ceramic surfaces.[16] After 5 min of preparation, the resin cement was activated for 40 s using a monowave LED light-curing unit (Radii Cal, SDI, Victoria, Australia). Next, the silicone mold was removed using scalpel blades. Six cylinders were made on the surface of each ceramic block, spaced at 3.0 mm intervals.[16,17] The specimens were stored at 100% relative humidity, at 37°C for 24 h previous to testing.

The microshear bond test (μSBS) was performed after positioning and fixing the ceramic slices in a mechanical testing machine (OM100, Odeme Dental Research, São José dos Pinhais, PR, Brazil). The resin cement cylinders were then aligned in the direction of force application. A 0.2 mm diameter orthodontic wire (NiCr, Morelli, Sorocaba, SP, Brazil) was used to load the cement cylinders perpendicular to the ceramic surface. The crosshead speed was set at 0.7 mm/min.[18] The procedure for the specimens tested immediately (24h - T0) consisted of loading three cylinders, and then obtaining the mean values. The remaining resin cylinders from each group were then submitted to aging by storing the ceramic specimens in relative humidity at 37°C for 30 days. After storage, the three remaining resin cylinders were loaded (30 days - T1) using the same protocol described above. Bonding strength values from T0 and T1 were compared within the groups and between both ceramics. Statistical analyses were performed using two-way ANOVA followed by the Tukey test. The bond strength of each specimen (MPa) was calculated using the following equation:

$$R = F / A$$

where R is the bond strength in MPa; F is the force required for specimen rupture (N); and A is the adhesive area of the specimens (mm^2).

The specimens were submitted to a microshear bond strength test, and the fractured surface of the specimens was evaluated under optical microscopy (Axiocam, Mitutoyo, Absolute, Tokyo, Japan) at 40× magnification to determine the failure mode, classified into: 1) adhesive failure; 2) cohesive failure; and 3) mixed failure.

Results

X-ray diffraction (XRD)

The XRD results are shown in Figure 1. Both ceramics presented comparable, narrow diffraction peaks, which confirm the formation of crystalline compounds of similar intensity. The diffraction peaks detected in the XRD patterns are characteristic of lithium metasilicate (ICCD 029-0829) and lithium disilicate (ICCD 040-0376) crystals, confirming the formation of these crystalline compounds for both ceramic materials tested.

Scanning Electron Microscopy (SEM)

After crystallization, the crystalline microstructure of the crystals became denser, and the surface etching caused changes in their superficial morphology. SEM images of the etched ceramic surfaces of both systems are shown in Figure 2. The 10% hydrofluoric acid etching caused IPS e.max CAD and Rosetta SM ceramics to form elongated spindle-shaped lithium disilicate crystals surrounded by a sparse glass matrix. The shape and size of these

crystals were very similar for both ceramics systems analyzed.

Porosity– (µCT)

The total porosity values found for both ceramics using micro-computed tomography scanning ranged from 0.05 to 0.11% for IPS e.max CAD and from 0.07 to 0.13% for Rosetta SM (Table 1). The *t*-Student test revealed no significant differences between the two ceramic systems for total porosity (P = 0.473). Uniform pore distribution was observed for both ceramics under µCT evaluation (Figure 3).

Three-point bending test.

Mean flexural strength (MPa) values and standard deviation for both ceramic systems are presented in Table 2. The flexural strength was found to be between 340 and 350 MPa for the two ceramic systems, and the *t*-Student test showed no significant differences for the materials tested (P = 0.652).

Microshear bond strength test and failure mode

Mean microshear bond strength (MPa) values and standard deviation of a self-adhesive resin cement for the ceramic's substrates tested both immediately and after 30 days are shown in Table 3. Bond strength values of the resin cement were almost similar for both ceramic systems, regardless of the different storage times (±17.0MPa). Two-way ANOVA revealed no significant differences between the ceramic materials for bond strength (P = 0.881), at storage time (P = 0.712).

The failure mode observed for the specimens was predominantly mixed failures (Table

4), followed by adhesive failures, irrespective of the ceramic material or storage time (Figure 4).

Discussion

The null hypothesis tested was accepted, since both CAD-CAM lithium disilicate glass-reinforced ceramics tested presented similar crystalline structures and morphological and mechanical properties. Lithium disilicate ceramics are commonly used for dental purposes, owing to their favorable properties, such as good fracture strength and satisfactory aesthetics.[4] The company that first developed and patented this material held the exclusive right to its production, limiting the option for the lithium disilicate-based ceramics available in the market. After the patent expired, other companies started to produce glass dental ceramics reinforced with lithium disilicate as well. However, little knowledge is available on whether these recently introduced lithium disilicate-based ceramics have the same quality as the precursor system first introduced.

Another important advantage reinforcing the overwhelming clinical approval of lithium disilicate ceramics is that they can be milled by simplified fabrication methods, such as laboratory and chairside CAD-CAM systems.[4-6] The lithium disilicate ceramics tested have been reported to have more lithium metasilicate when treated at temperatures below 780°C. Conversely, when temperatures above 780°C are used, three strong peaks (23.9, 24.6, and 30.1) of lithium disilicate have shown a greater amount of crystallization in previous studies.[19] An investigation using XRD revealed that the transformation of lithium metasilicate into lithium disilicate was dependent on the heating temperature, irrespective of the overall heating time.[8] In the present study, XRD results confirmed that the main crystalline components of Rosetta SM were similar to those of IPS e.max CAD, at the pre-crystallized and crystallized stages (Figure 1). This finding indicates that the ceramic materials evaluated

presented close conversion of lithium metasilicates into lithium disilicate crystals when submitted to the heat treatment program used in this investigation, which is the same as that suggested by the processing instructions of both manufacturers.

The morphological analysis provided by SEM enabled evaluation of the surface topography of the dental ceramics tested. After the heat treatment (crystallized stage), the crystalline microstructures became denser in both ceramic systems tested, further indicating a close conformation of the lithium disilicate crystals (Figure 2). A previous study has shown that these microstructures can become even denser and more homogeneous when restorations are subject to masticatory forces.[2] Lithium disilicate crystals have an average length of 0.5 μm in their pre-crystallized phase, and the average crystal size increases up to 3.0 μm after crystallization, as observed in the present study (Figure 2).[8,12,20]

The μ-CT scanning showed that both ceramics tested presented similar total porosity ($P = 0.473$). Pores were observed in the specimens of both ceramics (Figures 3A and 3B), and this porosity may interfere in the mechanical properties of these materials. The crystals present in dental ceramics have isotropic characteristics that play a significant role in modifying their properties, such as material hardness, flexural strength, modulus of elasticity and fracture toughness.[3,21] However, the presence of the pores may interfere with stress distribution, since they act as stress concentrating areas, and influence the mechanical proprieties by favoring mechanical failures. Therefore, it was clear whether the porosity found similarly in both ceramic materials had to be reduced in order to optimize the microstructure of the different lithium disilicate ceramics evaluated.

High flexural strength is commonly observed for lithium disilicate glass-reinforced ceramics, compared to conventional feldspathic porcelain or leucite glass-reinforced ceramics.[8] The definitive strength of ceramic materials seems undetermined, because of multiple factors influencing measurements, such as polishing procedures, stress rates,

testing method and environmental conditions, as well as specimen dimensions.[22-24] The results of the present study showed similar mean flexural strength values (±346.92MPa) for both ceramics evaluated (P = 0.652), corroborating previous investigations that tested the same materials.[2,8]

The bond strength of resin cements to lithium disilicate ceramics is an important factor for the longevity of these dental restorations.[17] The stress transferred through the ceramic restoration to the remnant tooth hinges on an adequate bonding interface, which may prevent failures.[18] According to the present study, no differences were found between the values for the bond strength of resin cement to both lithium disilicate ceramics (P = 0.881). The mean values for the bond strength of the resin cement to the ceramic substrates found in this study corroborate those of previous reports,[3] and are almost similar to each other (±17.0MPa). The specimens were stored in distilled water, which may cause bond degradation; however, the oral environment is even more challenging for ceramic/resin cement bonding interfaces.[25] Even after 30 days of water storage, no differences were verified for the bond strength values of the two ceramics (P = 0.712).

Additionally, no cohesive failures were found in this study, demonstrating that the μSBS test was probably performed correctly, despite the limitations involving laboratory adhesion tests to dental ceramics. The most prevalent failure modes detected were adhesive and mixed failures for both ceramic materials, irrespective of the storage time. The IPS e.max CAD system showed more adhesive failures in the immediate testing period (T0), where both ceramic systems showed similar failure distribution after 30 days of storage (T1). Data obtained from laboratory bond strength assessments should be analyzed with caution, and the direct relationship to clinical outcomes is limited. Thus, this study has focused on discussing only the bond strength and failure modes between the resin cements tested, as well as different ceramic materials under distinct storage conditions, without extrapolating

the results to clinical situations.

As seen, lithium disilicate-reinforced ceramics are one of the most important and versatile materials for dental rehabilitations, because of their excellent thermal and physical stability, as well as good resistance and aesthetic properties. Despite the limitations of an *in vitro* study, the results showed that both ceramics showed similar characteristics in the parameters tested, thus increasing the alternatives made available to laboratory technicians and dental practitioners.

Conclusion

Based on the results of the present study, both CAD-CAM lithium disilicate glass-reinforced ceramics tested showed similar characteristics, considering that they presented comparable crystalline structures with close intensities, and similar total porosity, flexural strength and bond strength.

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Figure legends

Figure 1A –XDR patterns of IPS e.max CAD group

Figure 1B –XDR patterns of Rosetta SM group

Figure 2A – SEM images: IPS e.max CAD group after etching. 10,000× magnification.

Figure 2B – SEM images: Rosetta SM group after etching. 10,000× magnification.

Figure 3A – 3D reconstruction of IPS e.max CAD group using micro-CT scan

Figure 3B – 3D reconstruction of Rosetta SM group using micro-CT scan

Figure 4A – Optical microscopy images: Mixed failure mode after microshear bond strength testing. 40× magnification.

Figure 4B – Optical microscopy images: Adhesive failure mode after microshear bond strength testing. 40× magnification.

Table legends

Table 1 – Total porosity percentage (SD) for the CAD-CAM lithium disilicate glass ceramics systems

Table 2 – Mean flexural strength (MPa) values (SD) for the CAD-CAM lithium disilicate glass ceramics systems (MPa)

Table 3 – Mean microshear bond strength (MPa) values (SD) of a self-adhesive resin cement to CAD-CAM lithium disilicate glass ceramics systems immediately (T0) and after 30 days (T1).

Table 4 - Failure mode percentage (%) following microshear bond strength test for the CAD-CAM lithium disilicate glass ceramics systems immediately (T0) and after 30 days (T1).

Table 1 – Total porosity percentage (SD) for CAD-CAM lithium disilicate glass ceramics systems.

Ceramic system	Total porosity
IPS e.max CAD	0.08 (0.030) A
Rosetta SM	0.10 (0.034) A

* Same uppercase letters indicate no significant difference between groups; *t*-Student test (P<.05).

Table 2 – Mean flexural strength (MPa) values (SD) for CAD-CAM lithium disilicate glass ceramics systems (MPa)

Ceramic system	Flexural strength
IPS e.max CAD	341.45 (61.44) A
Rosetta SM	352.39 (36.77) A

* Same uppercase letters indicate no significant difference between groups; *t*-Student test (P<.05).

Table 3 – Mean microshear bond strength (MPa) values (SD) of a self-adhesive resin cement to CAD-CAM lithium disilicate glass ceramics systems immediately (T0) and after 30 days (T1).

Ceramic system	T0	T1
IPS e.max CAD	17.89 (6.3) Aa	17.11 (5.9) Aa
Rosetta SM	17.27 (3.0) Aa	17.60 (4.7) Aa

Same uppercase letters indicate no significant difference between groups in rows; same lowercase letters indicate no significant difference between groups in columns; Two-way ANOVA and Tukey HSD test (P<.05).

Table 4 - Failure mode percentage (%) following microshear bond strength test for CAD-CAM lithium disilicate glass ceramics systems immediately (T0) and after 30 days (T1).

Group	Adhesive failures	Cohesive failures	Mixed failures
Rosetta T0	60%	0%	40%
Rosetta T1	70%	0%	30%
E.max T0	100%	0%	0%
E.max T1	80%	0%	20%

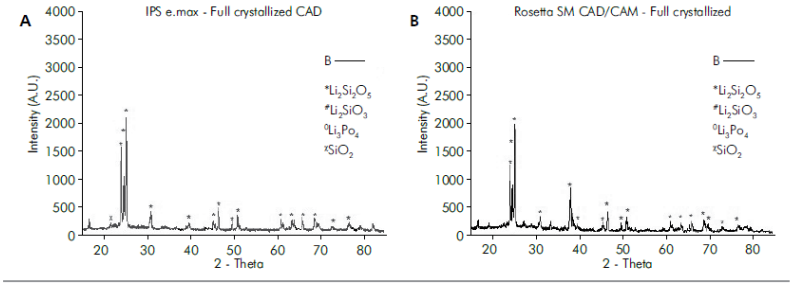

Figure 1. XDR patterns: A: IPS e.max CAD group; B: Rosetta SM group.

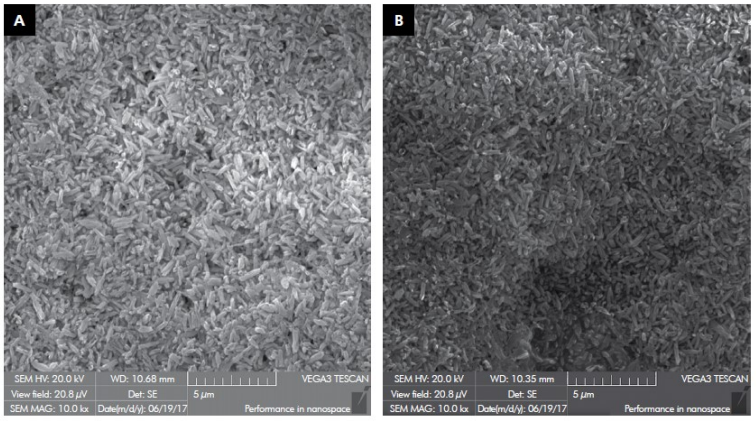

Figure 2. SEM images: A: IPS e.max CAD group after etching, 10,000× magnification; B: Rosetta SM group after etching, 10,000× magnification.

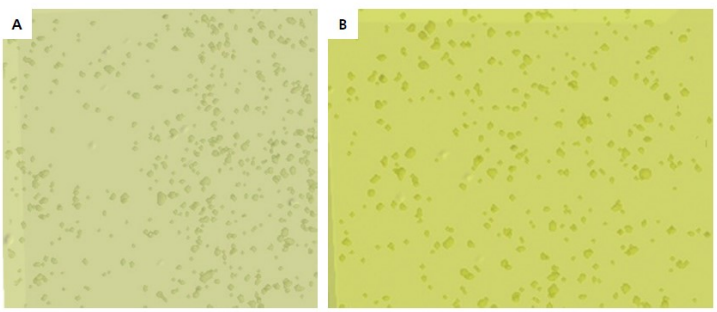

Figure 3. 3D reconstruction: A: IPS e.max CAD group using micro-CT scan; B: 3D reconstruction of Rosetta SM group using micro-CT scan

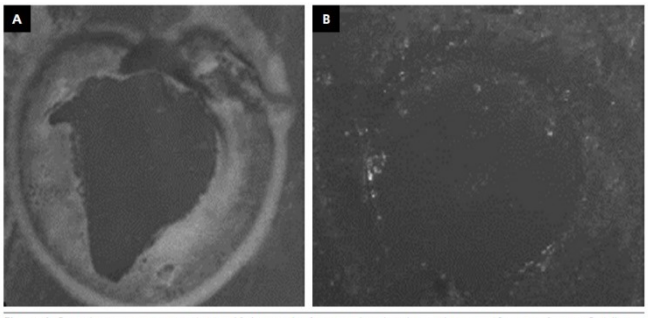

Figure 4. Optical microscopy images: A: Mixed failure mode after microshear bond strength testing. 40× magnification; B: Adhesive failure mode after microshear bond strength testing. 40× magnification.

CAPÍTULOS

3.2 CAPÍTULO 2

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A new method for analyzing dental ceramic crystals size and content using SEM image processing software

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ABSTRACT

The aim of this study is to present and validate a new method for evaluating the content of crystals from dental ceramics (average size and percentage) in images obtained by scanning electron microscopy (SEM) using the Image J processing software. The average size and the percentage of crystals present in 4 different lithium disilicate glass-reinforced ceramics were evaluated: T-Lithium (Talmax), Rosetta SM (Hass), IRIS CAD (Mainland) and IPS e.max (Ivoclar Vivadent). The specimens were prepared from pre-crystallized CAD/CAM blocks (3.0 mm^3) and were fully crystallized and treated with 10% hydrofluoric acid previously to performing SEM analysis. Using an image processing software (Image J) the average size and percentage of the crystals were assessed on the ceramic surface. The obtained data were tabulated and analyzed using 1-way ANOVA (α=0.05) comparing the results among the experimental groups. Significant differences were found for the IRIS CAD ceramic when comparing the crystal size to the other experimental groups ($p \leq 0.001$). According to the average size of crystals and total percentage of crystals per area, IRIS CAD ceramic exhibited a more deficient crystalline matrix when compared to the other ceramic materials tested. The proposed method was shown to allow easy and reliable evaluation of the crystal content in dental ceramics using SEM images associated to an open-access, image processing software.

Keywords: Ceramics; Dental materials; Lithium compounds; Physical Properties.

1. Introduction

The clinical use of dental ceramic restorations has grown considerably in the past years due to the increased demand for aesthetics and to the continuous improvements of ceramic materials (Poggio et al., 2017). The acceptable resistance to fracture, good aesthetics, and satisfactory bond strength to resin cements when performing adequate surface treatment of glass ceramics, are favorable factors towards the growing acceptance for these restorative materials (Tavares et al., 2020). Additionally to the conventional processing methods, the possibility of milling ceramics with simplified fabrication techniques, such as laboratory and chairside computer-aided design/computer-aided manufacturing (CAD/CAM) systems, contributed to a great clinical approval for glass dental ceramics (Raposo et al., 2020; Papadiochou and Pissiotis, 2018; Li et al., 2014; Goujat et al., 2018).

Lithium disilicate glass-reinforced ceramics are nowadays, one of the most used materials to produce all-ceramic indirect restorations worldwide due its versatility (Poggio et al., 2017; Spitznagel et al., 2018). This class of dental ceramics present typical lithium metasilicate crystals embedded in a glass matrix before the heat treatment, with typical acicular shaped grains (Lien et al., 2015). After the heat treatment (crystallized stage), the crystalline microstructure of this ceramic changes into a denser form (Kang et al., 2013). Lithium disilicate crystals have an average length of 0.5 μm, in their pre-crystallized phase and after crystallization, the average crystal size increases up to 3.0 μm depending on the ceramic composition and manufacturer (Ramos et al., 2016; Silva et al, 2017).

The composition and structure of dental ceramics are related to the physicomechanical properties of these materials, and characteristics such as resistance and aesthetics may be influenced by the glass matrix and crystalline content ratio (Ramos et al., 2016). Scanning electron microscopy (SEM) is a powerful tool that may allow evaluation of the size, shape and quantity of crystals present in the ceramic structure, enabling investigation of the morphological characteristics from these materials (Lien et al., 2015). However, analysis of ceramics can be challenging due to the complex structure of some materials. The use of an image processing software can be helpful to improve the analysis of images obtained by SEM, making possible to evaluate the characteristics of dental ceramics more accurately.

ImageJ (NIH, Bethesda, Maryland, USA) is an open-access, public domain Java-based software used for image processing. This software allows to display, edit, analyze, process, in addition to the ease of saving and printing 8-bit, 16-bit and 32-bit images. It is also possible to read several image formats, as TIFF, GIF, JPEG, BMP, DICOM, FITS and “raw” files. The software supports “stacks” with a series of images that share a single window. As the software is made up of several segments, operations classified as time consuming as reading an image file, can be executed in parallel with other operations (Gonzalez and Woods, 2009; Schroeder et al., 2021; Schneider et al., 2012). It is a very versatile software that can calculate area and pixel value statistics of user-defined selections and intensity-thresholded objects, besides measuring distances and angles.

Thus, the aim of this study is to present and validate a new method for evaluating the content of crystals from dental ceramics in images obtained by SEM using the Image J software for analyzing the average size and the percentage of crystals present in 4 different lithium disilicate glass-reinforced ceramics. The null hypothesis tested was that

no differences would be detected in the average size and percentage of crystals in the different dental ceramics evaluated using this new method.

2. Methodology

Four distinct commercial CAD-CAM lithium disilicate glass-reinforced ceramics blocks (HT-A2 C14) were selected for this study as described in Table 1. The specimens from each ceramic material were prepared according to the following methodologies:

Table 1. Experimental groups followed by their names and manufacturers.

Group	$LiSi_2$ Ceramic	Manufacturer
G1	IPS e.max CAD	Ivoclar Vivadent, Schaan, Liechtenstein
G2	Rosetta SM	Rosetta SM, Hass, Gangneung, Korea
G3	T-Lithium CAD	Talmax, Curitiba, PR, Brazil
G4	IRIS CAD	Mainland, Tianjin, China

2.1. Specimen preparation

The preparation of the specimens consisted in obtaining ceramic blocks with approximately 3.0 mm^3 dimension from the commercially available pre-crystallized CAD/CAM blocks (C14) using a water-cooled low-speed diamond saw (Isomet, Buehler GmbH, Düsseldorf, Germany). The specimens obtained (n=3) were then fully crystallized using a proper furnace (Programat P300, Ivoclar-Vivadent, Schaan, Liechtenstein) following the same firing program (P91). This furnace reaches the maximum temperature of 845°C, then stabilizes for a period of 7 minutes, after which it starts to cool slowly to prevent thermal shock. After crystallization, the ceramic surfaces were sequentially polished using silicon carbide papers (#600, 800, 1200, Norton, Guarulhos, SP, Brazil) for 20 s, and then etched with 10% hydrofluoric acid (HF) (Condac Porcelana, FGM, Joinville, SC, Brazil) for 20 s. Then, the specimens were washed using 70% alcohol and distilled water, followed by an ultrasonic bath with distilled water for 10 min, to remove the HF etching residues. Afterwards, the specimens were mounted on coded brass stubs and sputter-coated with gold palladium for 60 s at 45 mA (QR 150ES, Quorum Technologies, Laughton, Lewes, UK) to obtain better quality SEM images allowing visualization of the

crystals. Three images were taken from each ceramic material using SEM (VEGA 3, TESCAN, LMU, Kohoutovice, Czech Republic), with an accelerating voltage of 20.0 kV and original magnification of 20,000×. The images were previously analyzed and then obtained by a skilled operator.

2.2 Crystal area setting

For the analysis and treatment of the SEM images obtained from the ceramics, the Image J processing software was used. The SEM image was imported using the shortcut File followed by Open into the software, where the measurement scale must be calibrated for showing compatible results with the real scale from the SEM image. Data scale presented in SEM images was 5 μm, and therefore, this value is required to represent the calibration basis in the software. The Straight tool is selected, and a straight line is drawn under this scale (Fig. 1). To increase the accuracy of results, the operator should press the Shift key to assist in creating a straight line without any angulation. The scale is defined in menu, in Analyze followed by Set Scale. Through the dialog box, the first information presented in the Distance in pixels field, refers to the value of the straight line previously created. The Known distance field should be changed to 5 according to the figure scale (5 μm) and the Pixel aspect ratio is set to 1.0 to define 1:1 height/width proportion. Regarding to Unit of length, the operator must change it to “μm”, which corresponds to the real analytical value of the SEM image. Then, the Global checkbox must also be checked in order to use these measurement settings for all images to be posteriorly analyzed.

Furthermore, for obtaining reliable results, the caption must be removed by image cutting. For this, the Rectangle tool is chosen in the toolbar below the menu, and a perfect square area is designed to determine the total area of crystals, excluding the caption area from the selection. Additionally, the operator should use Shift key to create a perfect square during the process as shown in Figure 2. Then, the option Image followed by Crop must be selected in the menu. Afterwards, it is necessary to process the image into 8-bit conformation, corresponding to 256 different shades of gray, since this change will provide a correct binarization process for the SEM images in future steps. For this purpose, the operator must select Image option on menu followed by Type, and then 8-bit.

At these stages, it is recommended to duplicate the image file at each processing step, because the software does not have tools to allow reversibility of the previous stages performed. In case of error, it is mandatory to repeat the entire analysis process. This can be done in the menu shortcut Image followed by Duplicate. After duplication, the operator can use the second image as an option to proceed the analysis. For assuring better definition of the disilicate crystals, Contrast tool is used in order to reduce unsuitable image noise, since this will allow better differentiation of the glass matrix and crystalline phase. The option Process is chosen on menu, followed by Enhance Contrast. In the Saturated pixels board, 0.3% was used as the standard value for all groups tested in this study (Fig. 3). This value was considered as the best contrast option after several pilot analyses conducted by the authors.

Subsequently, it is necessary to binarize the SEM image in order to emphasize the crystals present in the field of analysis. For this, the menu tab Image is selected, followed by Adjust and Threshold. In this stage, the operator must be trained properly for establishing the threshold only in the areas corresponding to the crystalline phase (Fig.4-A). Thereafter, the dialog box must be closed after selecting Apply. It will be noticed that the image will be now two-toned, as a result of the binarization process. The black range on the SEM images refers to the crystals and will be analyzed for determination of Area, Standard Deviation of the gray values used to generate the Mean gray value and Area fraction. These patterns must be verified in Set Measurements, by clicking on Analyze. The Area tool will determine the selection area of the rectangular range in square pixels; the Mean Gray Value is responsible for the average gray value within the rectangular selection. This average value is obtained by the sum of all gray pixel values divided by the total number of pixels. The Area Fraction provide the percentage of pixels in the image or selection that have been highlighted in red color using Threshold tool (Fig. 4-B). After selecting the checkboxes in Set Measurements, data were obtained for each group by using the Analyze option in the menu, followed by Measure (Fig 4-C).

2.3 *Average amount of crystals*

To determine the average amount of crystals in the image, 3 experienced operators were previously trained by an external member for using the free-hand Straight tool from the ImageJ software in order to measure 20 units of the lithium disilicate crystals present

on the electron scanning microscopy images from each ceramic material according to the experimental groups. The measurements were taken individually by each operator, and the crystals were selected by simple randomization along the area of the image with the Rectangle tool. A line was individually traced along each selected crystal with the Straight tool, and the individual measurements showed in the software layout (Fig. 5A), were registered in micrometers (μm) and tabulated for further analysis. Only in cases where any doubts were presented during the measuring process, the external member intervened for convenience. The measurements were performed on ten different SEM images for each group by the 3 operators, totalizing 600 crystal readings per experimental group.

In order to simplify the analysis in the SEM images, it was necessary to use a tool that allowed to mark the crystals that were once measured, to avoid measuring the crystals more than a time (Fig. 5B). For this, the Paintbrush Tool was selected for highlighting the crystals after the measurements.

2.4. Statistical analysis

Data from crystal area setting and average amount of crystals were collected, tabulated, tested for homoscedasticity and submitted to 1-way analysis of variance (ANOVA) and Tukey HSD test, at $\alpha=0.05$ confidence level (SigmaPlot Systat Software Inc). Mean results and standard deviation were established for each experimental group.

3. Results

Mean lithium disilicate crystal size and standard deviation for the experimental groups are reported in Table 2. Significant differences were found for the IRIS CAD group when comparing the size of crystals to the other experimental groups ($p \leq 0.001$). T-Lithium CAD group presented rod-like crystals, although the values for this group were statistically similar to the IPS e.max CAD group. Despite some minor differences, groups 1 to 3 presented very similar mean crystal size and standard deviation.

Table 2. Mean lithium disilicate crystal size (μm) and standard deviation (SD) for the experimental groups.

$LiSi_2$ Ceramic	**Crystal size (μm)**
IPS e.max CAD	1.08 (0.07) A
Rosetta SM CAD	1.19 (0.06) A
T-Lithium CAD	1.12 (0.08) A
IRIS CAD	2.24 (0.11) B

*Different uppercase letters indicate significant difference between groups; Tukey HSD test ($p<0.05$).

The analysis of the larger area with lithium disilicate crystals content in the glass matrix was obtained through SEM images. According to the average size of crystals and total percentage of crystals per area, the IRIS CAD group exhibited a more deficient crystalline matrix when compared to the other experimental groups. The Rosetta SM CAD and T-Lithium CAD groups also showed reduced results per area when compared to the IPS e.max CAD group, which showed the higher crystal percentage per area. The quantitative results are reported in Table 3.

Table 3. Total crystal percentage per area (%) and standard deviation (SD).

$LiSi_2$ Ceramic	**Crystal (%)**
IPS e.max CAD	88.9 (1.3) A
Rosetta SM CAD	79.8 (4.7) B
T-Lithium CAD	76.3 (2.2) B
IRIS CAD	32.3 (5.7) C

*Different uppercase letters indicate significant difference between groups; Tukey HSD test ($p<0.05$).

4. Discussion

The results of the present study provided evidence that the new method was reliable for detecting differences in the average size and percentage of crystals among the distinct lithium disilicate glass-reinforced CAD-CAM ceramic materials tested. Thus, the null hypothesis tested was rejected. To the best of authors' knowledge, this is the first study to use the Image J software as a tool to quantify the crystal microstructure of lithium disilicate glass-reinforced ceramics or other dental ceramics as well. As a new accurate methodology, distinct types of ceramics could be also evaluated using this approach, with positive clinical implications.

Lithium disilicate ceramics have been routinely used for dental rehabilitation around the last 20 years, since they present favorable properties, such as good fracture strength, satisfactory bond strength to resin cements when properly surface treatment is performed, and satisfactory aesthetics (Poggio et al., 2017). At the beginning, a company developed and owned the patent for this material, being the only to make the product commercially available for the dental market. After the patent expired, other companies started to produce versions of this glass dental ceramic. However, there are few studies comparing the differences among the lithium disilicate-based ceramics recently introduced into the market, and whether they have the same quality in relation to the physical and mechanical properties as compared to the precursor system (Tavares et al., 2020). Thus, SEM assessments can be helpful to better investigate the structure of these new lithium disilicate glass-reinforced materials which become recently available. However, this can be challenging due to the complex structure of some ceramic materials, and the association of SEM images with an image processing software can allow more consistent analyses.

The Image J is software not doted of AI resources, and consequently, it requires technical knowledge regarding the forms and conformation of the objects to be analyzed during image processing steps. Therefore, 4 distinct lithium disilicate glass-reinforced ceramics were evaluated by the new method proposed, in order to assess whether a difference can be found in the microstructure of the materials evaluated in relation to the size and percentage of the crystals. Intending to improve this analysis process, the use of

a open-access image processing software with suitable functionality and accuracy could be a powerful tool for further studies. These attributions scientifically validate the ImageJ software as an excellent resource for biomedical image analysis. Additionally, the present study aimed to present ImageJ software as an appropriate tool for analyzing the microgeometry of different dental ceramics.

The microstructure of lithium disilicate crystals before heat treatment (crystallization) presents an acicular form; and after heat treatment, the crystals appear larger and more elongated (Silva et al, 2017). This is in accordance with our findings, in which some groups showed similar characteristics at the SEM analysis after heat treatment. However, even with the absence of reports in the literature, the microstructural arrangement of T-Lithium CAD and IRIS CAD groups presented markable differences when compared to the IPS e.max CAD and Rosetta SM CAD groups. This could determine changes in the slow crack growth pattern (fatigue), since this mechanism is related with the microstructure of glass ceramics (Silva et al, 2017). During the heat treatment process, the ceramic block changes from blue color to the original shade and translucency chosen. In this state, the ceramic contains 70 vol% of crystals with approximately 1.5 mm in size and its strength increases considerably (±360 MPa) (Schneider et al., 2012; Culp and McLaren, 2010). Further studies are needed to determine the differences between the process of crystal growth and the resulting microstructure of the lithium disilicate glass-reinforced ceramics tested, mostly for IRIS CAD and T-Lithium CAD materials, which showed discrepant results comparing to the other ceramics evaluated. Also, it is important to determine their fracture resistance and slow crack growth pattern after heat treatment. The precursor system, IPS e.max CAD, has shown more regular crystal size and higher crystal percentage per area than the other lithium disilicate glass-reinforced ceramics evaluated.

The Image J software is presented as an accurate and reliable tool to determine the average size and percentage of crystals in SEM imagens from lithium disilicate glass-reinforced ceramics. For the present study, the software assigns the use of tools such as Zoom in intent to determine the measurements more precisely. Due to the scarce literature, this method could be considered a suitable scientific approach for analyzing the microstructure of other dental ceramics as well. The binarization process during image processing is an extremely important step in determining the area representing ceramic crystals in an SEM image. From this process, the total gray matter is presented as part of

the crystalline matrix of these ceramics. Due to the presence of a massive number of crystals, the authors consider that the use of threshold instead of manual counting makes the analysis more accurate and easier to perform.

However, the new proposed methodology is limited for some reasons: it is not able to accurately distinguish the limit between the crystalline-glass matrix interface in the image and, therefore, it can generate higher or lower values regarding the area; and depends directly on parameters defined by the operator in the software. Thus, a previously trained operator must indicate an appropriate threshold for each SEM image to better characterize the microstructure. Further tools should be explored as well as new studies to improve the accuracy of microstructural analyzes of dental ceramics.

5. Conclusion

- The ImageJ software can be an accurate and reliable tool to determine the average size and percentage of crystals in dental ceramics;
- The IRIS CAD lithium disilicate glass-reinforced ceramic showed a more deficient crystalline matrix and differences on the average crystal size when compared to the other ceramic materials evaluated;
- More regular crystals and higher percentage of crystals were observed for the IPS e.max CAD ceramic.

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Figure legends

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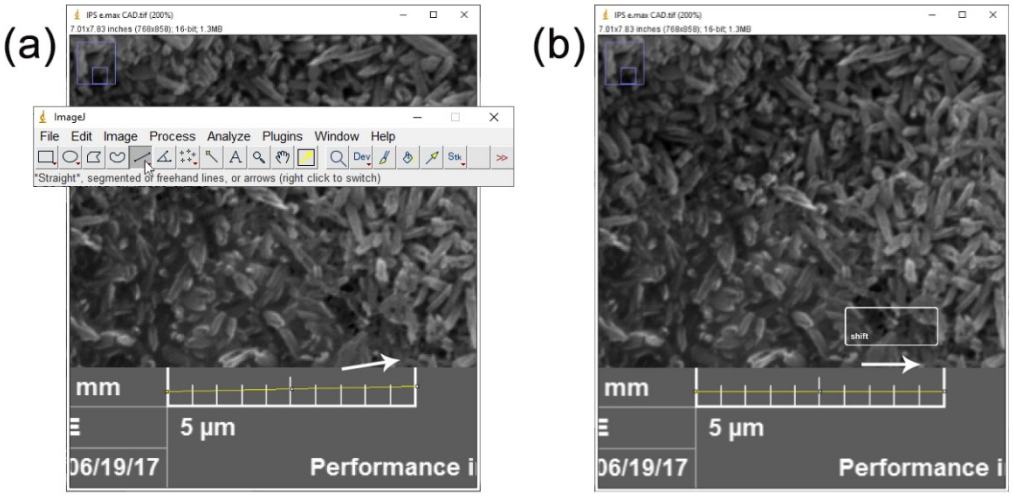

Figure 1AB: The Straight tool is selected, and a straight line is drawn under this scale

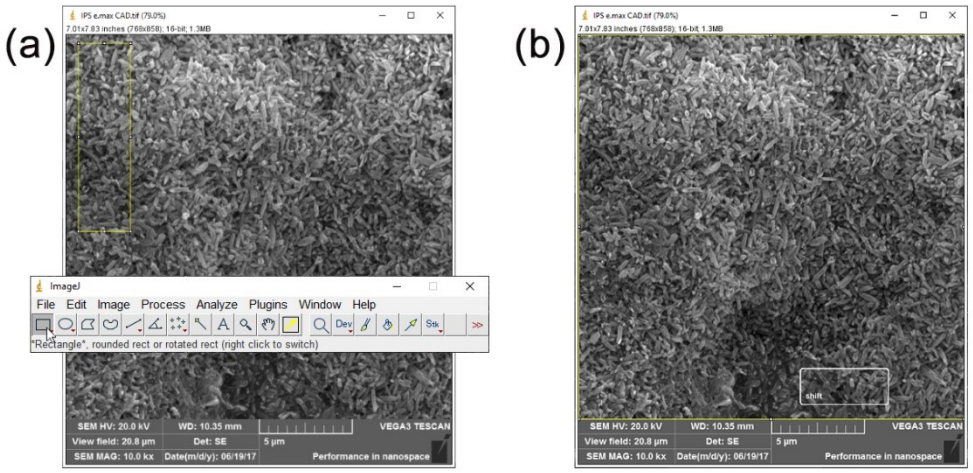

Figure 2AB: the operator should use Shift key to create a perfect square during the process

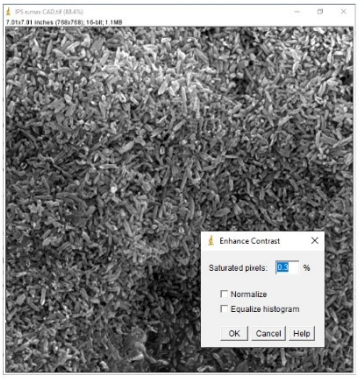

Figure 3: In the Saturated pixels board, 0.3% was used as the standard value for all groups tested in this study

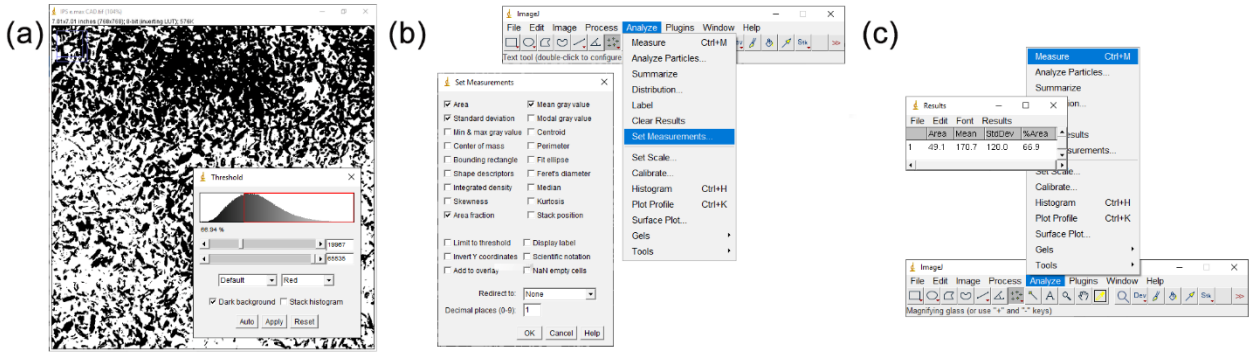

Figure 4A: The operator was trained to establishing the threshold only in the areas corresponding to the crystalline

Figure 4B: The Area Fraction provide the percentage of pixels in the image or selection that have been highlighted in red color using Threshold tool.

Figure 4C: Data were obtained for each group by using the Analyze option in the menu, followed by Measure.

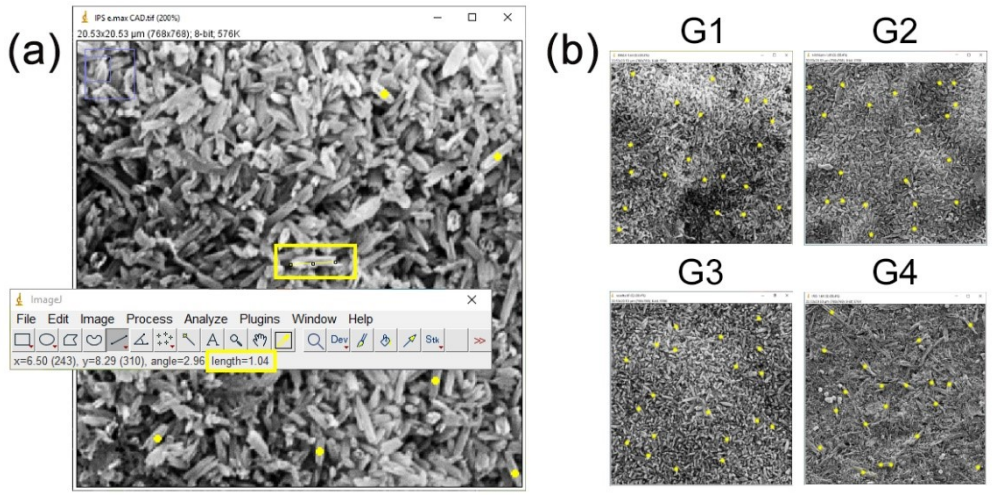

Figure 5A: The measurements were taken individually by each operator; a line was individually traced along each selected crystal with the Straight tool.

Figure 5B: After measured the crystal use a tool that allowed to mark them and avoid measuring the crystals again.

CAPÍTULOS

3.3. CAPÍTULO 3

Artigo será submetido ao periódico Brazilian Oral Research

Microstructural and mechanical analysis of four CAD-CAM lithium disilicate glass-reinforced ceramics

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Abstract

The aim of this study was to analyze the structural, morphological, and mechanical properties of four different lithium disilicate glass-reinforced ceramic materials for CAD-CAM systems (IPS e.max CAD, Rosetta SM CAD, T-lithium CAD and IRIS CAD). Four methodologies were used for evaluating the dental ceramics: the ceramic microstructure was analyzed using x-ray diffraction (XRD) (n=2); the morphological properties were analyzed by scanning electron microscopy (SEM), after hydrofluoric etching (n=2); the flexural strength was measured using the biaxial flexural strength test (n=10); and the bond strength of a self-adhesive resin cement to the ceramics was verified under a microshear bond strength test (n=10). After performing the tests, the data were tabulated and analyzed using one-way and two-way ANOVA followed by Tukey HSD test. All tests were performed using α=0.05 significance level. High peak positions corresponding to standard lithium metasilicate and lithium disilicate with similar intensities were observed for all ceramics in the XRD analysis, before and after heat treatment, respectively. SEM morphological analysis of the ceramics after acid etching showed different patterns of crystalline structure, mainly for the IRIS CAD group. The lowest biaxial flexural strength values were observed also for IRIS CAD group (P = 0.024). The bond strength values and failure modes of the resin cement were similar among the distinct ceramic systems, regardless of the storage times. Based on the results, no significant differences were found in the XRD analysis, which showed similar crystalline structures for the ceramics evaluated. Despite no significant differences were detected for the bond strength among the ceramics, the IRIS CAD group showed very distinct morphological characteristics in the SEM analysis and the lower biaxial flexural strength results as compared to the other ceramic systems.

Keywords: Dental Ceramics. Lithium disilicate. Physical Properties.

1 - Introduction

The evolution of restorative dentistry and the computer-aided design/computer-aided manufacturing (CAD/CAM) systems, allow the dentistry to indicate the best material for your patient.Currently, lithium disilicate glass-reinforced ceramic is one of the restorative materials most frequently used to produce indirect all-ceramic restorations[1].Today, several types of dental ceramics are available on the market, and due to the increased demand of patients and practitioners in terms of esthetics, biocompatibility, and long-term survival of restorations, all-ceramic systems have been continuously developed[2].

The lithium disilicate glass-reinforced ceramic can be used in the rehabilitation of single-unit and multiunit dental and implant-retained restorations[3].This class of dental ceramics has good physicomechanical properties, such as good esthetics, satisfactory bond strength to resin cements when proper surface treatment is performed (±18.00MPa), and good flexural resistance (±380MPa); All these factors favoring the growing acceptance of these ceramic materials[4,5].

There are two kinds of lithium disilicate glass ceramics available to use, heat pressed ceramic ingots and CAD/CAM ceramic blocks. Heat pressed lithium disilicate glass ceramic ingots are fully crystallized, and after heat pressing treatment, the ingots could be pressed into the designed shape such as crowns or inlays, being a process used in dental laboratories[7]. Usually, CAD/CAM lithium disilicate glass-reinforced ceramic blocks are partially crystallized to facilitate the milling process controlled by computer. After milling, the restoration undergoes a post-milling heat treatment for final crystallization to reach the strength and optical characteristics designed[8]. The digital workflow available with chairside CAD/CAM technology enabled dental clinicians to perform all the procedures required for obtaining an indirect restoration, making it possible even in single appointments, what increased the performance, since it is a faster method when compared to traditional techniques for obtaining the final restorations[9].

Lithium disilicate glass-reinforced ceramics were introduced back in the late 1990's as an exclusive product released by Ivoclar Vivadent. The material has evolved and in 2006 it emerged as the new generation of heat-pressed ceramics and after also available as CAD/CAM blocks, featuring improved mechanical and optical properties over the first-generation material. The patent

of this product has recently expired, and several manufacturers are producing other lithium disilicate-based ceramics. These manufacturers suggest that the new ceramics have mechanical, structural and morphological properties similar to the primary IPS e.max system (Ivoclar Vivadent, Schaan, Liechtenstein), but few studies comparing these materials are available[10].

The mechanical and morphological properties of the dental ceramics are influenced by its crystalline structure[11,12]. The x-ray diffraction analysis (XRD) are commonly used to evaluate the structural properties of ceramics, allowing to identify the peaks of the crystals present in the ceramic material and its crystalline phase[10-13], The scanning electron microscopy (SEM), could be used to analyzed the morphological characteristics of dental ceramics, with high resolution emission field protocols that enable to determine the size and shape of the crystal grains observed[14]. The flexural strength test is important to evaluate the flexural modulus and maximum force to fracture of dental ceramics, and these results are important for characterizing the load capacity of the material[15]. Additionally, the interaction of glass ceramics with resin cements and their bonding capability to these cements are important clinical parameters, modulated by the composition and susceptibility of these ceramics to surface treatment with hydrofluoric acid etching associated to silane coupling agents[16]. The microshear bond strength test is commonly used to evaluate the bond strength of resin cements to ceramic materials and characterize the failure patterns[17].

Thus, the aim of this study was to compare four distinct lithium disilicate glass-reinforced ceramics for CAD-CAM, using different methodologies: XRD, SEM, biaxial flexural test and microshear bond strength. The null hypothesis tested was that no differences would be detected in the microstructural and mechanical properties of the lithium disilicate glass-reinforced ceramic materials evaluated.

2 - Methodology

Four CAD-CAM lithium disilicate glass-reinforced ceramics were evaluated using HT-A2 C14 blocks: IPS E.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein), Rosetta SM CAD (Rosetta SM, Hass, Gangneung, Coréia), T-lítio CAD (Talmax, Curitiba, Paraná, Brasil) and IRIS CAD

(Mainland, Tianjin, China). The specimens for each ceramic material were prepared according to the respective methodologies, as follows:

2.1 - X-ray diffraction (XRD)

X-ray diffractogram patterns were performed (n=2) for each experimental group, at room temperature (25 °C) using a diffractometer (XRD-6000, Shimadzu Corp., Tokyo, Japan) with monochromatic Cu-K_1 (λ=1.54056Å) radiation. XRD scanning was carried out using the Cu-K1 emission (λ = 1.54056Å), generating a current of 15mA, 30kV, and a wavelength equal to 1.5406Å, with a continuous scanning interval of 2θ (20-80), with a step of 0.02s. The XRD patterns were compared with the JCPDS (Joint Committee on Powder Diffraction Standard) to identify the type of crystal and crystalline phase of both ceramic materials.[2-8] Two specimens were selected from each experimental group in different stages (pre-crystallized and crystallized) to perform the structural analysis. The crystallized specimens underwent heat treatment using a special furnace (Programat P300, Ivoclar Vivadent, Schaan, Liechtenstein), with the P91 program. This furnace reaches the maximum temperature of 845°C, then stabilizes for a period of 7 minutes, after which it starts to cool slowly to prevent thermal shock.

Traditionally, the structural analysis test requires the specimen to be reduced to powder, but the ceramic block was too rigid. Alternatively, the specimens were cut to a size of approximately 1 cm^3 using a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Then, they were placed on a metallic device filled with aluminum particles to start the test, which lasts an average of 1 hour and 40 minutes, for each specimen evaluation. Two graphs (diffractograms) were obtained for each experimental group, which were qualitatively interpreted.

2.2 - Scanning Electron Microscopy (SEM)

The morphological structures of the ceramics were analyzed following the heat treatment (crystallization) of the specimens. Initial preparation consisted of obtaining specimens (n=2) for each group with dimensions of approximately 3.0 mm^3 using a diamond saw (Isomet 1000, Buehler). Next, they were sectioned from the ceramic blocks at the pre-crystallized stage and

submitted to the crystallization process as previously described. All the ceramic surfaces were sequentially polished using silicon carbide papers (#600, 800, 1200, Norton, Guarulhos, SP, Brazil) for 20 s, and then etched with 10% hydrofluoric acid (HF) (Condac Porcelana, FGM, Joinville, SC, Brazil) for 20 s. The specimens were washed using 70% alcohol and distilled water in an ultrasonic bath for 5 min, to remove the HF etching residues. Afterwards, the specimens were mounted on coded brass stubs and sputter-coated with gold palladium for 60 s at 45 mA (QR 150ES, Quorum Technologies, Laughton, Lewes, UK) to obtain better quality SEM images allowing visualization of the crystals. Three images of each ceramic material were obtained using SEM (VEGA 3, TESCAN, LMU, Kohoutovice, Czech Republic), with an accelerating voltage of 20.0 kV and original magnification of 20,000×. The images were previously analyzed and then obtained and by a skilled operator. The images obtained from each experimental group were qualitatively analyzed.

2.3 - Biaxial flexural test

Ten ceramic discs (ø: 12 mm; thickness: 1.2± 0.2 mm – ISO 6872[18]) were obtained from the blocks of each experimental group evaluated, by milling the blocks into cylinders using a mechanical lathe with a diamond cutting instrument under irrigation, and then cutting the discs from the resulting cylinders in a diamond saw (Isomet 1000, Buehler). Subsequently, the specimens were sequentially polished using silicon carbide papers (#600, 800, 1200, Norton, Guarulhos, SP, Brazil), in a polishing machine (400 CS, EXAKT Technologies, Oklahoma City, OK, USA), under running water at 300 rpm. Lastly, the specimens were washed and stored dry until testing. All specimens underwent heat treatment (crystallization), carried out as previously described (n=10).

In the testing process, the specimens were placed in a mechanical testing machine (DL2000, EMIC, São José dos Pinhais, PR, Brazil), and the flexural strength was measured using a biaxial flexural strength test scheme, according to ISO 6872[18] standard for dental ceramics. To support the specimen, three hardened steel balls 3.2 mm in diameter, were positioned 120° apart on a support circle with 10 mm diameter. The disc shaped specimens were positioned concentrically on the supports and loading was applied at their center with a flat piston 1.2 mm in

diameter, at 0.5 mm/min cross-head speed until fracture.

Biaxial flexural strength (MPa) was calculated using the following equation, according to ISO 6872 guidelines[18].

$$S = -0,\ 238\ 7P\ X - Y\ /d2 \quad (1)$$

where, *S* is the maximum tensile stress (MPa) at the center of the specimen, *P* is the total load causing fracture (N), $X = 1 + v\ In\ (r2/r3)2 + (1 - v)/2$ ($(r2/r3)2$, and $Y = 1 + v\ 1 + In(r2/r3)2 + (1 - v)(r2/r3)2$, in which, v is the Poisson's ratio (assumed as 0.25); $r1$ is the radius of support circle (mm); $r2$ is the radius of loaded area (mm); $r3$ is the radius of specimen (mm); and *d* is specimen thickness at fracture origin (mm). Statistical analysis was performed using One-way analysis of variance (ANOVA) followed by the Tukey HSD test.

2.4 - Bond strength test

Ceramic slices were obtained from the CAD-CAM blocks of each experimental group (n =10) to perform the microshear bond strength test. One 3.0 mm slice was obtained per block using a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling. The ceramic slices were crystallized using the protocol described previously, and then embedded in polystyrene resin (Aerojet, Santo Amaro, SP, Brazil). Next, all the ceramic surfaces were sequentially polished using silicon carbide papers (#600, 800, 1200; Norton, Guarulhos, SP, Brazil) for 20 s each, and the specimens were washed and stored dry until testing.

Afterwards, the top surface of each ceramic slice was etched with hydrofluoric acid 10% (Condac Porcelana, FGM, Joinville, SC, Brazil) for 20 s, followed by rinsing with water for 30 s and air-drying for another 30 s. Then, the specimens were washed using 70% alcohol and distilled water in an ultrasonic bath for 5 min to remove the HF etching residues. One layer of a silane-coupling agent (RelyX™ Ceramic Primer, 3M-ESPE, St. Paul, MN, USA) was applied actively in the ceramic surface for 20 s, and left to react for 60 s. A self-adhesive resin cement (RelyX U200, 3M-ESPE) was manipulated according to the manufacturer's directions, and inserted into silicon molds (1.0 mm in diameter and height) positioned above the ceramic surface.[16] After 5 min of preparation, each resin cement cylinder was light-activated for 40 s using a polywave LED light-

curing unit (VALO, Ultradent, South Jordan, UT, USA). Next, the silicone mold was removed using scalpel blades to avoid pre-loading the cylinders. Thus, six cylinders were placed on the surface of each ceramic block, spaced at 3.0 mm intervals.[16,17] The specimens were stored in 100% relative humidity with distilled water, at 37°C for 24 h previous to testing.

The microshear bond test (μSBS) was performed after positioning and fixing the specimens in a mechanical testing machine (OM100, Odeme Dental Research, Luzerna, SC, Brazil). The resin cement cylinders were then aligned in the load application direction, and then, a 0.2 mm diameter orthodontic wire (NiCr, Morelli, Sorocaba, SP, Brazil) was used to load the cement cylinders perpendicular to the ceramic surface. The crosshead speed was set at 0.7 mm/min. The bond strength of each specimen (MPa) was calculated using the following equation:

$$R = F / A$$

where R is the bond strength (MPa); F is the force required for specimen rupture (N); and A is the adhesive area of the specimen (mm²).

The procedure for the specimens tested immediately (24h - T0), consisted of individually loading the cylinders, and then, obtaining the mean bond strength among the three specimens. The remaining resin cylinders from each group were then submitted to aging by storing the ceramic specimens in 100% relative humidity at 37°C for 30 days. After this period, the resin cylinders were individually loaded (30 days - T1) using the same protocol described to obtain the mean bond strength among the specimens after the storage. Bonding strength values from T0 and T1 were compared within the groups and among the different ceramics. Statistical analysis was performed using two-way analysis of variance (ANOVA) followed by the Tukey HSD test.

After the microshear bond strength test, the fractured surface of the resin cylinders was evaluated under optical microscopy (Axiocam, Mitutoyo, Absolute, Tokyo, Japan) at 40× magnification to determine the failure mode, classified as follows: 1) adhesive failure; 2) cohesive failure in resin cement; and 3) mixed failure; 4) cohesive failure in ceramic[16].

3 - Results

3.1 - X-ray diffraction (XRD)

The XRD results are shown in Figure 1. The four ceramic materials evaluated presented comparable, narrow diffraction peaks, which confirm the formation of crystalline compounds of similar intensity. The diffraction peaks detected in the XRD patterns are characteristic of lithium metasilicate (ICCD 029-0829) and lithium disilicate (ICCD 040-0376) crystals, confirming the formation of these crystalline compounds for all the ceramic materials tested.

3.2 - Scanning Electron Microscopy (SEM)

After crystallization of the specimens, the crystalline microstructure of the crystals became denser, and the surface etching caused changes in their superficial morphology. SEM images of the etched ceramic surfaces for all the ceramic systems are shown in Figure 2. The 10% hydrofluoric acid etching for 20 s, showed that the structure of crystals observed for the IPS e.max and Rosetta SM groups is equivalent with the findings of other research[4,10], with crystals showing an elongated spindle shaped form. The T-Lithium group presented a smaller and more rounded crystal structure, but with satisfactory symmetry and quantity. The IRIS group presented a marked smaller number of crystals compared to other groups, presenting irregular shape in relation to its length and diameter.

3.3 - Biaxial flexural strength test

The mean biaxial flexural strength (MPa) values and standard deviation for the ceramic systems evaluated are presented in Table 2. The one-way ANOVA showed significant differences for the materials tested (P = 0.024). The IPS e.max (G1: 367.6±57.6), Rosetta SM (366.6±47.4) and T-Lithium (335.5±64.8) ceramics presented statistically similar flexural strength results, with slight lower numeric values observed for the T-lithium group, despite no significant differences were detected. The IRIS group (299.7±50.6), showed marked lower flexural strength values, presenting significant differences comparing to the other groups.

3.4 - Microshear bond strength test and failure mode analysis:

The mean microshear bond strength (MPa) values and standard deviation of a self-

adhesive resin cement to the distinct ceramics substrates tested, both immediately and after 30 days, are shown in Table 3. The bond strength values found were very similar for all the ceramic systems, regardless of the different storage times (±17.0 MPa). Two-way ANOVA revealed no significant differences for the bond strength between the ceramic substrates (P = 0.161), irrespective of the storage time (P = 0.712). The failure mode distribution observed for the specimens of all experimental groups was predominantly formed by adhesive failures, followed by mixed failures (Table 3), irrespective of the ceramic material or storage time (Figure 4).

4 - Discussion

The null hypothesis tested in this study was rejected, since the four distinct lithium disilicate glass-reinforced ceramics CAD-CAM systems presented similar crystalline structures and bond strength characteristics, but marked differences were observed for the IRIS CAD in relation to the topography of the lithium disilicate crystals and flexural strength as compared to the other materials. The lithium disilicate glass-reinforced ceramics are routinely used for dental purposes, due to their favorable properties, such as good fracture strength and satisfactory aesthetics[6]. Initially, Ivoclar Vivadent owned the patent for these ceramics, having the exclusivity of its production. After the end of this patent, other companies offered new versions of glass ceramics reinforced by lithium disilicate. However, literature is scarce about these materials, and it is still unknown if these ceramics present similar properties when compared to the precursor system[10].

The lithium disilicate ceramics present an important advantage over other ceramic systems, because these materials can be processed by heat-pressing methods or by digital milling, simplifying the fabrication methods when using chairside or laboratory CAD-CAM systems. This fact made IPS e.max ceramic system, one of the most used materials for producing all-ceramic restorations currently. The CAD-CAM technology and the new generation of ceramic blocks were introduced to facilitate the milling process, increase cutting efficiency, and extend the life of milling tools[19]. For this, the blocks of reinforced ceramics such as lithium disilicate must be marketed in partially crystallized state, since this intermediate state inherit a mild to moderate strength and hardness, which can be easily machined by any popular CAD-CAM system[20]. In the present study,

XRD results confirmed that the main peaks and crystalline components of all materials analyzed, at the pre-crystallized and crystallized stages, showed results consistent with the literature for the precursor system (IPS e.max)[11]. This finding indicates that the ceramic materials evaluated, presented close conversion of lithium metasilicates into lithium disilicate crystals when submitted to the heat treatment program (crystallization) used in this investigation, which is the same suggested by the processing instructions of all manufacturers for these ceramic systems.

The SEM analysis allowed the evaluation of the topography of the lithium disilicate crystals, by etching the ceramic surface with hydrofluoric acid and dissolving the glassy phase to make the lithium disilicate crystals more visible[16]. From this step, it was possible to evaluate that the morphology and quantity of crystals from IPS e.max and Rosetta SM groups was equivalent, and that T-lithium group presented smaller and more rounded crystals, but with satisfactory symmetry and quantity. The IRIS CAD group presented a smaller amount of lithium disilicate crystals, having irregular shapes in relation to its length and diameter.

After the heat treatment (crystallized stage), the crystalline microstructures became denser in the ceramic systems tested, further indicating a close conformation of the lithium disilicate crystals. A previous study has shown that these microstructures can become even denser and more homogeneous when restorations are subject to masticatory forces[4]. Lithium disilicate crystals have an average length of 0.5 μm in their pre-crystallized phase, After heat treatment, the crystalline microstructure changed into a more dense form, and the size of the crystals increased up to 1.0 - 3.0 μm.[4]

The flexural strength is defined as a material property of resisting stresses that cause bending, without fracture.[20,21] This property combines compression and tensile forces and can be measured by uniaxial or biaxial tests[22].The lithium disilicate glass-reinforced ceramics when compared to conventional feldspathic porcelain or leucite glass-reinforced ceramics present higher flexural strength[14,15] The multiple factors influencing the total strength of ceramic materials, stress rates, polishing procedures, testing method and environmental conditions[23,24]. In this study, no significant differences were found for the biaxial flexural strength of the IPS e.max, Rosetta SM and T-lithium ceramic systems. However, the IRIS CAD system presented lower flexural strength

values when compared to the other groups[10].

Lithium disilicate glass ceramic has a relatively high strength, to perform full or partial coverage restorations in anterior or posterior area[3]. The ceramic blocks from Group 4 (IRIS CAD) showed the worst results in relation to flexural strength when compared to the other groups evaluated. This fact may be credited to the lower quantity and inadequate standardization of the lithium disilicate crystals observed in the SEM images of the IRIS CAD system, what may have had a direct influence on its flexural strength results[13], and one of the great advantages of these ceramics reinforced by lithium disilicate is that they have an adequate aesthetics with good flexural strength values.

The bond strength between the tooth tissues and ceramic substrates is important to improve the stress distribution from functional loading, being determinant for the longevity of these indirect restorations[25,26], improve a mechanical resistance to failure[27]. The bonding effectiveness of resin materials may influence the prognosis of the ceramic restoration and its clinical success[16], since dental ceramics are brittle materials. The microshear bond strength test showed no significant differences between the lithium disilicate ceramic substrates to a self-adhesive resin cement after proper surface treatment ($P = 0.161$). The specimens were submitted to storage for 30 days, to simulate a possible bond degradation, but no differences were verified on the bond strength values for all the ceramic substrates comparing to the immediate test ($P = 0.712$). Despite these results after the storage, the oral environment is even more challenging for ceramic/resin cement bonding interfaces and differences on the bond strength of adhesive restorations are expected after aging[16]. The failure mode was evaluated by percentage, the most prevalent failure modes detected were adhesive and mixed, for all ceramic substrates, irrespective of the storage time.

This in vitro study presents intrinsic limitations, such as difficulty in processing and standardizing the samples of ceramic blocks to perform the biaxial flexural test and the aging of the samples, for evaluated the bond strength was performed for 1 month. Further laboratory studies on this topic can analyze other important properties for dental ceramics like, elastic modulus, thermal expansion coefficient, fracture toughness, surface hardness, color, and

translucency and new clinical trials testing this CAD-CAM lithium disilicate glass-reinforced available for use.

5 - Conclusion

Based on the results of the present study, the four CAD-CAM lithium disilicate glass-reinforced ceramic systems tested showed distinct results in relation to the microstructural and mechanical analyses. Despite this fact, all the lithium disilicate ceramics evaluated showed similar XRD diffraction peaks and compatible bond strength to a self-adhesive resin cement. The IRIS CAD system presented very distinct results regarding the size, form and quantity of lithium disilicate crystals and reduced flexural strength, what can compromise its clinical applications.

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Figure legends

Figure 1A –XDR patterns pre-crystallized

Figure 1B –XDR patterns crystallized.

Figure 2A – SEM images: IPS e.max CAD group after etching. 10,000× magnification.

Figure 2B – SEM images: Rosetta SM group after etching. 10,000× magnification.

Figure 2C – SEM images: T-Lithium CAD group after etching. 10,000× magnification.

Figure 2D – SEM images: IRIS CAD group after etching. 10,000× magnification.

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Table legends

Table 1 – Mean flexural strength (MPa) values and standard deviation (SD) for the CAD-CAM lithium disilicate glass ceramics systems (MPa)Table 2 –

Table 2 – Mean microshear bond strength (MPa) values and standard deviation (SD) of a self-adhesive resin cement to distinct CAD-CAM lithium disilicate glass ceramics systems immediately (T0) and after 30 days (T1).

Table 3 - Failure mode distribution (%) following microshear bond strength test for the CAD-CAM lithium disilicate glass ceramics systems immediately (T0) and after 30 days (T1).

Table 1 - Mean values (SD) for measure the biaxial flexural strength, of the lithium disilicate glass ceramics tested (μm).

Ceramic block	**Flexural strength** (Mpa).
IPS e.max CAD	367.55 (57.6) A
Rosetta SM CAD/CAM	366.60 (47.39) A
T-Lithium CAD/CAM	335.46 (64.80) A
IRIS CAD/CAM	299.86 (50.60) B

* Same uppercase letters indicate no significant difference between groups (Tukey HDS test – $P < 0.05$)

Table 2 - Microshear bond strength (SD) of resin cement to the lithium disilicate glass ceramics tested full crystallized, immediately (T0) and after 30 days (T1) (MPa).

Ceramic blocks	T0	T1
IPS e.max CAD	10.49±2.13	10.79±1.93
Rosetta SM CAD	10.25±1.17	9.65±2.27
T-Lithium CAD	10.83±2.27	10.96±2.49
IRIS CAD	10.35±2.25	9.98±3.63

There is not a statistically significant difference between the input groups (P = 0.878). Mean values followed by different uppercase letters in columns and lowercase letters in rows are significantly different at ($p<0.05$).

Table 3 - Failure mode distribution (%) following microshear bond strength test for CAD-CAM lithium disilicate glass ceramics systems, immediately (T0) and after 30 days (T1).

Group	Adhesive failures	Cohesive failures	Mixed failures
E.max T0	65%	15%	20%
E.max T1	70%	10%	20%
Rosetta SM T0	70%	20%	10%
Rosetta SM T1	70%	5%	25%
T-Lithium T0	70%	10%	20%
T-Lithium T1	70%	10%	20%
IRIS TO	90%	5%	5%
IRIS T1	90%	5%	5%

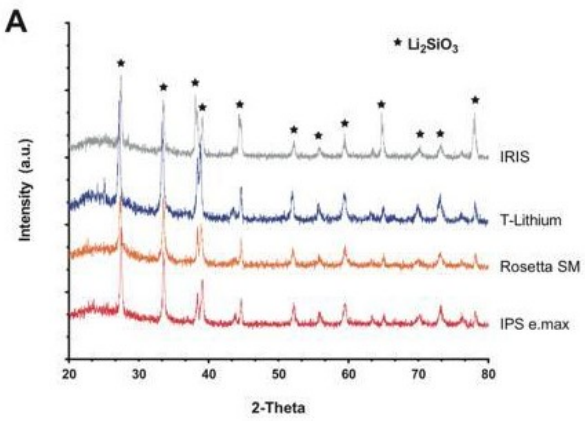

Figure 1a

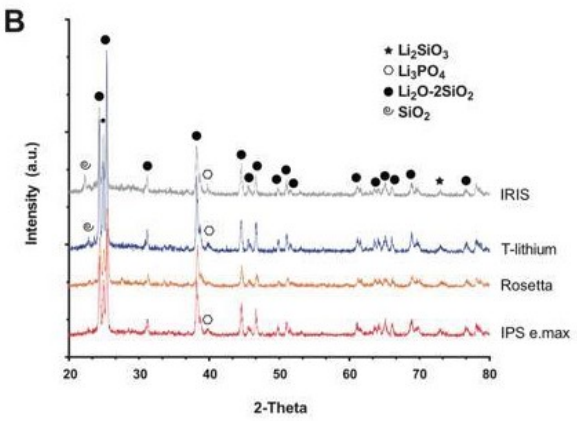

Figure 1b

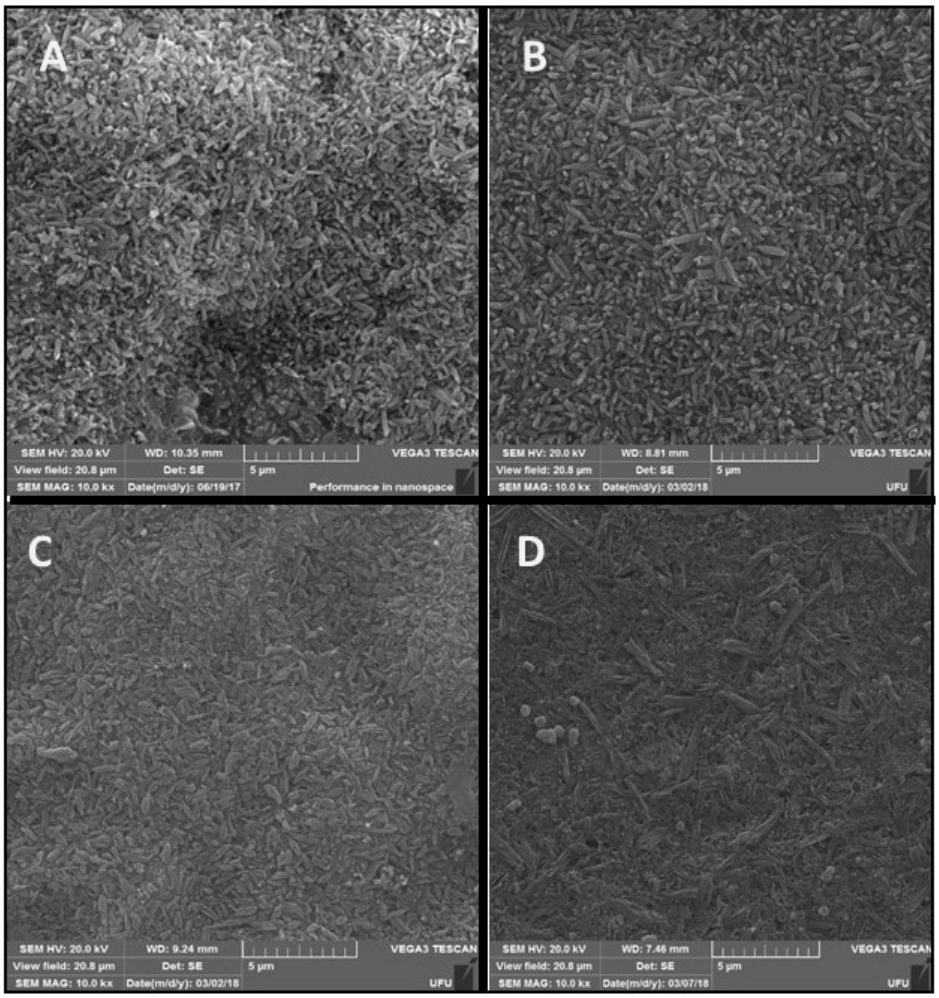

Figure 2.

CAPÍTULOS

3.4. CAPÍTULO 4

Artigo será submetido ao periódico Research society and Development

AESTHETIC AND FUNCTIONAL REHABILITATION IN A PATIENT WITH ANTERIOR SKELETAL OPEN BITE USING LITHIUM DISSILICATE GLASS-REINFORCED CERAMICS: A CASE REPORT

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RESUMO

O objetivo deste caso clínico foi relatar a substituição de coroas metalocerâmicas insatisfatórias nos elementos 12, 11, 21 e 22, por coroas cerâmicas reforçadas por dissilicato de lítio em paciente com mordida aberta esquelética anterior. Paciente de 55 anos de idade, procurou atendimento no Hospital Odontológico da Faculdade de Odontologia da Universidade Federal de Uberlândia se queixando de mau hálito e odor entre as coroas instaladas nos dentes anteriores. Após exame clínico e radiográfico foi constatado invasão de espaço biológico e a necessidade de realização de cirurgia de aumento de coroa clínica para reestabelecimento da distância biológica. Após cicatrização do periodonto, os núcleos metálicos pré-existentes foram mascarados com resina composta opaca e os elementos repreparados para receberem coroas totais em cerâmica pura. Após acabamento dos preparos, procedeu-se com moldagem de trabalho em dois passos, utilizando silicone por adição e fios afastadores. Os copings cerâmicos foram confeccionados em dissilicato de lítio e cerâmica de cobertura foi aplicada pela técnica de estratificação. Após realizar a provas das cerâmicas em relação a adaptação, função e resultados estéticos, a cimentação das coroas foi realizada utilizando isolamento absoluto modificado, seguido de profilaxia com pedra pomes e soro fisiológico, tratamento da superfície interna das peças cerâmicas e cimentação empregando cimento resinoso autoadesivo. Por último, foi confeccionada uma placa oclusal estabilizadora, para controle e tratamento da dor orofacial. Sendo assim, podemos concluir que, a mordida aberta esquelética foi um desafio para a substituição das restaurações indiretas, porém, deve-se levar em consideração todo os procedimentos necessários caso a paciente optasse pela cirurgia ortognática, devendo a mesma estar ciente das limitações do caso. Além disso, a utilização de cerâmicas reforçadas por dissilicato de lítio comprova a sua versatilidade para restaurações estéticas anteriores.

Palavras chaves: Cerâmica dental, Prótese Dentária, Mordida Aberta.

ABSTRACT

The aim of this clinical report is to present a case with replacement of unsatisfactory metal-ceramic crowns in the elements 12, 11, 21 and 22, by lithium disilicate glass-reinforced ceramic crowns in a patient with skeletal anterior open bite. A 55-year-old patient sought care at the Dental Hospital at the School of Dentistry, Federal University of Uberlandia, complaining of bad breath and odor between the metal-ceramic crowns of the antero-superior teeth. After clinical and radiographic examination, an invasion of the biological space was noticed and crown-lengthening surgery was indicated in order to restore the biological space. After healing, the pre-existing cast metal posts were masked with opaque composite resin and the teeth were reprepared for all- ceramic full-crowns. After finishing the preparations, impression was taken in two steps, using PVS and retraction cords. The ceramic copings were obtained in lithium disilicate and the veneering ceramic was applied by stratification. After testing the ceramics in relation to, fit, function and aesthetic results, cementation was performed using modified absolute isolation, followed by prophylaxis with pumice and saline, surface treatment of the ceramic restorations and luting using self-adhesive resin cement. Finally, an occlusal splint was used to control and treat the orofacial pain. Thus, we concluded that the anterior open skeletal bite was a challenge for this case; however, one must take into account the entire process that the patient would undergo if choosing for orthognathic surgery, and she is aware of the case limitations. In addition, the use of lithium disilicate glass-reinforced ceramics proved the versatility of this material for anterior aesthetic restorations.

Keywords: Ceramics, Dental Prosthesis, Open Bite.

RESUMEN

El objetivo de este caso clínico fue reportar la sustitución de coronas metalcerámicas insatisfactorias en los elementos 12, 11, 21 y 22 por coronas cerámicas reforzadas con disilicato de litio en un paciente con mordida abierta esquelética anterior. Una paciente de 55 años buscó atención en el Hospital Dental de la Facultad de Odontología de la Universidad Federal de Uberlândia, quejándose de mal aliento y olor entre las coronas instaladas en los dientes anteriores. Tras el examen clínico y radiográfico se constató la invasión del espacio biológico y la necesidad de cirugía clínica de aumento de corona para restablecer la distancia biológica. Después de la curación del periodonto, los núcleos metálicos preexistentes se enmascararon con resina compuesta opaca y los elementos se repararon para recibir coronas completas de cerámica sin metal. Una vez terminadas las preparaciones, se realizó el trabajo de moldeo en dos pasos, utilizando silicona por adición y espaciado de alambres. Las cofias cerámicas se realizaron con disilicato de litio y la cerámica de recubrimiento se aplicó mediante la técnica de estratificación. Después de realizar las pruebas cerámicas en relación a la adaptación, función y resultados estéticos, se realizó la cementación de las coronas mediante aislamiento absoluto modificado, seguido de profilaxis con piedra pómez y solución salina, tratamiento de la superficie interna de las piezas cerámicas y cementación mediante cemento de resina autoadhesivo. Finalmente, se realizó una placa estabilizadora oclusal para controlar y tratar el dolor orofacial. Por lo tanto, es posible concluir que la mordida esquelética abierta fue un reto para la reposición de restauraciones indirectas, sin embargo, es importante tener en cuenta todos los procedimientos necesarios si la paciente opta por la cirugía ortognática, y debe ser consciente de las limitaciones del caso. Además, el uso de cerámicas reforzadas con disilicato de litio demuestra su versatilidad para restauraciones estéticas anteriores.

1. INTRODUCTION

Anterior open bite (AOB) was first conceptualized in 1842, defined as the presence of a negative overbite between the upper and lower incisors, while the posterior teeth are in total occlusion (Keerthana, Thulasiram, & Kannan, 2020). AOB is among the malocclusions with the greatest aesthetic-functional interference, and can be broadly classified as dental or skeletal open bite (Burford & Noar, 2003). Skeletal open bite is usually more severe, in some cases, and there may only be contact in the last molars. Its main etiological factor is the unproportional growth of the bone bases, disproportioning several facial bones, leading the patient to a long face profile, in addition to the presence of deleterious habits, such as non-nutritive sucking and abnormal tongue function (Proffit, 2002; Burford & Noar, 2003). Early AOB treatment allows for a better prognosis and avoids more complex surgical interventions (Maciel & Leite, 2005).

Over the last decades, the use of ceramic materials in several areas of dentistry has increased and, consequently, the number of researches on the chemical properties and manufacturing techniques on the use of these materials has grown (Willard & Chu, 2018). The current use of ceramics in dentistry is wide and stems from a long historical research (Anusavice, 2013, Raposo *et al*., 2014; Butt, Thanabalan, Ayub, & Bourne, 2019). Dental ceramics are considered a good restorative option for oral rehabilitations, due to their mechanical, physical and aesthetic properties adequate for several clinical situations (Silva *et al*., 2017, Zhang et al., 2019). Its clinical indication was initially restricted to anterior regions, but after modifications on its composition, it was possible to associate important factors such as function, strength and aesthetics, allowing dental ceramic to be used also in the posterior region (Song, Ren, & Yin, 2016; Andrade, Silva, Vasconcelos, & Vasconcelos , 2017).

Dental ceramics can be classified according to type, composition/content, clinical indication and sintering temperature (Raposo *et al*., 2014). The chemical composition of ceramics is extremely important to define their possible applications. For a long time, feldspathic ceramics were the only option used in dental restorations, until the 1960s. As a versatile material, it is known for its polymorphism, despite its low tensile and flexural strength, ranging from 60 to 70 MPa. In addition, it demonstrates limited capacity for stress dissipation, allowing the accumulation of these at the ends and cracks of the restoration (Gomes, Assunção, Rocha, & Santos, 2008, Andrade *et al*., 2017). Thus, its indication

was limited only to anterior unitary all-ceramic crowns or as veneering ceramic over metallic frames to general applications (Amoroso *et al*., 2012).

In the early 90's, IPS Empress was introduced on the market aiming to improve the resistance of feldspathic ceramics. For this, the amount of leucite crystals was increased in the composition of the material and, therefore, an improvement of 30-35% was observed in flexural strength, and fracture resistance changed from 97 to 180 MPa (Gomes *et al*., 2008; Martins *et al*., 2010). Basically, the reinforcement in the feldspathic ceramic was responsible for reducing the propagation of cracks and microfractures, important points for better clinical performance. This material is indicated for rehabilitations such as inlays, onlays, veneers, anterior and posterior unitary crowns, and it is contraindicated for fixed partial bridges (Conceição & Sphor, 2005).

Thus, in 1998, the IPS Empress 2 system was introduced on the market by Ivoclar Vivadent (Garcia, Consani, Cruz, & Souza, 2011; Lien *et al*., 2015) with the intention of extending the indication of glass-reinforced ceramics. In 2006, the E.max Press (Ivoclar Vivadent, Schaan, Liechtenstein) emerged commercially as the new generation of pressable ceramics, with better results in their mechanical and optical properties (Kang, Chang, & Song, 2013). Among the ceramic systems, lithium disilicate glass-reinforced ceramic (SiO2Li2O) is indicated for most prostheses that require esthetics and longevity. The flexural strength values of this system range from 300 to 400 MPa, due to 60% to 65% of its volume being crystals. It has up to seven times more resistance as compared to feldspathic ceramics. In addition, another important feature is the possibility of being milled by CAD/CAM (Computer-Aided Design/Computer-Aided Manufactoring) technology, as was already possible with feldspathic and leucite-reinforced ceramic materials. The possibility of processing ceramics reinforced by lithium disilicate by CAD/CAM systems makes the prosthetic procedures more agile, since this is a very versatile material (Raposo *et al*., 2014; Andrade *et al*., 2017).

Other important characteristics for the clinical success of this material can be observed, such as good translucency, color stability, low thermal conduction, biocompatibility, marginal adaptation and resistance to abrasion (Conceição & Sphor, 2005; Kassardjian *et al*., 2016; Andrade *et al*., 2017). Thus, the increase in fracture resistance results in longevity and, as a consequence, can be indicated for making inlays, onlays, laminates, copings/infrastructures, single crowns and three-element fixed partial dentures up to the 2nd premolar region (Amoroso *et al*., 2012; Carvalho *et al*., 2012). However, the initial investment required for using this system may be high and failure in

their production can lead to micro fractures, which makes the ceramic fragile and compromises its clinical performance (Martins *et al*., 2010; Raposo *et al*., 2014).

Thus, the main objective of this case report is to present a case with replacement of metal-ceramic crowns of elements 12, 11, 21 and 22 by lithium disilicate glass-reinforced ceramic crowns in a patient with skeletal anterior open bite.

2. CLINICAL CASE REPORT

After healing, the pre-existing cast metal posts were masked with opaque composite resin and the teeth were reprepared for all- ceramic full-crowns. After finishing the preparations, impression was taken in two steps, using PVS and retraction cords. The ceramic copings were obtained in lithium disilicate and the veneering ceramic was applied by stratification. After testing the ceramics in relation to, fit, function and aesthetic results, cementation was performed using modified absolute isolation, followed by prophylaxis with pumice and saline, surface treatment of the ceramic restorations and luting using self-adhesive resin cement. Finally, an occlusal splint was used to control and treat the orofacial pain. Thus, we concluded that the anterior open skeletal bite was a challenge for this case; however, one must take into account the entire process that the patient would undergo if choosing for orthognathic surgery, and she is aware of the case limitations. In addition, the use of lithium disilicate glass-reinforced ceramics proved the versatility of this material for anterior aesthetic restorations.

A 55-year-old female patient sought care at the Dental Hospital at the School of Dentistry, Federal University of Uberlândia, with the main complaint regarding bad breath and odor between the metal-ceramic crowns of the antero-superior teeth. On the clinical examination, misfits were found in the margins of the crowns, associated with invasion of the biological space and gingival recession with exposure of the metallic structure (Figure 1).

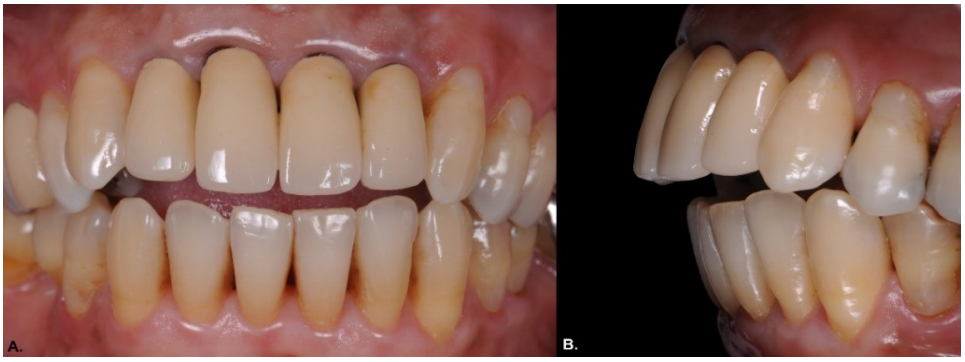


Figure 1. A. Initial front view, gingival recession with exposure of the metallic structure of the crowns 12, 21 and 22; B. Side view.

From the occlusal examination, it was possible to observe that the patient had an skeletal anterior open bite. This fact may be indicative for orthognathic surgery, but rejected the surgical procedure. The patient presented a long, narrow dental arch, with anterior roots very close and a thin gingival profile. She reported that her crowns were installed approximately 5 years ago, and all the four elements had cast metal posts. After radiographic examination, the patient was proposed to undergo crown-lengthening surgery to increase the clinical crown and to recover the biological space, followed by indirect restorative treatment using lithium disilicate glass-reinforced ceramic crowns due to the mechanical and aesthetic properties of this material.

Thus crown-lengthening surgery was performed to re-establish average 3.0 mm biological space between the end of the preparation and the top of the bone crest (Figure 2). When there is invasion of the biological space, there is damage to periodontal tissues and the final restoration. In order to have success in restorative treatment, the recovery of the biological space is then fundamental. Additionally, a marked separation of at least 1.0 mm between the roots was obtained in order to allow adequate impressions and marginal fit of the crowns.

Following 60 days of the periodontal surgery and perfect tissue healing, the metal-ceramic crowns were removed and the cast metal posts of teeth 12 and 21 were accidentally removed. Thus, due to the need to obtain new cast metal posts, cement residues were removed from the canals with using endodontic files and, after that, the root canal was isolated with petroleum jelly and red acrylic resin patterns were moulded (Dencrilay, Dencril, Pirassununga, SP) for making new cast metal posts (Figure 3). After testing the cast metal posts, they were luted using with zinc phosphate-based cement (SS White, Rio de Janeiro, RJ) (Figure 4). The temporary crowns were made by duplicating the previous crowns, employing putty VPS material (President, Coltene, Altstatten, Suíça) and acrylic resin RA (Vipi Flash® (Vipi, Pirassununga, SP, Brasil). It was possible to obtain reliable provisional according to the previous dental anatomy of the patient.

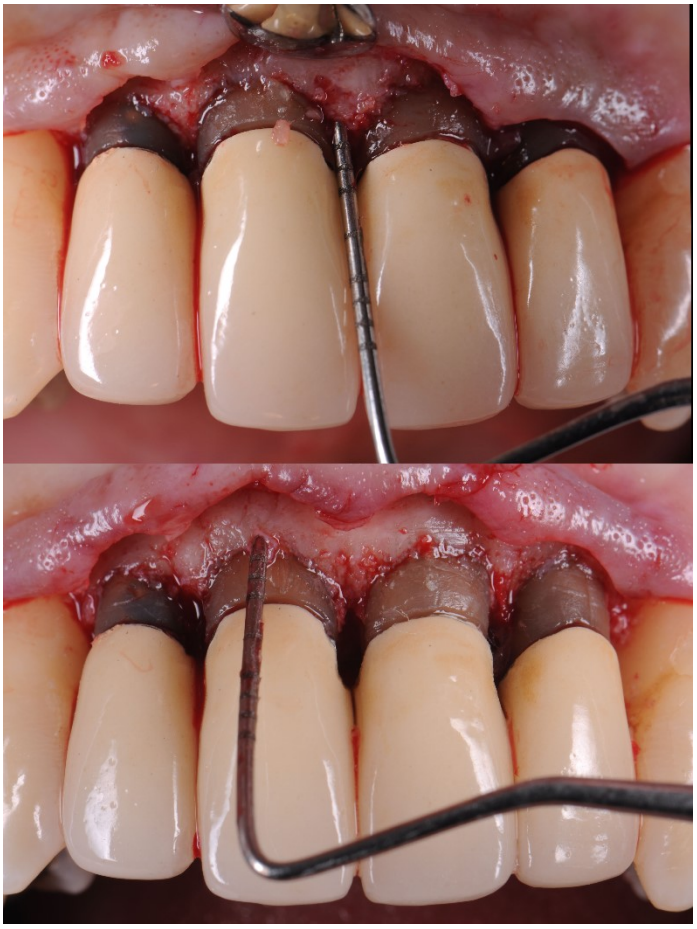


Figure 2. A. Periodontal probe showing 1.0 mm between the bone crest and the crown margin; B. Periodontal probe showing 3.0 mm distance after crown-lengthening.

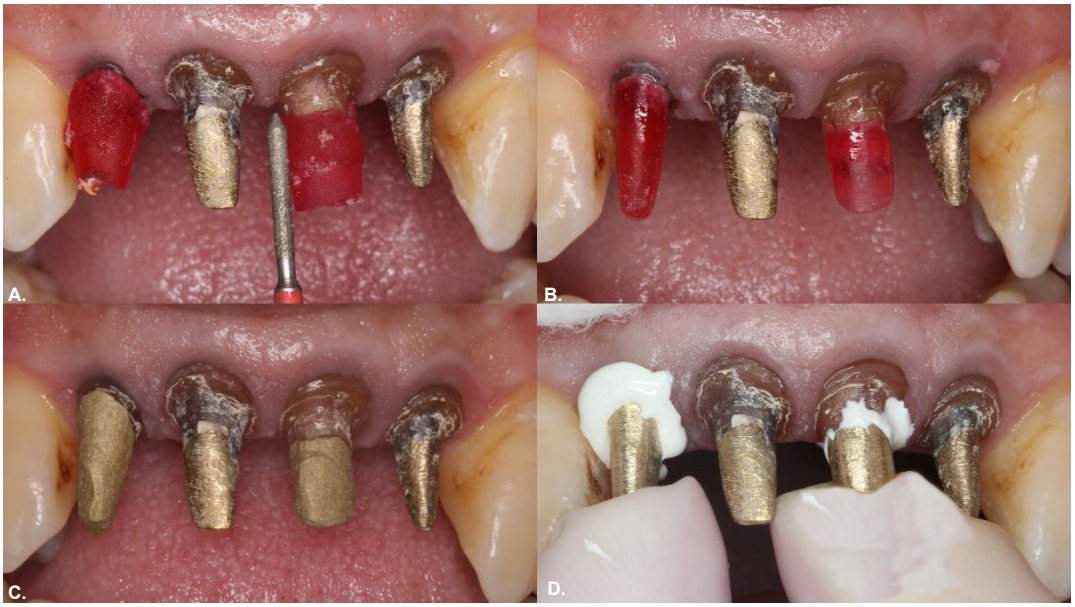


Figure 3. A. Molding patterns with red acrylic resin; B. Preparation of resin patterns; C. Adjustments of the cast metal posts; D. Lurting with zinc phosphate-based cement.

To achieve better aesthetics and improve the adhesion of the ceramic crowns, the cast metal posts were reduced and masked with an opaque composite resin. Grooves were made on the metallic surface of the coronary portion, followed by etching with phosphoric acid on the remaining dentin (Condac 37%, FGM, Joinville, SC) for 15 s, cleansing with water-spray and drying with absorbent papers. Then an universal adhesive system (Adper Single Bond Universal, 3M-ESPE, Saint Paul , MN, USA) was actively applied and light-activated for 20s. The composite resin (Filtek Z350 XT, WD, 3M-ESPE) was inserted over the entire metallic surface and light-cured on all aspects for 40 s. Thus, the metallic material became more discreet (Figure 5). Then, the refining of the preparations was performed to adjust the terminations in deep chanfers to allow the working

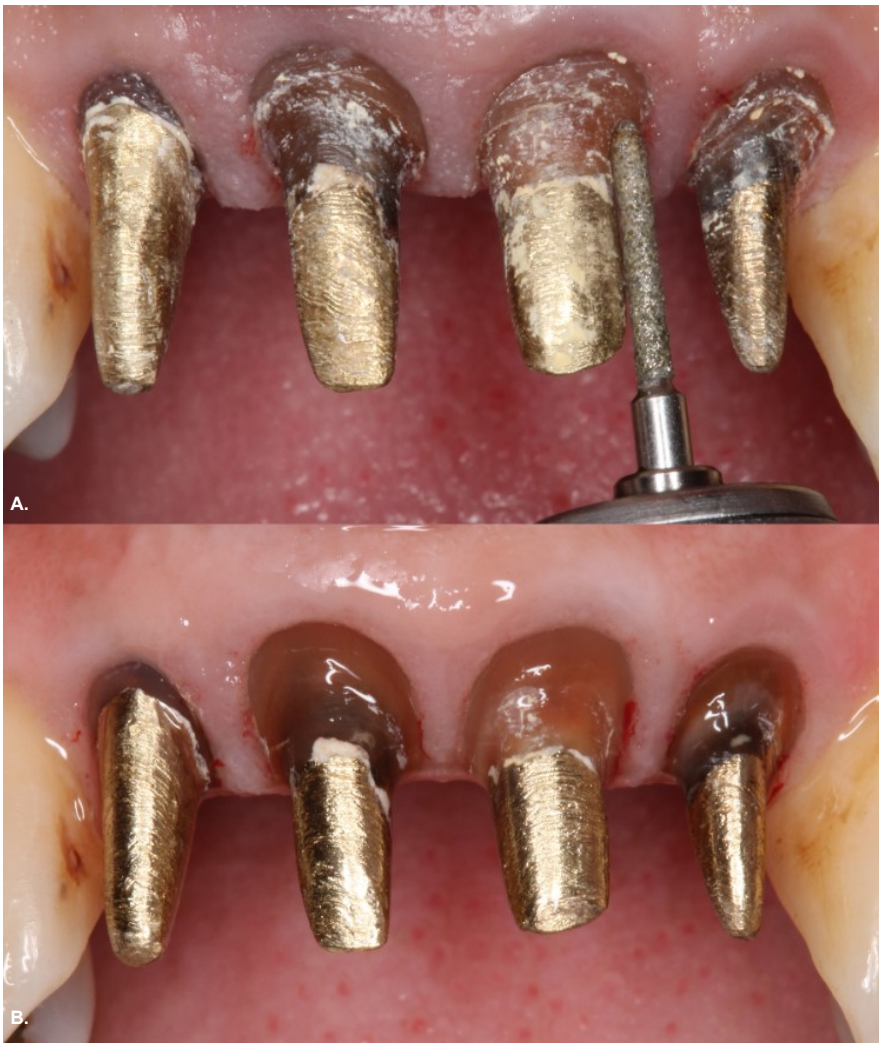


Figure 4. A. Cast metal posts; B. After teeth repreparation.

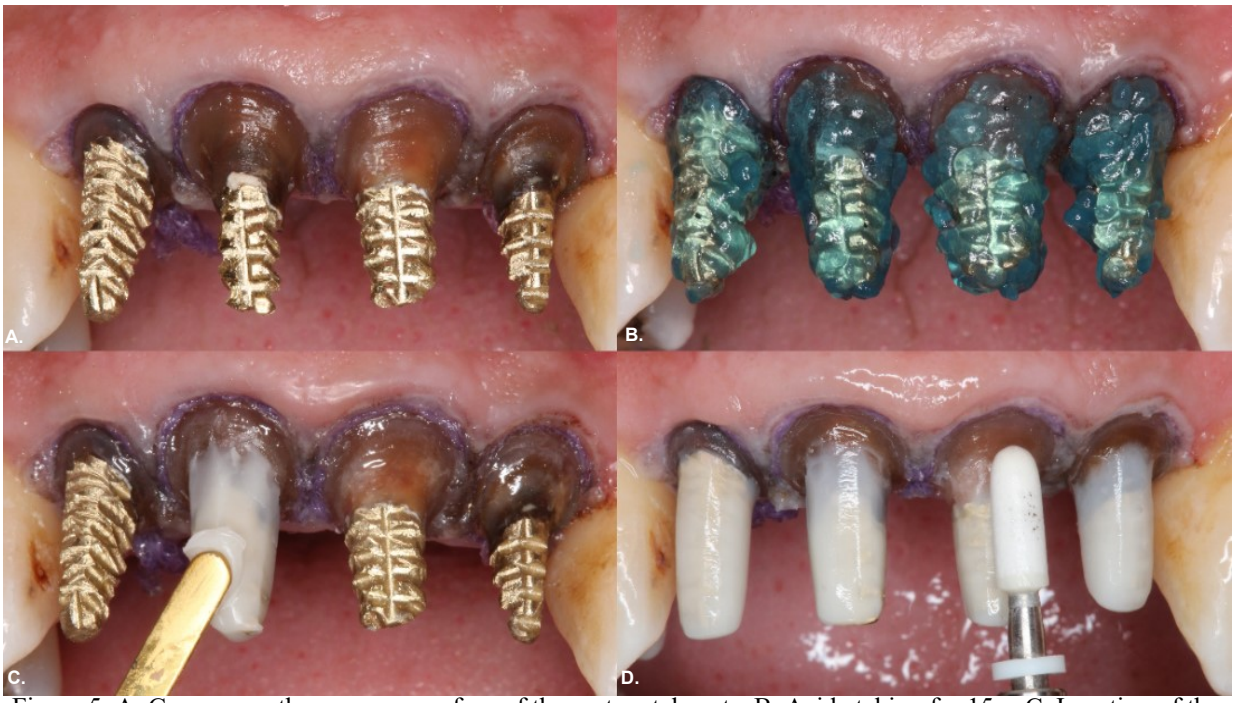
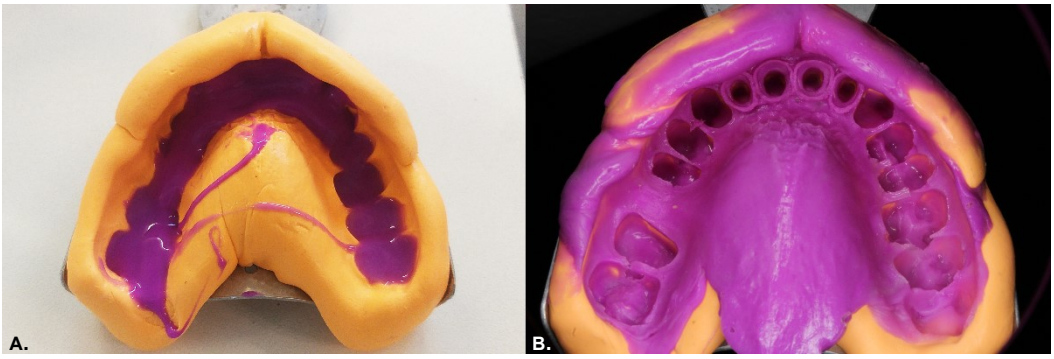


Figure 5. A. Grooves on the coronary surface of the cast metal posts; B. Acid etching for 15 s; C. Insertion of the composite resin; D. Finishing and polishing of composite resin after light curing.

impressions. First, a #000 retraction cord (Ultrapack, Ultradent, South Jordan, UT, USA) soaked in hemostatic solution (Hemostop, Dentsply) was inserted into the gingival sulcus, and subsequently, another retraction cord #00 (Ultrapack) was inserted above the first. After finishing and polishing the preparations, the tray was tested. A two-step impression technique was used, which consists of initially performing a preliminary impression with the VPS putty material (President, Coltene, Altstatten, Suíça), performing slight movements in different directions with the tray before the material is set to create space for the light material. In the second impression, the #00 retraction cords were removed, the light VPS paste (President, Còltene) was inserted under the gingival tissue and a light jet of air is applied between the preparations, followed by accommodation of the tray the heavy/light material. In this way, it is possible to reproduce the preparation and its margins with good precision (Figure 6). Then, an impression of the lower was taken with alginate following the manufacturer's recommendations (Hydrogum, Zhermack, Badia Polesine, Italy). In the same session, the intermaxillary register was taken using acrylic resin (Dencrilay, Dencril) and registration with a face bow with putty VPS for mounting the

die cast models in a semi-adjustable articulator. The temporary crowns were adjusted and relined according to the new preparations.

Figure 6. A. Two-step molding, first molding with heavy material and second molding with slurry; B. Reliable reproduction of the preparation.



From the impressions obtained, die cast models were generated in special high-resistance stone plaster (Durone IV, Dentsply), which were mounted in a semi-adjustable articulator (A7-Plus Padronizado, Bio-Art, São Calos, SP, Brazil), and later the upper model was individualized and die-cut in the region of elements 12, 11, 21 and 22. Each element was die-cut in order to facilitate the visualization of the end of the preparation for carrying out the edge sealing in the waxing. The waxing were included in a refractory material and then, the wax was removed in an oven at high temperature, thus, leaving adequate space to receive the ceramic material. This technique used lithium disilicate glass-reinforced ceramic ingots (IPS e.max, MO-2, Ivoclar Vivadent), which were subjected to high temperature and pressure in a special oven to be injected into the refractory mould, replacing the space left by the wax, forming the ceramic structure. Laboratory adjustments were carried out in the ceramic infrastructures, checking for the presence of deffects, the level of material expansion and polishing of the piece.

The ceramic structures initially obtained have not adapted perfectly to the preparations, and it was necessary to use fluid VPS (President, Còltene) to highlight the regions to be adjusted. During adjustments, the structures of teeth 21 and 22 were damaged. Due to this fact, preparations were refined and impressions were taken. For having used retractor cords recently, the new impressions were performed using the caps technique in order to not damage the periodontal tissues. The caps were made by duplicating the temporary crowns in an alginate container, placing it with the incisal face visible. Then, the crown was removed and the space left was filled with acrylic resin, with a slight excess for better handling. After necessary adjustments and relines, the

impressions were taken with a polyether-based material (Impregum Soft Regular, 3M-ESPE) (Figure 7). The caps were sent to the laboratory and new ceramic structures were made as described. After checking the ceramic structures on the preparations, an interocclusal register was made with acrylic resin, joining the four structures to keep them in position for subsequent transference impression and colour checking. Thus, a die cast model was obtained to apply the ceramic veneering on the lithium disilicate glass-reinforced ceramic structures. With the die-cast preparations and copings already adjusted, the ceramic sintering process begins. It is applied to a powder and liquid mixture with a brush, distributing the portions according to dental anatomy, dentin and enamel layers according to color selection recommended in clinical care. Through vibrations, the ceramic is condensed, and with absorbent paper, excess water is removed for greater compaction of the powder. After this process, the crowns are fired in an oven according to the manufacturer's instructions. The adjustments are completed in the laboratory.

Then the provisional are removed and the preparations are sanitized so that there is no trace of temporary luting material. With the parts in position, the proximal contact surfaces are analysed, using carbon paper strips (Accufilm II, Parkel). Too extensive contact can cause hypertrophy of the interdental papilla. With abrasive diamond rubbers, adjustments are made to the pieces with movements from cervical to incisal. At the end, a dental floss test is performed, checking the resistance between the contact surfaces. Then the entire extension of the cervical region is analysed for the presence of periodontal tissue ischemia. Then the functional adjustment are performed, the patient slowly performs maximum intercuspation while the functional contact points are checked. With a strip of carbon paper, the contact points are checked tooth by tooth for premature contacts or absence of function. In this case, the patient has anterior open bite, and no previous contact points were observed. After the adjustments in excursive movements, the shape, contour and colour are analysed. The part returns to the laboratory for extrinsic characterization. After making up the piece, the glazing step is carried out.

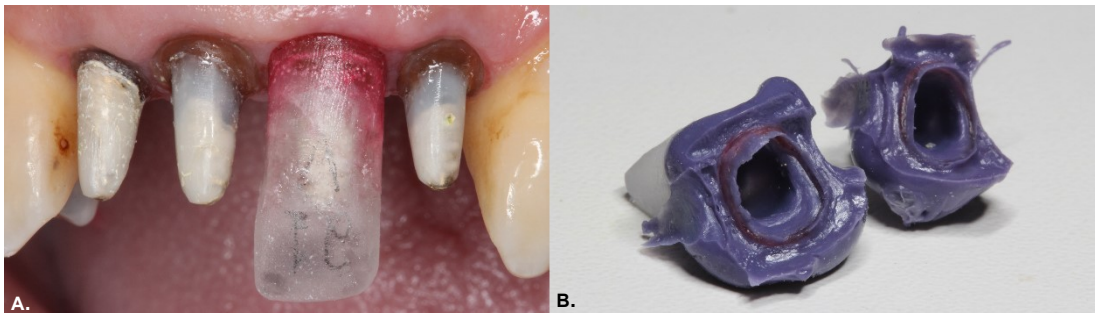


Figure 7. A. Relined cap in position after adjustments; B. Impression of the preparations with a cap and polyether-based material..

For luting the ceramic restorations, modified absolute isolation was initially performed, followed by prophylaxis in the preparations using pumice slurry (Figure 8). The surface treatment of the lithium disilicate glass-reinforced ceramic crowns was performed by etching the inner surface with 10% hydrofluoric acid (Condac Porcelain 10%, FGM) for 20 seconds. Immediately after, the crowns were submitted to an ultrasonic bath with 70%

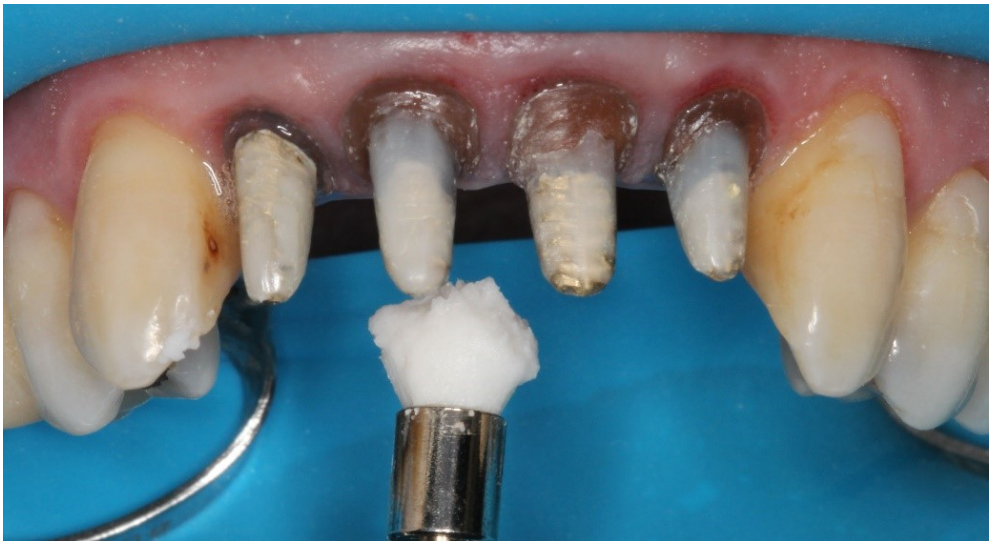


Figure 8. A. Modified absolute isolation and prophylaxis of preparations with pumice slurry.

alcohol por 3 min to remove remaining crystals, followed by water rising and air drying. After vigorous drying, a silane-coupling agent (Prosil, FGM) was actively applied for 20 s with repetitive movements on the inner surfaces of the crowns, allowing to react for 60 seconds (Figure 9).

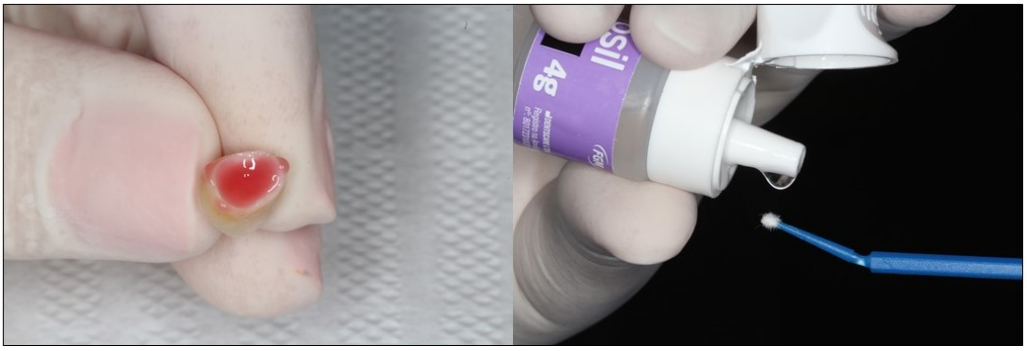


Figure 9. A. 10% hydrofluoric acid etching of the crowns for 20 se; B. Silane coupling agent.

Cementation of the crowns was performed using a self-adhesive dual-cure resin cement (SeT PP, A1, SDI, Victoria, Australia) (Figure 10) associated to self-mixing tips, which allowed direct insertion of the cement into the crowns. After 1 min, cement excess was removed, and chemical setting time of the resin cement was awaited for 5 min. Subsequently, each face of the crowns was light-activated for 60 seconds with a cordless LED unit (Radii Cal, SDI) (Figure 11). The patient was instricted in relation to care, maintenance, and hygiene.

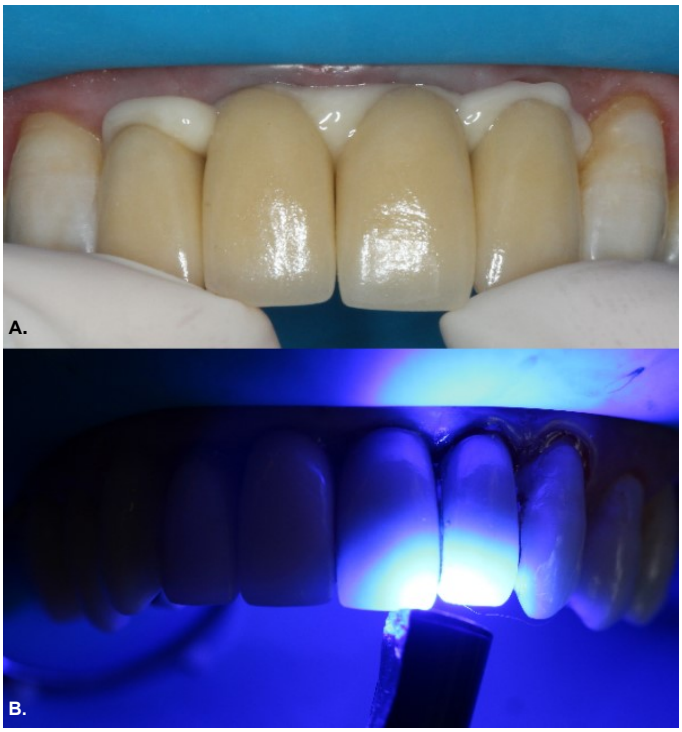


Figure 10. A. Self-adhesive resin cement; B. Light-curing for 60 s on each aspect.

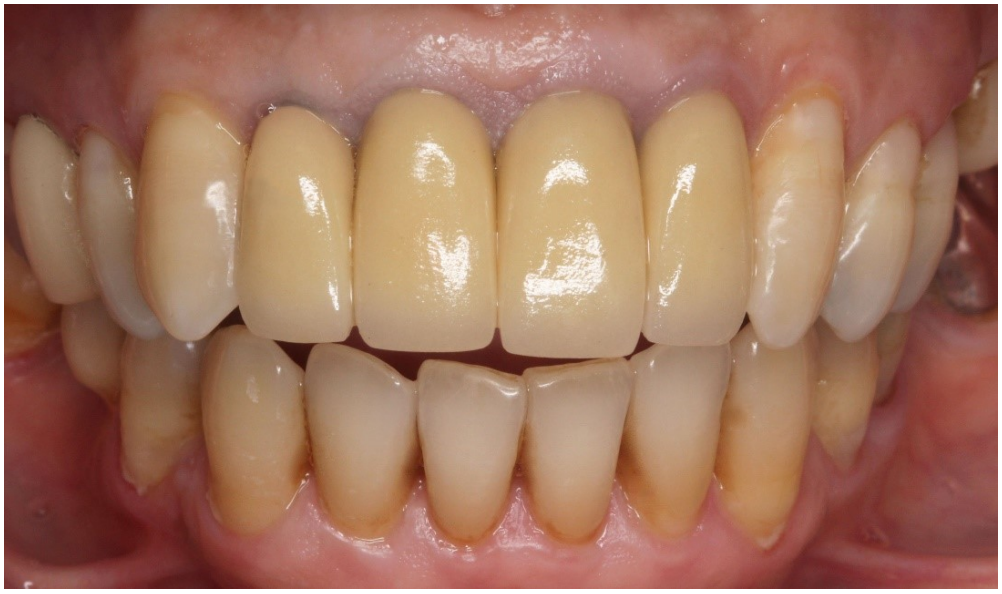


Figure 11. Adequate final aspect of the case after rehabilitation of teeth 12, 11, 21 and 22 with lithium disilicate glass-reinforced ceramic crowns.

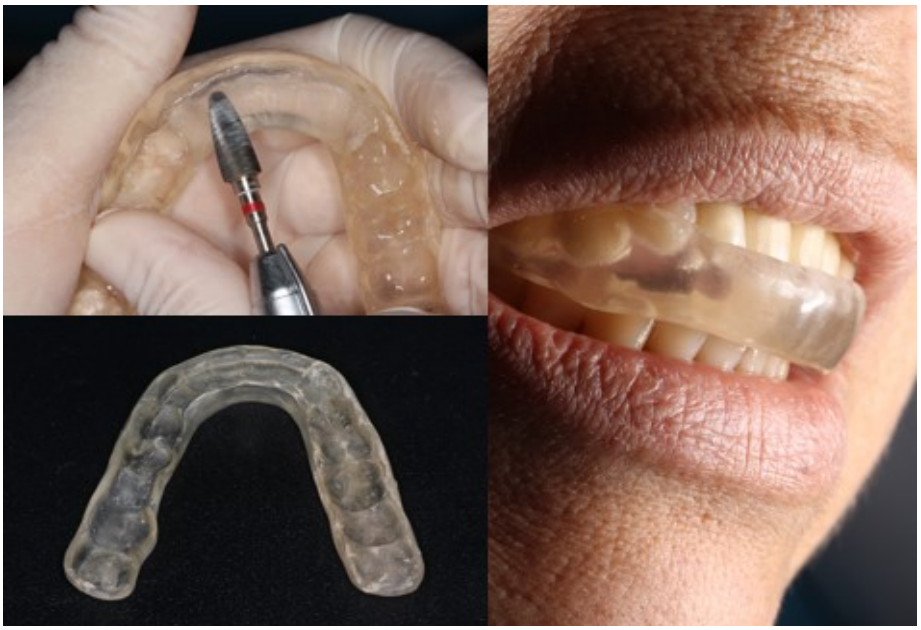


Figure 12. Occlusal splint installed following oral rehabilitation.

After rehabilitation was completed, a stabilizer occlusal splint was obtained. Due to the patient's occlusal imbalance and other external factors, she has reported orofacial pain and nocturnal bruxism. Initially, impression of both arches were taken with alginate (Hydrogum V). Then, a Lucia's JIG was made of acrylic resin (Dencrilay, Dencril, Pirassununga, SP) for deprogramming the proprioceptive memory and recording a centric relationship, allowing for stable accommodation of the condyles in the mandibular fossae. The interocclusal registration was performed with acrylic resin (Dencrilay, Dencril). And finally, the registration with the facebow was obtained for mounting the cast models on a semi-adjustable articulator. After the occlusal splint was installed, the necessary adjustments were made to bring occlusal balance and allow adequate excursive mandibular movements. Lastly, final polishing of the occlusal splint was carried out (Figure 12).

3. DISCUSSION

Ceramics as a restorative material have been used in Dentistry for over two hundred years and this type of material has been widely used due to the high demand for aesthetic restorations, concomitant with its great development in terms of physical properties. Among the available restorative materials, the aesthetic potential and biocompatibility of ceramics may be considered unique (Pagani, Miranda, & Bottino, 2003). In this sense, ceramics have good structural, morphological and mechanical properties, in addition to high potential to biomimetic dental characteristics, such as: translucency, fluorescence, chemical stability, biocompatibility, high compressive strength, linear thermal expansion coefficient similar to tooth structure, thermal conductivity similar to dental tissues and color stability (Kreidler, 2008; Zarone *et al*., 2016).

In the case presented, it was decided to use the ceramic system reinforced by lithium disilicate crystals. This system emerged to improve the clinical performance of conventional feldspathic ceramics with respect to mechanical properties (Aguiar *et al*., 2016). Its glassy structure has needle-shaped crystals, arranged in an intertwined manner, favoring its high flexural strength and making it difficult for crack propagation inside it (Kina, 2005; Carvalho *et al*., 2012). The longevity of lithium disilicate led to it being recommended for most rehabilitations.

The patient's anterior open bite is a challenge, both for functional rehabilitation and for the esthetics of the restoration. There are several etiological factors related to this vertical

malocclusion, including the presence of oral habits (Rijpstra & Lisson, 2016). Treatment depends on several factors, the patient's age, concerns and expectations. And for patients with permanent dentition at an advanced age, only surgical intervention can solve their vertical dimension combined with previous orthodontic treatment (Burford & Noar, 2003; Maciel & Leite, 2005). In some cases, the open bite can be resolved through elongated dental restorations, however, in the case described, the patient already has a narrow dental profile, which would leave the restored teeth longer than recommended (Ali *et al*., 2015; Bohner *et al*., 2017).

Regarding surgical intervention, this option was presented to the patient, who preferred not to undergo the procedure. The occlusal condition is directly related to the musculoskeletal system, so any change in the occlusal pattern may be directly related to orofacial pain and, in this specific case, the inability to perform the protrusive movement properly, overloads the muscle system and teeth later, causing possible consequences in the dental and periodontal tissues (Okeson, 2013). As a sign of occlusal derangement, posterior teeth present non-carious cervical lesions, together with a history of dental fractures during nocturnal bruxism.

It was decided to keep the molten metal cores, as their removal would bring a high risk of root fracture (Batista, 2019). Although the central and lateral incisors present a risk of fracture due to oblique forces, the installation of fused metal cores was chosen because of the low remaining amount of ferrule and preserved root canal (Pegoraro *et al*., 2012; Minguini *et al*., 2014; Mendonça *et al*. . 2017; Soares *et al.,* 2018;). The use of composite resin for masking the metallic core provides greater adhesion between the preparation and the ceramic crowns promoted by the silane. This agent provides adhesion between a material composed of reacting molecules on inorganic surfaces (ceramic) and organic surfaces (composite resin), thus managing to form a covalent bond between the two surfaces (Matinlinna & Vallittu, 2007). A study by Volpato *et al*. (2009), showed that several light sources influence the translucency of the ceramic, and if there is a darkened dental substrate or the use of a metallic core, it is necessary to mask the darkened region, minimizing undesirable aesthetic effects in the final restoration.

The success of the restoration depends on several factors, including a reliable molding of the dental preparation. Correct gingival retraction for correct reproduction of the preparation performed is essential. The two techniques used in this case are widespread in clinical daily life, which uses the use of individual unitary trays known as individual caps and the use of retractor wires in association with hemostatic agents (Zavanelli *et al*., 2016).

The use of retractor wires has the advantages of controlling sulcular moisture at the time of molding and the speed of the procedure (Fazekas et al., 2002; Zavanelli *et al*., 2016). However, even though the technique was first chosen among specialists in dental prostheses, there is a great deal of discussion in the literature regarding the trauma used to the sulcular epithelium with the use of astringent solutions that the technique employs. The use of headgear has the main advantage of not using a chemical agent, using only the mechanical method for removal, thus reducing the risk of trauma to the periodontium. In addition, it is easy to handle and economical, as it requires little material to fill the cap, but has the disadvantage of being unable to control the humidity of the gingival sulcus (Guedes & Machado, 2007; Pegoraro et al., 2013). The patient's gingival profile, as it is thin, has priority indication for molding with a cap, however, due to the daily practice of the clinic, the first molding option performed in the clinical case was the molding technique with retractors. Subsequently, it was necessary to carry out two impressions with caps, completing a reliable copy of all subgingival preparations (Mendes & Pagani, 2001). Thus, using the two main molding techniques for fixed prostheses, this, when well indicated in coherent and well executed situations, work very well.

There are different cementing agents available for different clinical indications. There are zinc phosphate cements, glass ionomer cements and resin cements. Among the resin cements, to facilitate and improve clinical daily life, three more options were developed (Pegoraro *et al*., 2013). First, conventional resin cement was developed, which in its process required acid etching and application of an adhesive system. To simplify the process, conventional resin cement can be combined with the application of a self-etching adhesive. In 2002, the self-adhesive cement was then developed, which allows excluding various steps, such as acid conditioning, washing and drying, and application of the adhesive system, thus simplifying clinical management and leading to less room for errors in the cementing process (Radovic *et al*., 2008). Still, the surface treatment of the prosthetic piece is necessary.

Self-adhesive cement has adequate mechanical properties, in addition to dimensional stability and micromechanical adhesion (Gerth *et al*., 2006). With the advantage of tolerance to moisture and not having postoperative sensitivity (Mazioli *et al*., 2017). Lithium disilicate-based ceramics have a reasonable volume of vitreous matrix, which is acid-sensitive, and which after silanization enables greater chemical adhesion (Mair & Padipatvuthikul, 2010). Mazioli *et al*. (2017) carried out a study, which analyzed the bond strength of different cements, conventional resin cement and self-adhesive resin cement

in ceramics based on lithium disilicate. After microshear tests and analysis under an optical microscope, it was observed that the conventional resin cement showed higher strength values, being the most suitable option for cementing ceramics based on lithium disilicate. The simplicity in the handling and clinical use of the self-adhesive cement is presented as a major advantage, therefore, it brings a lower incidence of technical errors in surface preparation. Thus, to obtain a good result, it is necessary to follow the faithful preparation of the prosthetic piece and the correct indication of the cement.

4. CONCLUSION

The present clinical case report allows us to conclude that, in order to be successful in dental rehabilitation, it is important to re-establish esthetics with function. All steps for the elaboration of the prostheses have a direct relationship between them, which, when neglected, put the final result of the treatment at risk. The realization of indirect restorations that respect the biological distance and return the desired esthetics to the patient are essential for the success of this treatment.

The use of ceramics reinforced by lithium disilicate proves its versatility, favorable aesthetics, with good optical properties and its biocompatibility, mimicking natural teeth. The application of composite resin for masking the metallic post adds to the cementation better substrate, reinforcing its adhesion to the ceramic restoration and to the dental preparation.

At the beginning of this case, the issue of performing orthognathic surgery to re-establish the present skeletal open bite was mentioned, which would lead to occlusal stability. The surgical procedure would provide relief from the overload present in the posterior teeth, would re-establish the excursive movements and would alleviate the patient's orofacial pain problems. However, it is important to take into account the patient's age, the entire process that she would go through before and after orthognathic surgery, morbidity and expenses with the surgical procedure, and she should be aware of the limitations of the case.

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CONCLUSÕES

4. CONCLUSÕES

Dentro das limitações metodológicas impostas pelo delineamento experimental dos estudos *in vitro* e um caso clínico, pode-se concluir que:

- **Capítulo 1:** Não foram observadas diferenças morfológicas (MEV, DRX e porosidade) e mecânicas significantes (flexão biaxial e microcisalhamento) entre duas marcas comerciais distintas de cerâmicas reforçadas por dissilicato de lítio para CAD/CAM (IPS e.max CAD e Rosetta SM CAD).

- **Capítulo 2:** A técnica proposta para avaliação morfológica e quantitativa dos cristais cerâmicos empregando imagens de MEV associadas a programa de processamento de imagens se mostrou efetiva. Os materiais IPS e.max CAD, Rosetta SM CAD e T-lithium CAD apresentaram resultados similares e condizentes com a literatura empregando esta técnica. O material IRIS CAD apresentou variações acentuadas na quantidade, tamanho e forma dos cristais.

- **Capítulo 3:** Foram observadas diferenças morfológicas (MEV) e mecânicas (flexão biaxial) entre as demais cerâmicas reforçadas por dissilicato de lítio para CAD/CAM testadas em relação ao material IRIS CAD. Entretanto, suas estruturas cristalinas (DRX) e resistência de união (microcisalhamento) apresentaram similaridade.

- **Capítulo 4:** Neste relato de caso, foi demonstrado que a mordida aberta anterior esquelética é um desafio para reabilitações orais. Porém, deve-se levar em consideração todo o processo caso se optasse pela cirurgia ortognática, estando a paciente ciente das limitações do caso. Além disso, neste caso comprovou-se a versatilidade de cerâmicas reforçadas por dissilicato de lítio para restaurações estéticas anteriores.

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