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Luís Felipe Guilardi

ASPECTOS MECÂNICOS E TOPOGRÁFICOS RELACIONADOS ÀS CARACTERÍSTICAS DE SUPERFÍCIE E ADESIVAS DE DIFERENTES CERÂMICAS ODONTOLÓGICAS

Santa Maria, RS
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Tese apresentada ao Curso de Doutorado do Programa de Pós-Graduação em Ciências Odontológicas, Área de Concentração em Odontologia, ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do grau de **Doutor em Ciências Odontológicas**.

Orientadora: Profa. Dra. Marília Pivetta Rippe
Coorientador: Prof. Dr. Luiz Felipe Valandro

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Santa Maria, RS
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DEDICO ESTE TRABALHO:

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“Podemos julgar nosso progresso pela coragem de nossas perguntas e pela profundidade de nossas respostas, nossa disposição de abraçar o que é verdadeiro e não o que é bom”.

Carl Sagan

RESUMO

ASPECTOS MECÂNICOS E TOPOGRÁFICOS RELACIONADOS ÀS CARACTERÍSTICAS DE SUPERFÍCIE E ADESIVAS DE DIFERENTES CERÂMICAS ODONTOLÓGICAS

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Esta tese compõe quatro artigos científicos que avaliam as características mecânicas de cerâmicas odontológicas relacionadas às suas características microestruturais e adesivas após cimentadas e envelhecidas. O primeiro estudo mostra o comportamento mecânico à fadiga e ao impacto de cinco cerâmicas (feldspática, dissilicato de lítio, silicato de lítio, rede cerâmica infiltrada com polímero, e zircônia) quando polidas e quando submetidas a simulação da usinagem CAD/CAM (Computer-Aided Design/ Computer-Aided Manufacturing). Espécimes de cada material foram confeccionados para testes de fadiga (12×12×1,2mm^3;n=15;Stepwise) e impacto (15×10×2mm^3;n=15), e foram polidos (lixa d'água granulação #2500) ou desgastados (lixa d'água #60) simulando o CAD/CAM. Análises de rugosidade, topografia, fractografia, elementos finitos e correlação de Pearson (rugosidade vs. dados de impacto e fadiga) foram realizadas. Maior rugosidade foi em geral correlacionada com menor resistência do material à fadiga e ao impacto. A simulação da fresagem em CAD/CAM aumentou significativamente a rugosidade e reduziu a performance em fadiga e resistência ao impacto dos materiais. O segundo artigo investigou efeito do condicionamento de uma cerâmica feldspática com ácido fluorídrico (HF: HF10%/1min) e da termociclagem (Tc:5-55°C/12.000 ciclos) na sua carga de falha em fadiga quando cimentada ao análogo de dentina (G10) usando dois cimentos diferentes. Espécimes cerâmicos (Mark II; Ø=10mm; espessura=1mm) e G10 (Ø=10mm; espessura=2,5mm) foram confeccionados e divididos segundo 3 fatores: HF (sem; com), cimento (autoadesivo-RelyXU200; convencional-MultilinkAutomix), Tc (sem; com). Foram executados o teste de fadiga Staircase (n=20; 250.000 ciclos;20Hz), análise de ângulo de contato, topografia e fractografia. O HF mais a aplicação do silano proporcionaram maior molhabilidade à cerâmica, porém sem melhorar os dados de fadiga. Quando a cerâmica foi condicionada com HF houve redução mecânica significativa após termociclagem, independentemente do cimento. Os últimos dois artigos buscaram investigar o efeito de diferentes protocolos de cimentação na resistência à fadiga de uma zircônia monolítica cimentada sobre G10 com e sem envelhecimento. Os espécimes de zircônia (Ø=10mm; espessura=0,7mm) e G10 (Ø=10mm; espessura=2,8mm) apresentaram dimensões iguais em ambos trabalhos. No primeiro, a zircônia foi jateada (óxido de alumínio 45µm) e cimentada no G10 com 4 diferentes cimentos (ionomérico, resinoso autoadesivo, resinoso convencional com monômero-fosfato [MDP] no primer, e resinoso convencional com MDP no cimento) e o teste de fadiga (n=20;Staircase) foi executado. O cimento ionomérico mostrou os menores valores de resistência à fadiga, e o convencional com MDP no primer os maiores. A Tc não reduziu os valores de carga em fadiga. No último artigo, avaliou-se a resistência à fadiga da zircônia monolítica sob 3 fatores: tratamento da zircônia (jateamento com óxido de alumínio-OA ou tratamento triboquímico-TT); cimento resinoso convencional (sem e com MDP); envelhecimento (sem e com: Tc+60 dias em água). Realizou-se a cimentação (zircônia/G10), envelhecimento e o teste de fadiga (n=20;Staircase). A carga de falha dos espécimes jateados com OA e cimentados com cimento contendo MDP reduziu significativamente após termociclagem. Nos trabalhos envolvendo cimentação, a fractografia detectou que as falhas (trinca radial) tiveram origem na face de cimentação da cerâmica. Conclui-se que a rugosidade, tratamento de superfície, sistema de cimentação e o envelhecimento influenciam o comportamento mecânico de cerâmicas odontológicas.

Palavras-chave: 10-metacriloiloxi-decil-dihidrogenofosfato. Adesão. CAD/CAM. Características de superfície. Carregamento Cíclico. Cerâmicas Dentárias. Distribuição de Tensões. Envelhecimento. Limite de fadiga. Resistência ao impacto. Restaurações monolíticas. Tratamento da cerâmica.

ABSTRACT

MECHANICAL AND TOPOGRAPHICAL ASPECTS RELATED TO SURFACE AND ADHESIVE CHARACTERISTICS OF DIFFERENT DENTAL CERAMICS

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ADVISER: Marília Pivetta Rippe
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The present thesis composes four scientific articles that evaluate the mechanical characteristics of dental ceramics related to their microstructural and adhesive characteristics after bonding and aging. The first study shows the mechanical behavior to fatigue and impact test of five ceramics (feldspathic, lithium disilicate, lithium silicate, polymer infiltrated ceramic network, and zirconia) when polished and when submitted to CAD/CAM (Computer-Aided Design/Computer-Aided Manufacturing) milling simulation (as-milled). Specimens of each material were prepared for fatigue ($12\times12\times1.2mm^3$, n=15, Stepwise) and impact ($15\times10\times2mm^3$; n=15) tests, and were polished (sandpaper #2500-grit) or ground (sandpaper #60-grit) simulating CAD/CAM. Analysis of roughness, topography, fractography, finite elements and Pearson correlation (roughness vs. impact and fatigue data) were performed. Greater roughness was generally correlated with material lower resistance to fatigue and impact. Simulation of CAD/CAM milling significantly increased the materials roughness and reduced fatigue performance and impact resistance. The second article investigated the effect of etching a feldspathic ceramic with hydrofluoric acid (HF: HF10%/1min) and thermocycling (Tc:5-55°C/12,000 cycles) on its fatigue failure load when cemented to the dentin-like material (G10) using two different cements. Ceramic specimens (Ø=10mm; thickness=1mm) and G10 (Ø=10mm; thickness=2.5mm) were prepared and divided according to three factors: HF (without; with), cement (self-adhesive- RelyX U200, conventional- Multilink Automix), Tc (without; with). The Staircase fatigue test (n=20; 250,000 cycles;20Hz), the contact angle, topography and fractography analyses were performed. The HF plus the silane application provided greater wettability to the ceramic, but without improving the fatigue data. When the ceramic was conditioned with HF there was significant mechanical reduction after thermocycling, regardless of the cement. The last two articles aimed to investigate the effect of different cementation protocols on the fatigue strength of a monolithic zirconia cemented on G10 with and without aging. The zirconia (Ø=10mm; thickness=0.7mm) and G10 (Ø=10mm; thickness=2.8mm) specimens presented equal dimensions in both works. In the first, the zirconia was sandblasted (45µm aluminum oxide) and cemented in G10 with four different cements [ionomeric, self-adhesive resin cement, conventional resin cement with monomer-phosphate (MDP) in the primer, and conventional resin cement with MDP in the cement] and the fatigue test (n=20; Staircase) was performed. The ionomeric cement resulted in a lower fatigue resistance, and the conventional resin cement with MDP in the primer performed better. Tc did not reduce fatigue load values. In the last article, the fatigue resistance of monolithic zirconia was evaluated under three factors: zirconia treatment (AO-sandblasting with aluminum oxide or TT-tribochemical treatment); conventional resin cement (without or with MDP); aging (without or with: Tc + 60 days in water). Cementation (zirconia/G10), aging and the fatigue test (n=20; Staircase) were performed. The fatigue performance of specimens sandblasted with AO and cemented with MDP-containing cement was significantly reduced after aging. In the works involving cementation, the fractographic analyses detected that all failures (as radial cracks) had origin in the ceramics' intaglio surface. It is concluded that the roughness, surface treatment, cementation system and aging can influence the mechanical behavior of dental ceramics.

Keywords: 10-methacryloyloxy-decyldihydrogen-phosphate. Adhesion. Aging. Cad/Cam. Ceramic treatment. Cyclic loading. Dental ceramics. Fatigue limit. Monolithic restorations. Resistance to impact. Stress distribution. Surface characteristics.

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SUMÁRIO

1. INTRODUÇÃO GERAL E REVISÃO DE LITERATURA

Atualmente muitas opções de materiais dentários estéticos totalmente cerâmicos para a fabricação de restaurações estão disponíveis para os clínicos. Estes materiais cerâmicos são comumente disponíveis na forma de pó (ex. cerâmicas vítreas) e na forma pré-processada, como em blocos cuboides ou discos. Estas diferentes formas de apresentação das cerâmicas odontológicas advem da inserção e desenvolvimento da tecnologia em odontologia, principalmente através dos sistemas de usinagem em CAD/CAM (Computer-Aided Design – CAD / Computer-Aided Manufacturing - CAM) (BELLI et al., 2017). A maioria dos materiais é totalmente produzida em um ambiente industrial ideal, garantindo padrões de qualidade dificilmente alcançados em condições clínico/laboratoriais (LI; CHOW; MATINLINNA, 2014). Ao contrário das técnicas tradicionais aditivas, este processo parte de uma peça inteira e produz a restauração através de um sistema subtrativo (MÖRMANN, 2006). Os métodos tradicionais de fabricação das restaurações cerâmicas têm sido descritos como demorados, tecnicamente sensíveis e imprevisíveis devido às suas muitas variáveis, sendo o CAD/CAM uma boa alternativa tanto para os dentistas quanto para os laboratórios para um melhor fluxo de trabalho na confecção das restaurações (LI; CHOW; MATINLINNA, 2014).

Este conceito permitiu a aplicação inédita em odontologia de cerâmicas policristalinas de alta resistência densamente sinterizadas, como o dióxido de zircônio (ZrO_2) e o óxido de alumínio (Al_2O_3), cujos pós são isostaticamente prensados e pré-sinterizados antes da usinagem (‘*green bodies*’). Alguns materiais vítreos monolíticos também são oferecidos em um estágio meta-sinterizado para facilitar a usinagem, e posteriormente a peça é submetida a uma queima final de cristalização, como para as cerâmicas vítreas reforçadas por dissilicato de lítio e as reforçadas por silicato de lítio (BELLI et al., 2017).

O fluxo digital de trabalho e seus componentes de fabricação fornecem alta precisão e acurácia, previsibilidade, eficiência, substancial redução do tempo de trabalho e uma ampla gama de materiais restauradores e protéticos com propriedades físicas, ópticas e biológicas que frequentemente excedem as das fabricadas convencionalmente (MIYAZAKI et al., 2009; BLATZ; CANEJO, 2019). Os sistemas CAD/CAM inicialmente eram limitados a fabricar inlays, onlays e coroas unitárias. Agora, com a infinidade de tecnologias CAD/CAM, sistemas, fresadoras e outras ferramentas disponíveis, não há praticamente nenhum limite para o tipo de restauração dental que pode ser fabricada, desde inlays, onlays, coroas, facetas, laminados, pilares de implantes e restaurações para próteses dentárias fixas e removíveis para pacientes parcialmente e completamente desdentados (BLATZ; CANEJO, 2019).

As cerâmicas odontológicas são classificadas de acordo com sua fase cristalina e técnica de processamento, sendo atualizadas à medida que novos materiais restauradores surgem na busca por combinar alta resistência, tenacidade à fratura, estética ideal e desempenho clínico de longo prazo (ZHANG; KELLY, 2017). Uma característica em comum entre as cerâmicas dentárias, mesmo para aquelas com alta resistência (ex. zircônia tetragonal policristalina), é a capacidade relativamente baixa de absorver energia antes da ocorrência da fratura, ou seja, elas são friáveis/pouco tenazes (DENRY; HOLLOWAY, 2010). A tenacidade à fratura é uma propriedade capaz de modificar esse comportamento, e isso tem levado à busca de novos materiais, como por exemplo os materiais híbridos (e.g., resinas nanocerâmicas - LAVA Ultimate; cerâmica infiltrada com polímero - VITA Enamic) que possuem uma matriz ou estrutura de rede polimérica combinando componentes orgânicos e inorgânicos (HE; PURTON; SWAIN, 2011; FACENDA; BORBA; CORAZZA, 2018).

Do ponto de vista clínico, dois parâmetros são imprescindíveis na hora de selecionar um sistema restaurador, estética e resistência mecânica. Estética especialmente para a região anterior e resistência mecânica, para que as restaurações sejam capazes de suportar cargas oclusais funcionais e parafuncionais, especialmente no setor posterior (SAKAGUCHI; POWERS, 2012). Ambos os parâmetros estão intimamente correlacionados, e alterações físicas do material buscando-se melhor estética ou maior resistência, invariavelmente terão consequência direta nas propriedades do material. A fase cristalina da cerâmica, sua porosidade (DELLA BONA; NOGUEIRA; PECHO, 2014) e sua rugosidade (AWAD et al., 2015) são capazes de influenciar as suas propriedades ópticas. O aumento de conteúdo de fase cristalina de uma cerâmica vítrea na busca por maior resistência, por exemplo, implica em uma perda nas suas propriedades ópticas, tornando-a mais opaca (SAKAGUCHI; POWERS, 2012), e quanto menor o teor de conteúdo vítreo, maior a resistência da cerâmica (RAMOS et al., 2016; WENDLER et al., 2017; NISHIOKA et al., 2018; VENTURINI et al., 2019). Até o momento, uma proporção inversa entre resistência (ex. desempenho mecânico) e propriedades ópticas (ex. aparência estética) ainda é uma equação predominante.

Segundo Kruzic et al. (2018), a microestrutura das cerâmicas influencia significativamente na resistência à fratura e no comportamento em fadiga através dos seus diferentes mecanismos de tenacificação, tais como *crack bridging* (ex. compósitos e cerâmicas híbridas), formação de uma zona de deformação plástica ao redor da ponta da trinca (ex. cerâmicas híbridas), deflexão da trinca (ex. cerâmicas vítreas reforçadas por partículas cristalinas), e tenacificação por transformação de fase (ex. zircônia tetragonal policristalina parcialmente estabilizada).

1.1 TIPOS DE CERÂMICAS:

Assim, os materiais cerâmicos atuais usados em odontologia podem ser classificados em 3 grupos de acordo com a sua composição e microestrutura (GRACIS et al., 2015), os quais estão dispostos abaixo:

1.1.1 Cerâmica híbrida:

A cerâmica híbrida contém uma infraestrutura cerâmica (86 wt%) infiltrada por polímero (14 wt%), sendo um exemplo de marca comercial a VITA ENAMIC (VITA Zahnfabrik). Este material é destinado à confecção e aplicação em consultório (*chairside*), já que não necessita de queima para sua cristalização. Quando comparado às cerâmicas a base de sílica, possui uma maior capacidade de suportar carga, melhor módulo de elasticidade (E = ±30 GPa; aproxima-se ao da dentina humana $E_{dentina}$ = 18 GPa), e propriedades de usinagem mais favoráveis (AWADA; NATHANSON, 2015). Além disso, é um material de processamento rápido, custo reduzido e preciso no ajuste final da restauração (BOTTINO et al., 2015).

Em termos de adesão, o tratamento de superfície das cerâmicas híbridas requer condicionamento com ácido fluorídrico (HF), seguido pela aplicação de primers à base de silano, como para as vitrocerâmicas (CAMPOS et al., 2016).

1.1.2 Cerâmicas a base de sílica:

São definidas como materiais cerâmicos inorgânicos, que contêm uma fase vítrea reforçada por cristais, tipicamente leucita ou dissilicato de lítio. A matriz vítrea oferece alta translucidez, ótima estética e aparência natural, contudo, devido a sua friabilidade e baixa tenacidade à fratura, elas precisam ser adesivamente cimentadas (BEIER; DUMFAHRT, 2014). Uma boa resistência de união a longo-prazo para este material pode ser estabelecida através do condicionamento da cerâmica com ácido fluorídrico e posterior aplicação de um agente de ligação silano (STRASSER et al., 2018). O tempo e concentração do ácido irá depender do conteúdo cristalino da cerâmica, sendo que, de acordo com os fabricantes, para a cerâmica feldspática convencional indica-se uma concentração de 10% durante 1 a 2 min, para a cerâmica feldspática reforçada por leucita 10% durante 1 min, e o dissilicato de lítio deve ser condicionado com uma concentração e tempo reduzidos, 5% por apenas 20 s, respectivamente. Após a limpeza dos precipitados resultantes do condicionamento ácido, o agente de ligação silano é aplicado e a restauração está pronta para ser cimentada utilizando-se um cimento resinoso (STRASSER et al., 2018).

1.1.2.1 Cerâmica feldspática tradicional:

Estas cerâmicas são descritas como os materiais mais translúcidos e estéticos e são tipicamente utilizadas como porcelanas de revestimento para estruturas metálicas e cerâmicas ou como facetas laminadas, inlays e onlays. Também têm sido utilizadas na confecção de coroas monolíticas através do uso de blocos para o sistema CAD/CAM. Alguns exemplos mais populares são a VITABLOCS Mark II (VITA Zahnfabrik), VITABLOCS RealLife Ceramic Blocks (VITA), e VITABLOC TriLux Forte (VITA). Alguns destes materiais apresentam-se na forma de blocos multicamada policromáticos, os quais possibilitam maior capacidade de mimetização do dente natural.

Apesar das baixas propriedades físicas das cerâmicas feldspáticas, vários estudos clínicos apresentam excelentes taxas de sobrevivência e sucesso para este material. O estudo de Otto e Mörmann (OTTO; MÖRMANN, 2015) relatou uma taxa de sobrevivência de 95% para coroas em molares e pré-molares feitas de cerâmica feldspática (VITABLOCS Mark II) para CAD/CAM após um acompanhamento de 12 anos. Em uma série de casos com inlays e onlays (VITABLOCS Mark I), mostrou-se uma probabilidade de sobrevivência de 88,7% após 17 anos de acompanhamento (OTTO; SCHNEIDER, 2008) e de 87,5% após 27 anos (OTTO, 2017), sendo que 62-65% das falhas ocorreram devido à fratura da cerâmica (OTTO; SCHNEIDER, 2008; OTTO, 2017). O estudo de Reiss (REISS, 2006) mostrou alta taxa de sucesso (84,4%) e sobrevivência (89%) para inlays com aproximadamente 18 anos de acompanhamento. O autor relatou que os pré-molares alcançaram um resultado melhor que os molares, que os dentes vitais provaram ser melhores que os dentes não vitais, e o uso de adesivos dentinários aumentou a probabilidade de sucesso. As causas mais frequentes de falha foram a fratura da cerâmica e/ou dos dentes (REISS, 2006).

1.1.2.2 Cerâmicas vítreas reforçadas por leucita:

Este material possui uma alta translucidez e maior resistência em comparação com cerâmicas feldspáticas tradicionais. É indicado especialmente para coroas anteriores e inlays/onlays posteriores. De acodo com Nejatidanesh et al. (2015) as restaurações parciais posteriores confeccionadas em CAD/CAM mostraram uma taxa de sobrevida em 5 anos de 96% para a cerâmica fedspática CEREC Blocs e 94,6% para a cerâmica vítrea reforçada por leucita Empress CAD Blocs. Após 7 anos de acompanhamento, Guess e colaboradores (GUESS et al., 2013) relataram taxas de sobrevivência de 100% para restaurações de cobertura parcial feitas de dissilicato de lítio prensado (IPS e.max-Press, Ivoclar Vivadent) e de 97% para restaurações de cobertura parcial CAD/CAM feitas de cerâmica vítrea reforçada por leucita (ProCAD, Ivoclar Vivadent). Assim, as cerâmicas de vidro reforçadas com leucita têm sido amplamente substituídas por cerâmicas de silicato de lítio, as quais possuem boa translucidez e melhores propriedades físicas.

1.1.2.3 Cerâmicas a base de silicato de lítio:

Esta cerâmica tem se tornado muito popular para muitas indicações, especialmente para coroas monolíticas, inlays e onlays. Este material é o mais resistente dentre as cerâmicas a base de sílica em odontologia, e apresenta em média 407 MPa de resistência flexural (CONEJO et al., 2017). As cerâmicas de silicato de lítio possuem uma fase cristalina que consiste em dissilicato de lítio e ortofosfato de lítio, o que aumenta a resistência à fratura sem influenciar negativamente a sua translucidez. Estes materiais devem ser cristalizados em um forno de sinterização após a fresagem. Eles também podem ser pigmentados e glazeados. Excelentes taxas de sucesso estão bem documentadas na literatura recente (RAUCH et al., 2018; MALAMENT et al., 2019), especialmente para restaurações unitárias (CONEJO et al., 2017). Além disso, Yildiz et al. (2013) reportaram que tanto a confecção de onlays de dissilicato de lítio através do sistema analógico via prensagem quanto pela fresagem em CAD/CAM têm valores elevados e clinicamente aceitáveis de resistência à fratura. Com relação a coroas a base de dissilicato de lítio prensado, Malament et al. (2019) mostraram uma excelente longevidade quando estas foram cimentadas em áreas de incisivos, pré-molares e molares, apresentando uma taxa de sobrevivência cumulativa estimada em 96,5% para coroas monolíticas após 10,4 anos e de 100% para coroas e.max bicamada após 7,9 anos.

Considerando ainda este grupo de cerâmicas, vale ressaltar que recentemente um novo material foi lançado no mercado, o silicato de lítio reforçado por zircônia (VITA Suprinity PC - VITA Zahnfabrik; e Celtra Duo - Dentsply Sirona), o qual combina o desempenho estético da vitrocerâmica com a resistência mecânica proporcionada pelos cristais de metassilicato e zircônia (MONTEIRO et al., 2018a). São indicados para coroas posteriores, anteriores e implantossuportadas, bem como para restaurações inlay e onlay. Assim, também é importante ressaltar que estudos *in vitro* observaram desempenho similar deste tipo de cerâmica, às já conhecidas cerâmicas à base de dissilicato de lítio (WENDLER et al., 2018).

1.1.3 Cerâmicas a base de óxidos:

As cerâmicas policristalinas à base de óxido metálico para usinagem em CAD/CAM, como por exemplo a zircônia, são caracterizadas por possuírem excelentes propriedades mecânicas, as quais são significativamente maiores que as das cerâmicas à base de sílica. Em geral as restaurações são fresadas a partir de blocos pré-sinterizados, possibilitando um material menos duro e mais fácil de ser fresado, que são 20 a 25% maiores que a restauração final a fim de compensar a contração que ocorre no material durante o ciclo final de sinterização.

Os valores de resistência flexural da zircônia tetragonal policristalina estabilizada por ítria convencional varia entre 1000 e 1500 MPa (CONEJO et al., 2017). As primeiras gerações de zircônia possuiam translucidez muito limitada e, portanto, eram indicadas como copings e estruturas que necessitavam de uma cobertura cerâmica mais estética. Mesmo proporcionando taxas de sucesso ao nível das coroas e próteses fixas metalo-cerâmicas, ainda possuíam um problema recorrente de lascamento ou delaminação da cerâmica de cobertura (SAILER et al., 2015; PJETURSSON et al., 2015, 2017) e, portanto, as zircônias monolíticas passaram a ser uma tendência para a solução deste problema. Sendo assim, as gerações mais recentes de zircônia possuem uma translucidez significativamente maior (STAWARCZYK et al., 2017; SOUZA et al., 2018). A segunda geração da zircônia ainda apresenta uma composição semelhante à de primeira geração, sendo parcialmente estabilizada na fase tetragonal (3Y-TZP), porém tendo uma redução no número e tamanho dos grãos de óxido de alumínio (Al_2O_3), sendo estes realocados na estrutura da zircônia, permitindo maior passagem de luz e consequentemente uma maior translucidez (STAWARCZYK et al., 2017).

Como a zircônia de segunda geração ainda possui uma translucidez muito inferior à das vitrocerâmicas, surgiu o desejo de uma zircônia mais translúcida. Assim, o próximo estágio no desenvolvimento da zircônia monolítica veio com uma mudança para incluir alguma fase transparente no produto final para reduzir a sua opacidade. Isto foi alcançado usando-se um maior conteúdo de óxido de ítrio para produzir zircônias parcialmente estabilizadas, 4 mol% (4Y-PSZ [zircônia parcialmente estabilizada por ítria]) ou 5 mol% (5Y-PSZ), resultando em uma quantidade maior de partículas de fase cúbica (*c*) (ZHANG; LAWN, 2018). O maior conteúdo de fase cúbica resulta em um aumento considerável na translucidez do material (STAWARCZYK et al., 2017), porém em uma redução muito significativa na sua resistência flexural (PEREIRA et al., 2018) e na sua tenacidade à fratura (ZHANG, 2016). As zircônias mais translúcidas (e.g., 5Y-PSZ) têm sido amplamente indicadas para uso como coroas e pontes parciais fixas anteriores e posteriores. No entanto, um estudo recente revelou uma taxa de falha de 2,06% para as restaurações na região anterior e de 0,99% para as restaurações na região posterior em até 5 anos de acompanhamento (SULAIMAN et al., 2016). Dados clínicos de longo prazo sobre reabilitações com zircônia monolítica ainda são escassos (SPITZNAGEL; BOLDT; GIERTHMUEHLEN, 2018). A aplicação destes materiais em restaurações minimamente invasivas, como facetas, inlays e onlays, ainda precisa ser avaliada. Consequentemente, é necessário ter cuidado na indicação e inserção de tais cerâmicas (CHRISTENSEN, 2016; SOUZA et al., 2018).

Alguns exemplos de zircônias para CAD/CAM altamente translúcidas incluem a Katana Zirconia Block (Kuraray Noritake), CEREC Zirconia (Dentsply Sirona), VITA YZ (VITA Zahnfabrik), Lava Zirconia Block (3M ESPE), Zenostar T (Ivoclar Vivadent, Wieland Dental) e a IPS e.max ZirCAD (Ivoclar Vivadent).

1.2 PROCESSAMENTO EM CAD/CAM:

Embora as restaurações cerâmicas produzidas por usinagem de blocos cerâmicos possam otimizar e melhorar a confiabilidade estrutural do material em si, o efeito do processo de usinagem na estabilidade a longo prazo dessas restaurações deve ser levado em consideração. Isto porque os sistemas CAD/CAM utilizam processos abrasivos de usinagem que apresentam alto potencial de geração de danos na superfície e subsuperfície do material, podendo reduzir a integridade da restauração final (MARSHAL et al., 1983; FRAGA et al., 2017; ROMANYK et al., 2019), principalmente quando localizados em zonas como na região gengival dos conectores de PPFs, nas margens e ângulos internos da restauração (CANNETO et al., 2016). Quando estes danos gerados são mais severos que os defeitos pré-existentes no material, eles assumem um papel importante no controle da resistência (MARSHAL et al., 1983). De acordo com Romanyk et al. (2019) e Fraga et al. (2017), os espécimes usinados em CAD/CAM mostram evidência de dano inserido pela usinagem na forma de trincas laterais e radiais, lascas, danos na subsuperfície e tensões residuais. As trincas geralmente estão localizadas na periferia ou exatamente na origem da falha do material (ROMANYK et al., 2019). Estes danos introduzidos durante a usinagem subtrativa limitam a resistência do material e não são eliminados durante a cristalização (silicato e dissilicato de lítio), sinterização (zircônia) ou *annealing* (recozimento) (silicato de lítio - Celtra Duo) do material. Ademais, a superfície interna permanece classicamente intocada (como fresada), enquanto que a superfície externa de uma restauração pode ser finalizada, polida e/ou glazeada (FRAGA et al., 2015).

Fraga et al. (2017) mostraram que além de produzir uma rugosidade significativamente maior, a usinagem reduziu a resistência à fadiga da zircônia (Vita In-Ceram 2000 YZ for inLab;Vita Zahnfabrik) em 40%, do dissilicato de lítio (IPS e.max CAD; Ivoclar Vivadent) em 33% e da cerâmica leucítica (IPS Empress CAD; Ivoclar Vivadent) em 29% quando comparados aos seus grupos que receberam polimento após a usinagem. Devemos considerar que este estudo não avaliou os espécimes após a sua cimentação, o que poderia ter limitado o efeito da rugosidade, criada pela usinagem, na resistência dos materiais, como observado por de Kok e colaboradores (DE KOK et al. 2017). Considerando o exposto acima e sabendo que restaurações totalmente cerâmicas podem falhar devido à presença de trincas e defeitos em sua superfície de cimentação (KELLY et al., 1990; THOMPSON et al., 1994; THOMPSON; REKOW, 2004), a literatura têm apontado este efeito da usinagem com certa preocupação e cautela, uma vez que superfícies rugosas podem prejudicar as propriedades mecânicas da cerâmica por acúmulo de danos e redução da vida útil quando sob uso clínico (carga cíclica intermitente) (KELLY et al., 2017; DE KOK et al. 2017; VENTURINI et al., 2018).

A capacidade de um material em resistir à propagação de trincas, considerando que possui defeitos internos e externos, é um fator importante que afeta o desempenho da restauração cerâmica. De fato, a mecânica de fratura de materiais cerâmicos no desempenho clínico foi definida com base na ‘teoria do elo mais fraco’ de Griffth (1921) e pelo mecanismo de crescimento lento de trincas (crescimento subcrítico), onde qualquer defeito interno existente no material pode crescer até um tamanho crítico quando submetido a estímulos mecânicos e atuar como um iniciador de trinca, levando à fratura prematura da cerâmica sob cargas muito menores do que o esperado (GONZAGA et al., 2011). Ainda existe uma lacuna na compreensão de como a rugosidade pode interferir no desempenho clínico de diferentes materiais cerâmicos durante a aplicação de cargas funcionais, principalmente quando estes são cimentados utilizando-se diferentes protocolos de cimentação.

Além da presença de cargas cíclicas intermitentes durante a mastigação no ambiente bucal, sobrecargas podem ocorrer e serem imediatamente transferidas para as estruturas dentárias. O hábito parafuncional involuntário, a mastigação de alimentos duros e o impacto sofrido em acidentes podem gerar tensão excessiva e extrema às estruturas dentárias, muitas vezes causando trincas e/ou fraturas do elemento dental/restauração/implante (SILVA et al., 2011; ANDERSSON, 2011; 2013; ZALECKIENE et al., 2014). Nesses cenários, o mecanismo de fratura pode ser completamente diferente do considerado na literatura por meio dos tradicionais testes estáticos e de fadiga (BORBA et al., 2011; RODRIGUES et al., 2018; NISHIOKA et al., 2018). Testes de impacto são muito raramente relatados na literatura odontológica e estudos que correlacionam a influência de padrões superficiais no impacto e resistência à fadiga de materiais cerâmicos dentários com características microestruturais distintas, são escassos.

Desta forma torna-se fundamental investigar o efeito da rugosidade resultante da usinagem em CAD/CAM nas propriedades mecânicas de cerâmicas disponíveis para uso em odontologia. Portanto, o primeiro estudo da presente tese investigou o comportamento mecânico (teste de resistência ao impacto e teste de resistência à fadiga) de diferentes materiais cerâmicos, os quais apresentam diferentes composições, quando os mesmos são polidos ou quando a sua superfície é mantida com os defeitos inseridos durante a simulação da usinagem em CAD/CAM (polido vs. simulação de fresagem em CAD/CAM).

É de fundamental importância considerarmos que a superfície das cerâmicas, posteriormente à sua confecção via CAD/CAM, deve ser submetida a um tratamento superficial previamente à cimentação (ex. jateamento, condicionamento com ácido fluorídrico, aplicação de um primer cerâmico, aplicação de um agente de ligação, etc.) para que se possa otimizar a união micromecânica e química entre a cerâmica e o cimento. Este passo é fundamental na obtenção de uma adesão adequada e estável (ex. menos susceptível à degradação da interface), consequentemente melhorando o desempenho e previsibilidade a longo prazo para tais restaurações (STRASSER et al., 2018).

1.3 PRINCIPAIS TRATAMENTOS DE SUPERFÍCIE DAS CERÂMICAS:

Os métodos de tratamento de superfície cerâmica mais comuns são o condicionamento com ácido fluorídrico ou o jateamento com partículas de óxido de alumínio revestidas ou não com sílica (MALAMENT; SOCRANSKY, 2001; ALBAKRY; GUAZZATO; SWAIN, 2004; GUAZZATO et al., 2004). Embora a aplicação do condicionamento ácido seja bem conhecida por aumentar a força de união através do desenvolvimento de uma superfície uniformemente rugosa, ela irá depender do tipo de cerâmica e poderá exigir uma variação em termos de concentração e tempo de aplicação (STRASSER et al., 2018). Por outro lado, o jateamento é relativamente fácil de utilizar, porém pode gerar perda variável de volume e também levar a danos residuais severos (ZHANG et al., 2004; GUAZZATO et al., 2004).

Tem sido demonstrado que tais tratamentos de superfície (ex. condicionamento ácido, jateamento, desgaste) aumentam as irregularidades na superfície da cerâmica (BORGES et al., 2003; ALBAKRY; GUAZZATO; SWAIN, 2004; ADDISON; FLEMING, 2004; VENTURINI et al., 2015), afetando drasticamente a sua resistência à fratura, podendo levar a uma falha prematura (ZHANG et al., 2008; QUINN; QUINN, 2010). Fatores laboratoriais e clínicos são frequentemente discutidos como sendo “controladores” da resistência do material cerâmico, incluindo as características do material (KRUZIC et al., 2018), processamento em laboratório, tratamento de superfície (ALBAKRY; GUAZZATO; SWAIN, 2004; GUAZZATO et al., 2004), tipo de cimento (MALAMENT; SOCRANSKY, 2001), e características do ambiente bucal. Devido ao fato das falhas clínicas partirem de defeitos presentes na superfície interna/de cimentação da restauração cerâmica (KELLY et al., 1990), o efeito dos métodos de tratamento da superfície (DE KOK et al. 2017; VENTURINI et al., 2018) bem como do tipo e sistema de cimentação utilizados (FLEMING; HOOI; ADDISON 2012) têm recebido muita atenção

(KELLY et al., 2010; ANAMI et al., 2016; CAMPOS et al., 2017; CANNETTO et al., 2016). O condicionamento com ácido fluorídrico (HF) seguido de silanização é o tratamento de superfície mais amplamente aceito para as cerâmicas vítreas (SOUZA et al., 2011). O HF causa uma dissolução seletiva na matriz vítrea da superfície cerâmica, criando microporos retentivos, sulcos e trincas na superfície condicionada, favorecendo a retenção micromecânica (AIDA; HAYAKAWA; MIZUKAWA, 1995; CHEN et al., 2014). Um agente de ligação silano é então aplicado na superfície condicionada para promover uma ligação química entre a fase inorgânica da cerâmica (ex. a sílica) com a fase orgânica do cimento resinoso (ex. polímero) (SÖDERHOLM; SHANG, 1993; ÖZCAN; VOLPATO, 2015; MATINLINNA; LUNG; TSOI, 2018). No entanto, isso tem se tornado cada vez mais discutível, pois alguns estudos têm mostrado não haver necessidade de utilizar o condicionamento com HF para melhorar a adesão entre cimentos resinosos e vitrocerâmicas (PEUMANS et al., 2016; WILLE; LEHMANN; KERN, 2017; EL-DAMANHOURY; GAINTANTZOPOULOU, 2018), sendo também demonstrado que o condicionamento HF pode até levar a um efeito de enfraquecimento da cerâmica (ADDISON; FLEMING, 2004; VENTURINI et al., 2015). Esse impacto deletério é explicado pelo fato de que a corrosão por HF aumenta a população de defeitos na interface de união da cerâmica. Com isso, pode aumentar a suscetibilidade da cerâmica à ocorrência do crescimento lento de defeitos críticos sob as constantes tensões mastigatórias, uma vez que a resistência à fratura da cerâmica é inversamente proporcional ao defeito mais crítico presente na restauração, conforme descrito pela lei de Griffith (1921). Do ponto de vista clínico, o efeito de enfraquecimento acima mencionado é particularmente relevante quando se considera a evidência que mostra que as cerâmicas vítreas falham a partir de defeitos existentes na sua superfície de cimentação (COSTA et al., 2015; MONTEIRO et al., 2018b). Estudos recentes corroboram esses achados, mostrando resultados piores ou iguais de resistência adesiva (PEUMANS et al., 2016), resistência à flexão biaxial (XIAOPING; DONGFENG; SILIKAS, 2014) e para a carga em fadiga (VENTURINI et al., 2018) ao condicionar vitrocerâmica com ácido fluorídrico previamente à cimentação, comprovando que esse assunto ainda requer avaliações e considerações futuras. Além disso, o ácido fluorídrico tem toxicidade potencialmente perigosa conhecida de outras aplicações e também deve ser considerado quando aplicado em odontologia (ÖZCAN; ALLAHBEICKARAGHI; DÜNDAR, 2012).

Já o jateamento é o protocolo mais indicado para a adesão à zircônia. Este tratamento consiste em tornar a cerâmica rugosa através do jateamento com pequenas (até 60 µm) partículas de óxido de alumínio ou de óxido de alumínio revestido por sílica, também chamado de tratamento triboquímico, aplicadas sob baixa pressão (< 2 bar) (STRASSER et al., 2018).

Na sequência busca-se otimizar a adesão química a esta superfície através do uso de primers cerâmicos que contenham monômeros fosfato (MDP - methacryloyloxydecyl dihydrogen phosphate) em sua composição (ex. Clearfil Ceramic Primer, Kuraray Noritake), os quais têm se mostrado mais efetivos na adesão a óxidos metálicos (BLATZ; VONDERHEIDE; CONEJO, 2018). Por fim, cimentos resinosos duais ou de auto-cura devem ser utilizados para garantir uma polimerização adequada. Excelentes resultados de sucesso a longo prazo têm sido relatados utilizando-se este protocolo de cimentação (BLATZ; VONDERHEIDE; CONEJO, 2018).

Apesar dos estudos *in vitro* e clínicos mostrarem a importância do tratamento mecânico da superfície da zircônia a fim de criar microrretensões, este tipo de tratamento promove a inserção de defeitos críticos na cerâmica, os quais podem agir como fatores de concentração de tensão, reduzindo o limiar de tensão necessário para que ocorra a falha (KHAN et al., 2017; MOON et al., 2016; SPAZZIN et al., 2017). Enquanto alguns estudos mostram um efeito deletério do jateamento nas propriedades mecânicas da zircônia (ZHANG et al., 2004; ZHANG et al., 2006), outros estudos mostram não existir este efeito (SCHERRER et al., 2011; ÖZCAN et al., 2013; SOUZA et al., 2013; OBLAK et al., 2014; ANAMI et al., 2016; CAMPOS et al., 2017), principalmente quando os defeitos são adequadamente preenchidos pelo sistema de cimentação, o que reduz drasticamente a população de defeitos e restabelece a resistência inicial do material (SPAZZIN et al., 2016). Além disso, o estudo de Yang et al. (2010) mostrou que reduzir a pressão durante o jateamento ou omití-lo completamente pode ser recomendado em combinação com os novos primers cerâmicos para melhorar a durabilidade da adesão e reduzir a influência negativa nas propriedades mecânicas da zircônia de quando se utiliza o jateamento com alta pressão.

Outro fator que pode afetar a resistência da zircônia após o jateamento é a transformação de fase, de tetragonal para monoclínica, resultando no mecanismo de tenacificação por transformação que leva à concentração de tensões de compressão ao redor das trincas, reduzindo ou limitando a sua propagação e, portanto, aumentando a resistência do material (SCHERRER et al., 2011). De acordo com Scherrer et al. (2011), melhores resultados de performance em fadiga podem ser encontrados quando o jateamento é realizado na superfície da zircônia. Porém, deve-se considerar que isto só é válido para as zircônias de primeira e segunda gerações, uma vez que as zircônias de terceira geração são totalmente estáveis e não sofrem este tipo de transformação de fase (PEREIRA et al., 2018).

1.4 AGENTES CIMENTANTES:

A cimentação é um dos passos finais na sequência de procedimentos clínicos para restaurações indiretas. Existem dois objetivos para o procedimento de cimentação: ajudar a reter a restauração no local e manter a integridade da estrutura dentária remanescente. A retenção é obtida por fricção (ou embricamento micromecânico) e por adesão que consiste na união entre dente preparado, cimento e restauração, ou uma combinação de ambos os mecanismos. Um efetivo selamento interfacial depende da capacidade do cimento de preencher as irregularidades entre o dente e a restauração e de resistir à ação do ambiente bucal, a curto e longo prazo. A adesão também é importante nesse contexto, pois uma forte ligação entre o agente cimentante e os substratos dentários pode ajudar a impedir que as bactérias colonizem a interface, minimizando o trânsito de fluidos que possam causar hipersensibilidade dentinária (CALLISTER; RETHWISCH, 2012). Portanto, o sucesso clínico das restaurações cerâmicas está diretamente relacionado à cimentação, à qualidade e à durabilidade da união entre o sistema cimentante com o substrato e com a cerâmica (PEUTZFELDT et al., 2011), a qual dependerá da interação química e mecânica entre adesivo e aderente (CALLISTER; RETHWISCH, 2012).

De acordo com de Kok e colaboradores (DE KOK et al., 2018), quando a superfície de cimentação da cerâmica de dissilicato de lítio é rugosa, em consequência da fresagem em CAD/CAM por exemplo, e não é adequadamente aderida ao substrato, ocorre um impacto deletério significativo nas suas propriedades mecânicas. Porém, quando existe uma boa adesão ao substrato, o efeito da rugosidade interna passa a não influenciar no resultado final de resistência à fratura da cerâmica. Por outro lado, o estudo de Rodrigues et al. (2018) mostrou que o ajuste interno de uma cerâmica a base de dissilicato de lítio (e.max CAD, Ivoclar Vivandent) com ponta diamantada pode reduzir drasticamente a resistência à fadiga de tal cerâmica mesmo quando esta é cimentada adesivamente, e quanto maior a granulação da ponta utilizada menor será a resistência. Deve-se considerar que no referido estudo, a ponta diamantada, além de inserir defeitos na superfície, produziu um desgaste de 60 µm (±5 µm) podendo este ter influenciado na distribuição das tensões geradas durante o teste de fadiga e por isso a cimentação adesiva não foi suficiente para neutralizar os efeitos da rugosidade superficial.

Evidencia-se que a qualidade da adesão cerâmica/cimento/substrato possui um papel muito significativo e determinante na resistência das restaurações cerâmicas, podendo limitar o efeito da sua rugosidade interna quando a adesão é adequadamente realizada. Neste sentido, um fator relevante a ser considerado como preditor do desempenho da restauração parece ser a capacidade do cimento de preencher completamente os defeitos da superfície cerâmica (THOMPSON et al., 1998). Cimentos resinosos com alto conteúdo de partículas inorgânicas são mais viscosos e podem diferir dos materiais menos viscosos em termos de seu potencial para gerar um contato íntimo entre o polímero infiltrante e os defeitos presentes na superfície da cerâmica (FLEMING; HOOI; ADDISON 2012).

A restauração cerâmica pode ser cimentada com diferentes cimentos, desde o fosfato de zinco, ionômero de vidro, ionômero de vidro reforçado por resina e cimentos resinosos, e o sucesso desta etapa irá depender da composição do material cerâmico, do substrato remanescente, do preparo protético dentário, do agente cimentante e da técnica de cimentação definitiva utilizados (BLATZ; SADAN; KERN, 2003; BLATZ et al., 2008). Os cimentos resinosos são indicados para a cimentação adesiva de restaurações cerâmicas e proporcionam melhor adesão tanto à cerâmica quanto ao substrato dentário/núcleo, bem como apresentam propriedades mecânicas e ópticas superiores (maiores possibilidades de seleção de cor e maior estabilidade de cor), além de maior resistência à hidrólise e à tração (PEUTZFELDT et al., 2011) do que os cimentos não resinosos. Estudos comprovando o adequado desempenho clínico e laboratorial da cimentação adesiva têm sido relatados há décadas. Um estudo clínico avaliando a sobrevivência de restaurações vítreas (Dicor) após 16 anos de acompanhamento mostrou que as restaurações cimentadas com cimento resinoso obtiveram uma taxa de sobrevivência mais favorável quando comparadas às cimentadas com cimento ionomérico ou de fosfato de zinco (MALAMENT; SOCRANSKY, 2001).

Contudo a cimentação não-adesiva, como os cimentos de ionômero de vidro, também tem sua indicação, no caso de restaurações de zircônia que possuem dimensões adequadas para esse tipo de abordagem (4 mm ou mais de altura do preparo e ângulo de convergência entre 6 e 15 graus) (STAWARCZYK et al., 2017; BLATZ; VONDERHEIDE; CONEJO, 2018). Outra alternativa são os cimentos de ionômero de vidro modificado por resina e o cimento resinoso autoadesivo, sendo que fornecem um certo nível de adesão aos tecidos dentários e à cerâmica, requerem menor tempo e sensibilidade técnica, e não exigem a aplicação prévia de um primer cerâmico (BLATZ et al. 2016). No entanto, as restaurações de zircônia mais finas, pouco retentivas e/ou que dependam da adesão (ex. próteses fixas adesivas, facetas e laminados cerâmicos) requerem uma cimentação adesiva através de agentes de união resinosos.

Enquanto muitos estudos focaram no efeito do condicionamento com HF e do jateamento na resistência de união aos materiais cerâmicos, poucos estudos investigaram o efeito de tais tratamentos na resistência à fratura de cerâmicas cimentadas submetidas às cargas cíclicas. Os testes de resistência à fadiga que aplicam cargas cíclicas sobre cerâmicas cimentadas são clinicamente mais relevantes do que os testes de flexão quando se procura investigar os efeitos relacionados às interações cerâmica-cimento (KELLY et al., 2017). De acordo com Arola (2017), a fadiga deve ser uma preocupação central no desenvolvimento de novos materiais odontológicos e na avaliação do sucesso das práticas restauradoras. Um maior reconhecimento das contribuições da fadiga para as falhas das restaurações e o desenvolvimento de abordagens que melhor simulem as condições clínicas são essenciais para estender a definição de saúde bucal a longo prazo (AROLA, 2017).

Portanto, adicionalmente ao estudo avaliando o efeito da rugosidade pós usinagem, os demais estudos desta tese buscaram investigar o efeito de diferentes tratamentos de superfície de uma cerâmica vítrea (com e sem condicionamento com ácido fluorídrico) e de uma cerâmica policristalina (jateamento com óxido de alumínio ou tratamento triboquímico) e da utilização de diferentes cimentos na resistência à fadiga de tais materiais quando estes são cimentados a um material análogo da dentina.

Assim, considerando os contextos expostos acima, quatro artigos científicos foram desenvolvidos e serão apresentados no presente trabalho, os quais encontram-se publicados ou submetidos em periódicos internacionais de alto fator de impacto na área de materiais dentários.

O primeiro artigo intitula-se ***"Fatigue performance and impact strength of distinct CAD/CAM dental ceramics: effect of surface roughness (polished vs. CAD/CAM milling simulation)"*** e buscou avaliar o efeito da rugosidade da superfície (material polido vs. simulação de fresagem em CAD/CAM) no comportamento de fadiga (carga de falha em fadiga, número de ciclos para a falha e probabilidade de sobrevivência) e na resistência ao impacto de cinco cerâmicas dentárias indicadas para restaurações monolíticas confeccionadas via CAD/CAM. O segundo artigo, que se intitula ***"Fatigue failure load of a bonded simplified monolithic feldspathic ceramic: influence of hydrofluoric acid etching and thermocycling"***, avaliou o efeito do condicionamento com ácido fluorídrico e da termociclagem na carga de falha por fadiga de restaurações cerâmicas feldspáticas cimentadas com dois cimentos resinosos de polimerização dual (sistema autoadesivo ou convencional auto-condicionante). Já no terceiro artigo, intitulado "***Fatigue failure load of cemented simplified monolithic zirconia: influence of luting system and aging***", objetivou-se avaliar a carga de falha em fadiga de restaurações simplificadas de zircônia monolítica cimentadas a um análogo de dentina com diferentes sistemas de cimentação, e avaliar o efeito do envelhecimento nas condições testadas. Por fim, o quarto artigo intitula-se ***"Effect of zirconia surface treatment, resin cement and aging on the load-bearing capacity under fatigue of thin simplified full-contour Y-TZP restorations"*** e investigou o efeito do tratamento de superfície da zircônia (jateamento com óxido de alumínio ou tratamento triboquímico) e do envelhecimento no comportamento à fadiga de restaurações finas monolíticas de Y-TZP (zircônia tetragonal policristalina) cimentadas com 2 tipos de cimentos resinosos, contendo ou não o monômero fosfatado MDP, a um substrato análogo à dentina.

2. ARTIGO 1 - FATIGUE PERFORMANCE AND IMPACT STRENGTH OF DISTINCT CAD/CAM DENTAL CERAMICS: EFFECT OF SURFACE ROUGHNESS (POLISHED VS. CAD/CAM MILLING SIMULATION)

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Original Research Article

Fatigue performance and impact strength of distinct CAD/CAM dental ceramics: effect of surface roughness (polished vs. CAD/CAM milling simulation)

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Short title: Roughness effect on fatigue and impact results of dental ceramics.

ABSTRACT

Purpose: To investigate the effect of surface roughness (polished vs. CAD/CAM milling simulation) on fatigue behavior and impact strength of five dental ceramics for manufacturing CAD/CAM monolithic restorations.

Methods: Specimens of five ceramics (FC- feldspathic; PICN- polymer-infiltrated ceramic-network; ZLS- zirconia-reinforced lithium silicate glass-ceramic; LD- lithium disilicate glass-ceramic; YZ- yttria-stabilized tetragonal zirconia polycrystal) to be tested under fatigue ($12 \times 12 \times 1.2 mm^3$; n=15) and impact ($15 \times 10 \times 2 mm^3$; n=15), were divided into two groups according to surface treatment: polished ‘p’ (#2500-grit SiC papers) and CAD/CAM milling simulation ‘gr’ (grinding with #60-grit SiC paper). Impact strength was tested using Dynstat method and the fatigue through the stepwise method (40N-660N; step of 20N; 10,000 cycles/step; 20Hz frequency). Roughness, topographic, fractographic and finite element analyses were performed. The impact strength data were analyzed by Weibull, the fatigue by Kaplan-Meier and Mantel-Cox (Log rank), and Pearson correlation was used to correlate roughness vs. impact and fatigue data.

Results: CAD/CAM milling simulation led to significantly ($p<0.05$) greater roughness (Ra and Rz) and lower fatigue performance. CAD/CAM milling simulation statistically reduced the impact strength for PICN, LD and YZ ceramics, and promoted a more irregular topography with scratches and grooves. Fractographic and FEA analyses depicted the origin of failure at the higher stress concentration side during the impact test, where the pendulum impacted. The failure origin of the fatigue tested samples was at the side subjected to tensile tension during test.

Conclusions: The CAD/CAM milling simulation significantly decreased the fatigue performance and the impact strength of the evaluated ceramic materials.

Keywords: Ceramics. Computer-Aided Design/Computer-Aided Manufacturing. Surface Properties. Resistance to Impact. Fatigue Behavior.

1. Introduction

The use of ceramic materials for manufacturing fixed dental prostheses (FDPs) is intensely common nowadays in dental practice. The major processing technique that has been explored is the use of Computer-Aided Design and Computer-Aided Manufacturing (CAD/CAM) systems as they enable increased process reliability, high cost-effectiveness and a substantial reduction in working time [1].

Dental ceramics are classified according to their crystalline phase (i.e. resin matrix ceramics, silicate ceramics, and oxide ceramics) and processing technique (i.e. layering build-up, pressing and CAD/CAM milling), undergoing updates as new restorative materials emerge in the search to combine adequate resistance and fracture toughness with ideal aesthetics and long-term clinical performance [2]. A common characteristic of dental ceramics, even for those with high strength (e.g., yttria-stabilized tetragonal zirconia polycrystal), is their relatively little capacity to absorb energy prior to fracture, i.e. they are brittle [3]. Still in this context, it is already clear that the ceramic microstructure influences its mechanical properties, and generally the higher the crystalline content, the better its mechanical performance [4]. The ceramic crystalline phase, porosity [5] and roughness [6] can also influence its optical properties.

An important aspect to be considered is that the CAD/CAM milling invariably creates a rougher surface, being that the inner surface classically remains untouched (as-milled), while the external surface of a restoration can be finished, polished and/or glazed [7]. In this regard and considering that all-ceramic restorations may fail from cracks and flaws at their inner surface (bonding surface) [8-10], the literature has pointed out this effect of milling with some concern and caution, since rougher surfaces may impair the mechanical properties of the ceramic by damage accumulation and lifetime reduction when under clinical service (cyclical intermittent loading) [11-13].

The capacity of a material to resist crack propagation considering that it has internal and external defects is an important factor that affects the performance of the ceramic restoration. Fracture toughness is a property capable of modifying this behavior, and this has led to the search for new materials such as hybrid materials (e.g., VITA Enamic) with a polymer network structure combining organic and inorganic components [14,15]. In fact, the fracture mechanics of ceramic materials in clinical performance has been defined based on the 'weakest link theory' [16] and by the slow-crack growth mechanism (sub-critical crack growth), where any existing inner defect in the material may grow to a critical size when subjected to mechanical stimuli and act as a crack initiator, leading to the ceramic prematurely fracturing under loads much smaller than expected (nominal strength of the material) [17]. There is still a gap in understanding how roughness can interfere with the clinical performance of different ceramic materials during functional loading and high impact loads.

Nevertheless, in addition to the presence of intermittent cyclic loading during mastication in the oral environment, overload can occur and be immediately transferred to the dental structures. The involuntary parafunctional habit, the chewing of hard foods and the impact suffered in accidents can generate excessive and extreme stress to the dental structures, often causing cracks and fracture of the dental element/restoration/implant [18-20]. In such scenarios, the fracture mechanism may be completely different from that considered in literature through static and fatigue tests [21-23]. Impact tests are rarely reported in the dental literature, and to the best of the authors' knowledge, studies which correlate the influence of surface patterns (polished vs. CAD/CAM milling simulation) on the impact and fatigue performance of dental ceramic materials with distinct microstructures are scarce.

Thus, this *in vitro* study aimed to investigate the effect of distinct surface patterns (polished vs. CAD/CAM milling simulation) on the fatigue performance and impact strength of five dental ceramic biomaterials for chairside clinical CAD/CAM monolithic restorations. The hypothesis of the study was that the CAD/CAM milling simulation of the ceramics and the type of ceramic material would significantly influence the fatigue performance and impact strength results.

2. Materials and Methods

The main features of the materials used in this study are presented in Table 1.

2.1 Preparation of specimens

2.1.1 Specimens for the fatigue test

Forty (40) ceramic plates were produced for each material based on the ISO 6872 [24] with a square modified shape to optimize the sample preparation time for the biaxial test, based on the study by Ramos et al. [25] which showed a similar stress distribution pattern between disc plates and square plates. For the FC (Feldspathic Ceramic - Vitablocs Mark II), PICN (Polymer-Infiltrated Ceramic-Network - VITA Enamic), ZLS (Zirconia-reinforced Lithium Silicate - VITA Suprinity), and LD (Lithium Disilicate glass-ceramic - IPS e.max CAD) groups, the specimens were cut in a high precision machine with a diamond saw (Isomet 1000, Buehler; Lake Bluff, IL, USA) under constant water cooling into their final dimensions of $12.0 \times 12.0 \times 1.2$ mm^3. The zirconia ceramic plates YZ (yttria-stabilized tetragonal zirconia polycrystal - VITA YZ T) were cut to a 20% larger size ($14.4 \times 14.4 \times 1.44$ mm^3) due to shrinkage after sintering.

After, all the specimens were sequentially polished with #280-, #400- and #800-grit sizes silicon carbide (SiC) papers and divided into two groups according to the surface treatments, polishing or CAD/CAM milling simulation (*Section 2.2*).

2.1.2 Specimens for the impact strength test

Thirty (30) rectangular (15.0 × 10.0 × 2.0 mm^3) specimens were produced for each material, following the same methodology described above. The YZ specimens were cut to a 20% larger (18.0 × 12.0 × 2.4 mm^3) due to shrinkage after sintering. The specimens were then polished (SiC papers #280-, #400- and #800-grit sizes) and divided into two groups according to the surface treatments, polishing or CAD/CAM milling simulation (*Section 2.2*).

2.2 Surface treatment

In order to emphasize differences in microstructure and material composition, the authors chose to standardize the polishing and the CAD/CAM milling simulation protocols for all materials rather than using different protocols to achieve equal roughness values. The same protocols were used for the specimens of the two tests performed (impact and fatigue tests).

Polishing ‘p’ – the specimens were polished in a grinding machine (ECOMET Grinder/Polisher, Buehler) under constant water cooling with SiC papers in the sequence of #1200 and #2500-grit sizes (Buehler) to create a smooth and standardized surface between the different ceramic materials.

CAD/CAM milling simulation ‘gr’ – the specimens were ground with #60-grit SiC paper (P60 - Black Stone Waterproof; BOSCH, Campinas, SP, Brazil), applying light finger pressure with oscillatory movements and constant water cooling for 30 s. This protocol was used in another study to simulate the surface roughness left by the CAD/CAM milling process [22] for a lithium disilicate glass-ceramic (IPS e.max CAD, Ivoclar Vivadent). Zucuni et al. [26] simulated the CAD/CAM milling (Ra = 1.8 ± 0.2 µm) on a pre-sintered Y-TZP ceramic (IPS e.max ZirCAD, Ivoclar Vivadent) with #220-grit SiC paper and found a lower roughness value (Ra = 1.2 ± 0.2 µm). Therefore, a coarser grit SiC paper (#60-grit) was also used in the present study to grind the pre-sintered (green body) Y-TZP specimens (YZ group), which is softer than the other ceramics during grinding.

After the surface treatments, the ZLS (Beginning chamber temperature 400 ºC for 4 min; Vacuum 1 at 410 ºC; Temperature increase at 55 ºC/min; Vacuum 2 at 839 ºC; Crystallization temperature 840 ºC for 8 min; Ending temperature 680 ºC) and LD (Beginning chamber temperature 403 ºC for 6 min; Temperature increase at 90 ºC/min; Vacuum 1 at 550 ºC; Crystallization temperature 1 at 820 ºC for 10 s; Vacuum 2 at 820 ºC; Temperature increase at 30 ºC/min; Crystallization temperature 2 at 840 ºC for 7 min; Ending temperature 700 ºC) specimens were crystallized in a porcelain furnace (Ivoclar Programat P100, Ivoclar

Vivadent AG, Schaan, Liechtenstein) and the YZ specimens were sintered (T0 = 25 ºC; Temperature rate increase 17 ºC/min; T1 = 1530 ºC; Holding time= 120 min; Cercon Heat, Degudent GmbH, Hanau-Wolfgang, Germany), both following the manufacturers' instructions.

2.3 Roughness analysis

The roughness of the specimens was measured with a profilometer (Mitutoyo SJ-400, Mitutoyo Corporation, Japan) according to the ISO 4287 [27] parameters (Ra - arithmetic mean of the absolute roughness values of the peaks and valleys measured from a medium plane; and Rz – average distance between the five highest peaks and five lowest valleys found in the standard) and after the crystallization and sintering processes have been performed. Ra and Rz were measured in micrometers (μm), with cut-off wavelengths of 0.8 mm (n=5), and λs=2.5 μm. Six measures were performed on each specimen, three on each axis (*x* and *y*).

2.4 Fatigue testing – Stepwise method (n=15)

The test specimens were subjected to the biaxial flexure fatigue strength test (piston-on-three-ball test, according to ISO 6872 [24]) on an electrodynamic machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA) through the application of cyclic loading using the Stepwise method. The specimen was placed on the top of three steel spheres (2.5 mm in diameter, 120° apart from each other, and forming a circle of 10 mm in diameter) with the treated surface facing down (tensile side) and the load was applied perpendicular to the center of the top surface on the specimen by a cylindrical steel piston with flat tip of 1.6 mm in diameter. A polyethylene film was placed between the supporting balls and the specimen to equally distribute the contact pressures. In addition, an adhesive tape was fixed on the compression side of the discs to provide uniform contact between the piston and the specimen and to prevent the spreading of the fragments after fracture. The test was performed with the specimens submerged in distilled water.

The fatigue data was determined using the Stepwise methodology described by Magne and Knezevic [28]. Cyclic load was applied at a frequency of 20 Hz, starting with a load of 20 N for 5,000 cycles (preconditioning phase to guarantee predictable positioning of the sphere with the specimen), followed by load increases of 20 N per step (i.e. 40, 60, 80, 100 …) at a maximum of 10,000 cycles each step. All the specimens were tested until their fracture. The fatigue failure load (FFL) and number cycles for failure (CFF) were recorded for statistical purposes.

2.5 Impact strength – Dynstat method (n=15)

The impact strength consists in the dynamic breaking of the sample and reading the amount of energy used to break it on the scale of the apparatus. A Dynstat apparatus (Zwick & Co., Eisingeu, Germany) was used according to the ISO 13802 [29] by which an impact test can be performed, where a flat pendulum is dropped to impact on the specimen that was fixed in a jig. The principle of the impact strength test is schematically drawn in figure 1. Corrected absorbed energy (E_c) was recorded, and the impact strength of each specimen was calculated according to the following formula:

The impact strength (a_{cU}) was calculated in kilojoules per square meter (kJ/m^2) with equation 1. Eq. (1) - $a_{cU} = \frac{E_c}{hb} \times 10^3$, where E_c is the corrected energy absorbed by breaking the test specimen [J], h is the height and b is the width of the specimen [mm].

2.6 Topographic analysis

Topographic micrographs were made to compare the polished and CAD/CAM milling simulation conditions of the different ceramic materials at 100× and 1,000× magnification. Representative specimens (n=2) were ultrasonically cleaned (1440 D, 50/60 Hz, Odontobras, Ind. e Com. Equip. Med. Odonto. LTDA, Ribeirao Preto, Sao Paulo, Brazil) in isopropyl alcohol for 10 minutes, completely air-spray dried, gold sputtered and analyzed on a Scanning Electron Microscope – SEM (XL 20, FEI Company, GG Eindhoven, the Netherlands).

2.7 Fractographic analysis

The specimens of the fatigue test were evaluated after failure in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss; Göttingen, Germany) at 100× magnification to determine their crack origin region. Representative specimens of each group (n=2) were ultrasonically cleaned (1440 D, 50/60 Hz, Odontobras) in isopropyl alcohol for 10 minutes, completely air-spray dried, sputtered with a gold-palladium alloy and then subjected to SEM analysis (200× magnification - VEGA3 Tescan; Brno-Kohoutovice, Czech Republic).

After the impact test, the specimens were evaluated in stereomicroscope (Olympus, Shinjuku, Tokyo, Japan) at 100× magnification. Representative specimens were selected, ultrasonically cleaned (1440 D, 50/60 Hz, Odontobras) in isopropyl alcohol for 10 minutes, completely air-spray dried, gold sputtered, and subjected to SEM analysis (XL 20, FEI Company, GG Eindhoven, the Netherlands) at 90× magnification.

2.8 Finite Element Analysis - FEA

A simplified three-dimensional FEA model of the impact test set-up with the same dimensions as the test set-up was created. The model consisted of the direct environment of the test sample of the impact tester supports and the part of the hammer in contact with the test sample, and the test sample itself. The surfaces in the interface between the impact tester and the test material were modeled with contact surfaces with a friction coefficient of 0.45. The Finite Element modeling was carried out using FEMAP software (FEMAP 11.1.2; Siemens PLM software, Plano, TX, USA), while the analysis was done with NX Nastran software (NX Nastran; Siemens PLM Software, Plano, TX, USA). For the dimensions of the specimen, see figure 2. The model was composed of 30,952 parabolic tetrahedron solid elements. The mechanical properties of the materials of the test samples used were based on previous studies (Table 1) and the support and the hammer were made of steel (E = 200 GPa and Poisson ratio 0.3). The investigated materials are supposed to have brittle behavior in the impact test, so the deformation is linear to the load to failure. The kinetic energy of the pendulum in the impact tester is transferred into displacement energy of the test sample at the point of contact with the pendulum. Therefore, the impact on the test sample can be represented as a static force. A load of 100 N was applied on the hammer back surface. The nodes at the bottom of the impact tester were fixed and the nodes at the bottom of the pendulum were allowed to slide along the surface only.

2.9 Statistical analysis

SPSS v.23.0 (SPSS IBM, Chicago, IL, USA) was used to execute statistical tests with $\alpha = 0.05$. After assuring a normal and homogeneous distribution of the roughness data (Shapiro-Wilk and Levene's tests, respectively), a two-way analysis of variance (Two-way ANOVA) and a Tukey HSD multiple comparison post-hoc tests were used to observe statistical differences. Pearson correlation analysis was run to identify any possible linear correlation between roughness (*Ra*) and the impact strength or fatigue failure load of each ceramic. The Pearson correlation coefficient value obtained in the analysis can range from +1 to −1, where +1 infers a total positive linear correlation, 0 to no linear correlation, and −1 to a total negative linear correlation. The correlation intensity was classified according to Cohen [30], where values between 0.10 to 0.29 are considered a weak correlation, 0.30 to 0.49 as medium, and 0.50 to 1.00 interval is considered a high correlation.

Fatigue data (fatigue failure load – FFL, number of cycles for failure – CFF and survival rates) were analyzed by means of the Kaplan-Meier and Mantel-Cox (Log-rank) tests.

The impact test data were analyzed through Weibull analysis [31] using the SuperSMITH Weibull 4.0k-32 software (Wes Fulton, Torrance, CA, USA) in order to determine the characteristic strength (σ_o - strength at a failure probability of approximately 63%) and the Weibull modulus (expresses the mechanical reliability of the material) of the groups. The Weibull plot has the 95% confidence bounds of the estimate for the Weibull shape parameter (Weibull modulus, m) on the Y-axis, and 95% confidence bounds for the estimate of the characteristic strength (σ_o) on the X-axis. If contour plots intersect, Weibull parameters are not statistically different [32].

3. Results

The specimens subjected to CAD/CAM milling simulation had a rougher surface (*Ra* and *Rz*) than polished ones, both for fatigue (Table 2) and impact (Table 3) tests.

The results of the two tests (fatigue and impact) generally showed lower values for the feldspathic ceramic (FC), followed by the polymer-infiltrated ceramic-network (PICN), intermediary results for high-crystalline content lithium-based glass-ceramics (LD and ZLS), and the highest values for the polycrystalline ceramic (YZ) (Tables 2 and 3). The pairwise comparison showed that the CAD/CAM milling simulation process statistically reduced the fatigue failure load and the number of cycles for failure of all the ceramic materials (Table 2 and Fig. 3). The survival rate analysis (Table 4 and Fig. 3) showed that the CAD/CAM milling simulation conditions led to a lower survival rate than the polished conditions for all the evaluated materials, meaning that the failure occurs earlier when the surfaces are rougher, apart from the material type (Table 4). The characteristic strength of the impact test results was statistically reduced for PICN (76% of decrease), LD (43%) and YZ (72%) ceramics after the CAD/CAM milling simulation (Table 3). The Weibull modulus did not differ within each type of ceramic (Table 3).

The Pearson Correlation analysis between roughness and fatigue data (FFL) showed high and negative correlation (i.e. the higher the roughness, the lower the fatigue failure load) for all the materials, except for FC which had a medium correlation value (Table 2). For impact data, a high and negative Pearson correlation coefficient could be found for the PICN, ZLS, LD and YZ materials, and no correlation for FC (Table 3).

SEM micrographs revealed that the CAD/CAM milling simulation groups presented a more irregular topography compared to the polished ones, presenting scratches and grooves on the ceramic surface. It was also possible to observe some differences between the materials, highlighting their physical and mechanical particularities (Fig. 4).

The fractographic analysis of the fatigue test specimens (Fig. 5) shows that the failure origin is located on the side under tensile stress (down side) where higher stress concentration is found, from which the crack propagated towards the compression stress side (upper or opposite side). For impact test specimens, the fractography (Fig. 6) shows the failure origin located at the principal stress concentration region in the impact strength test, according to the FEA analysis showed in the figure 2.

The FEA analysis showed that the maximum principal stress concentrates at the surface when the pendulum impacts the specimen, for all the materials, as demonstrated by the representative image in figure 2.

4. Discussion

Survival analysis and impact strength are two analyses used in dentistry to determine the mechanical behavior of dental ceramic materials. The results of our study showed that both the impact and the fatigue results were influenced by the ceramic material and the surface pattern, and therefore the study hypothesis was accepted. The CAD/CAM milling simulation of the ceramics surfaces significantly decreased the fatigue performance of all the evaluated ceramic materials and also the impact strength of most of them. Thus, the rougher surface has a damaging effect on the impact and fatigue results (Tables 2 and 3).

Despite having a negative effect, the rough surfaces play a relevant role for improvements in micromechanical bonds [12,33,34]. The overall effect of this roughness, which is unavoidable with CAD/CAM milling, is a decrease in the mechanical properties of dental ceramics, especially if these defects are not properly filled by the adhesive bonding [12,33,34]. Hence, the importance of a strong and stable adhesion between ceramics and resinous materials in the filling of the defects, consequently improving the mechanical behavior, must be highlighted. From this point of view, studies investigating the effect of smoother or rougher ceramic surfaces on the mechanical behavior of ceramics bonded to the substrate should be conducted for better understanding of the overall effects of this subject.

It is well known that the flaw population itself influences the mechanical performance of ceramic brittle materials, since a larger flaw population increases the chances of crack initiation [2,16,17,35]. The decreased strength is related to the largest and sharper flaws, i.e.

those present after coarse grinding [36], as observed in our study (Table 2 and 3; Fig. 4). The fracture toughness measures the material resistance to the crack growth, and, generally, the greater the toughness, the greater the material resistance will be. It is generally possible to correlate the material toughness (Table 1) with its strength, and the polished Y-TZP which has higher toughness ($K_{Ic} = \pm 4.5$) also had better mechanical behavior (Tables 2 and 3). It is also shown by other studies [22,37,38] that the greater the diamond bur grit size, the greater the damage, and the strength is then more intensely reduced.

Since the materials used in the present study have different physical and chemical characteristics and also distinct composition, they tend to respond differently under a required/specific stress [4,39,40]. Considering previously reported values for biaxial flexural strength and fatigue strength [23,39-41], and based on the characteristics of the materials evaluated in the present study (Table 1), our results for impact strength and fatigue failure load are consistent and show lower values for the feldspathic ceramic (FC), followed by the polymer-infiltrated ceramic-network (PICN), with intermediary values for the high-crystalline content lithium-based glass-ceramics (LD and ZLS), and the highest values for the Y-TZP (YZ).

According to Kruzic et al. [42], the microstructures of the ceramics have a significant influence on their fracture and fatigue behavior through their different toughening mechanisms, such as crack bridging (for dental composites and PICN, for example), the formation of a plastic deformation zone around the crack tip (PICN), crack deflection (FC, LD and ZLS) or transformation toughening (YZ) [42]. Generally, the lower the glass content (YZ < ZLS and LD < PICN < FC; Table 1), the higher the ceramic strength. This is corroborated by many studies [23,39-41] and also by our results (Tables 2 and 3).

The purpose of the impact test is to ascertain the behavior of specified test specimens under defined impact loading conditions, and to assess the material's flexural strength based on the impact strength results. The swinging pendulum (Fig. 1B) first measures the energy required to break a specimen and then calculate its strength. As demonstrated by Brostow and Lobland [43], the impact strength is connected to the material brittleness, and as the machining process introduces defects on the ceramic surface, its surface roughness increases, in turn jeopardizing its biaxial flexural strength [7,44]. Thus, the evaluation of impact strength between CAD/CAM milled and polished ceramic materials is very pertinent. The characteristic strength from the impact data was statistically reduced for PICN (76% of decrease), LD (43%) and YZ (72%) ceramics after they were submitted to the CAD/CAM milling simulation, and the Weibull modulus remained statistically equal comparing polished and CAD/CAM milling simulation conditions within each type of ceramic (Table 3). The polished YZ material had the highest impact strength, and the impact strength was reduced to the same level of polished ZLS and LD materials when the CAD/CAM milling simulation was performed (Table 3).

Previous literature has reported that the ceramic microstructure would influence the wear behavior of dental ceramics [45]. In our study, the ceramic microstructure did not influence the polishing result when the same protocol was applied, and all the materials had the same roughness after polishing (Tables 2 and 3; Fig. 4). However, different roughness values were reported after the CAD/CAM milling simulation (Tables 2 and 3). The authors suggest that those differences, aside from being influenced by the microstructure and material composition, were caused by grinding the lithium-based ceramics (LD and ZLS) in their meta-sintered/pre-crystallized stage and the zirconia (YZ) ceramic in its pre-sintered ('green body') phase, which facilitates grinding and allows a rougher surface [4], with the final crystallization and sintering processes being unable to eliminate the milling defects [4,46].

The linear Pearson's correlation coefficient found a high negative linear correlation for the materials, showing that when the roughness is higher, the fatigue failure load and impact strength decrease. The exception was the feldspathic ceramic, which had a weak and a medium correlation for impact and fatigue results, respectively (Tables 2 and 3). This difference may have been caused by the small variation in roughness values between the FC polished and CAD/CAM milling simulation conditions, which may have reduced the roughness effect on the outcomes. Moreover, the distinct effect (weak and medium correlations) for FC materials more strongly affected the fatigue than the impact results, which may be associated to differences in test geometry and fracture mechanics, since the cyclic intermittent loading of brittle materials leads to slow crack growths in defects over time, different from impact testing [39,47,48] (Table 2).

The topographic analysis shows a great difference in the surface of the ceramics treated with the CAD/CAM milling simulation process, presenting a more irregular surface with scratches, pits, and fissures (Fig. 4). Although the materials have been ground using the same protocol, it is still possible to observe that they present surface characteristics quite different from each other owing to their differences in properties and microstructures [4]. This becomes more evident when we compare the two materials that are structurally more similar (the lithium disilicate glass-ceramic, LD, and the zirconia-reinforced lithium silicate, ZLS): we observe that they behaved (Tables 2 and 3) and are microstructurally (Fig. 4) more similar, as corroborated by Ramos et al. [39] and Belli et al. [4]. It is also possible to observe that the feldspathic ceramic has some defects (e.g., bubbles and fissures) that remain on the surface after polishing (Fig. 4). As described by Griffith's law [16] and slow crack growth mechanism [17], the presence of critical defects leads to damage accumulation and jeopardizes the material strength. According to the fracture analysis performed by Quinn et al. [49] for the feldspathic ceramic (FC), intrinsic volume flaws (microstructural heterogeneities and pores), as demonstrated in Figures 4 and 5 (dashed arrows), could be the nature of strength-limiting defects, with the polishing process being unable to remove all the subsurface damage from the earlier CAD/CAM milling simulation.

The fractographic analysis of the fatigue tested specimens corroborate other studies [10,25,50,51] and shows that the failure origin was on the side under tensile stress (bottom side in Fig. 5) where higher stress concentration is found, from which the crack propagated toward the compression stress side (upside or opposite side). Meanwhile, the failure origin and crack propagation for the impact test specimens (Fig. 6) are located at the principal stress concentration region during the test, being in accordance with the representative FEA analysis (Fig. 2), and the study of Thomaidis et al. [52], at the part of the specimen at the height corresponding with the point of contact with the support and to the tensile tension side where the pendulum impacts the specimen.

As an *in vitro* study that presents limitations, we point out the fact that the specimens were not cemented as the main limitation of this study, as discussed above, which could lead to greater energy absorption during the tests because the ceramics would be adhesively bonded to materials with lower elastic modulus (cement and substrate – dentin or epoxy resin) that are capable of absorbing and dissipating energy. Also, adhesive bonding is capable of filling the defects created by the roughening process, thereby increasing the ceramic material resistance and its structural reliability [12,33,53,54].

5. Conclusion

- The microstructure plays an important role in the mechanical behavior of the ceramic, and the higher the crystalline content, the greater the fatigue data and impact strength.
- The fatigue performance and the impact strength of the evaluated dental ceramics were significantly affected by the greater roughness.
- CAD/CAM milling simulation significantly decreased the fatigue performance of all the evaluated materials and also the impact strength of PICN, LD and YZ ceramic materials.

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TABLES

Table 1 – Ceramic materials used in the study and respective characteristics: commercial name, manufacturer and lot number; chemical content and microstructure, fracture toughness (K_{IC}) in $MPa.m^{0.5}$, Weibull modulus (*m*), Elastic modulus (*E*) in GPa, and Poisson's ratio (*ν*).

Ceramic Material	**Commercial Name**	**Manufacturer**	**Lot number**	**[1] Chemical content (wt%) and microstructure**	**Crystal size (µm)**	**Fracture toughness (K_{Ic}) in $MPa.m^{0.5}$**	**[1] Weibull modulus (*m*)**	**[1] Elastic modulus (*E*)**	**Poisson's ratio (*ν*)**
Feldspathic glass-ceramic **FC**	Vitablocs Mark II for Cerec® and inLab®; shade A2C, size I-14	VITA Zahnfabrik, Bad Säckingen, Germany	56731	56–64% SiO_2, 20–23% Al_2O_3, 6–9% Na_2O, 6–8% K_2O, 0.3–0.6% CaO, 0.0–0.1% TiO_2. Average particle size = ±4 µm.	[2] ±15 µm	[3] 0.84 ± 0.06	13.15	E = ±45 GPa	[3] *ν* = 0.23
Polymer infiltrated ceramic network **PICN**	VITA Enamic; 2 M2-HT EM-14	VITA Zahnfabrik	67300	86% inorganic ceramic (58–63% SiO_2, 20–23% Al_2O_3, 9–11% Na_2O, 4–6% K_2O, 0.5-2% B_2O_3, <1% ZrO_2, <1% CaO); 14% organic polymer (UDMA-urethane dimethacrylate, TEGDMA-triethylene glycol dimethacrylate).	[2] ±20 µm	[3] 0.86 ± 0.27	20	E = ±30 GPa	[3] *ν* = 0.28
Zirconia-reinforced lithium silicate glass-ceramic **ZLS**	VITA Suprinity; A2-HT PC-14	VITA Zahnfabrik	72790	56–64% SiO_2, 15–21% Li_2O, 1–4% K_2O, 3–8% P_2O_5, 1–4% Al_2O_3, 8-12% ZrO_2, 0-4% CeO_2, 0-6% Pigments. Homogeneous, fine crystalline structure.	[1] ±0.5 µm	[3] 1.25 ± 0.79	8.9	E = ±70 GPa	[3] *ν* = 0.23
Lithium disilicate glass-ceramic **LD**	IPS e.max CAD for Cerec® and inLab®; LT A2 / C 16	Ivoclar Vivadent AG, Schaan, Liechtenstein	W93126	58–80% SiO_2, 11–19% Li_2O, 0–13% K_2O, 0–8% ZrO_2, 0–5% Al_2O_3.	[1] ±1.5 µm	[3] 1.23 ± 0.26	8.63	E = ±95 GPa	[3] *ν* = 0.22
Yttria-stabilized tetragonal zirconia polycrystal **YZ**	VITA YZ-55/19 T White for inLab	VITA Zahnfabrik	76030	90.9–94.5% ZrO_2, 4-6% Y_2O_3, 1.5-2.5% HfO_2, 0-0.3% Al_2O_3, 0-0.3% Fe_2O_3.	[1] ±500 nm	[1] ±4.5	14	E = ±210 GPa	[4] *ν* = 0.32

[1]As disclosed by manufacturers; [2]Belli et al. [4]; [3]Ramos et al. [39]; [4]Borba et al. [21].

Table 2 – Roughness (Ra and Rz) of the specimens submitted to the fatigue test (Mean and Standard Deviation – SD); Pearson correlation coefficient (R) between *Ra* and fatigue failure load (FFL); survival analysis of the FFL and number of cycles for failure (CFF) of the fatigue stepwise test; and FFL percentage decrease from polished to CAD/CAM milling simulation condition within each material (p/gr).

Groups	† **Roughness (µm), Mean (SD)**		**R (p value)**	^{f} **Fatigue Test (Stepwise, n=15); mean and 95% confidence interval (CI)**		**FFL decrease from p/gr**
	Ra	***Rz***	**FFL × *Ra***	**FFL (N) - Mean (CI)**	**CFF - Mean (CI)**	
pFC	0.05 (0.01) ^{E}	0.61 (0.09) ^{E}	-0.39 (0.03)	65.3 (60.7 – 69.97) ^{G}	20,409 (18,704 – 22,114) ^{G}	±8%
grFC	1.08 (0.06) ^{D}	7.08 (0.33) ^{D}		60.0 (60.0 – 60.0) ^{H}	15,382 (15,019 – 15,745) ^{H}	
pPICN	0.06 (0.01) ^{E}	0.59 (0.10) ^{E}	-0.71 (0.00)	106.7 (100.4 – 112.9) ^{E}	39,867 (36,596 – 43,139) ^{E}	±20%
grPICN	2.13 (0.19) ^{B}	12.57 (0.96) ^{B}		85.33 (80.7 – 89.97) ^{F}	28,450 (26,184 – 30,716) ^{F}	
pZLS	0.06 (0.02) ^{E}	0.55 (0.11) ^{E}	-0.66 (0.00)	218.7 (192.0 – 245.3) ^{C}	97,127 (83,802 – 110,451) ^{C}	±32%
grZLS	1.98 (0.23) ^{B}	12.25 (1.05) ^{B}		148.0 (134.9 – 161.1) ^{D}	61,726 (55,759 – 67,693) ^{D}	
pLD	0.07 (0.02) ^{E}	0.59 (0.11) ^{E}	-0.69 (0.00)	232.0 (204.7 – 259.3) ^{BC}	103,994 (90,610 – 117,379) ^{BC}	±32%
grLD	1.69 (0.18) ^{C}	10.65 (0.96) ^{C}		158.7 (147.5 – 169.8) ^{D}	67,894 (62,433 – 73,354) ^{D}	
pYZ	0.12 (0.01) ^{E}	1.14 (0.17) ^{E}	-0.97 (0.00)	600.0 (578.0 – 621.98) ^{A}	287,207 (276,350 – 298,064) ^{A}	±56%
grYZ	3.94 (0.45) ^{A}	21.27 (2.18) ^{A}		264.0 (248.1 – 279.9) ^{B}	120,845 (113,026 – 128,664) ^{B}	

Different uppercase letters in each column indicate significant statistical difference based on the † Two-Way ANOVA and Post-hoc Tukey HSD ($p \leq 0.05$) and ^{f} Kaplan-Meier and Mantel-Cox (Log rank) test ($p \leq 0.05$).

Table 3 – Roughness (Ra and Rz) of the specimens submitted to the impact test (Mean and Standard Deviation – SD); Pearson correlation coefficient (R) between *Ra* and impact strength; Weibull analysis (Mean and 95% Confidence Interval - CI) of the impact strength test (Characteristic strength - σ_o in kJ/m^2; and Weibull modulus – *m*); and the decrease of 'σ_o' from polished to CAD/CAM milling simulation condition.

Study Groups	**† Roughness (μm), Mean (SD)**		**R (p value)**	***f* Weibull Analysis**		**σ_{oIt} decrease from p/gr**
	Ra	***Rz***	***Ra***	**Characteristic strength σ_{oIt} (kJ/m^2) - Mean (CI)**	**Weibull modulus (*m*) Mean (CI)**	
pFC	0.14 (0.02) ^{E}	0.80 (0.08) ^{F}	-0.15 (0.42)	0.83 (0.72– 0.94) ^{F}	4.10 (2.86 – 5.87) ^{AB}	±13%
grFC	1.12 (0.12) ^{D}	4.67 (0.43) ^{E}		0.72 (0.62– 0.83) ^{F}	4.26 (3.61 – 5.02) ^{AB}	
pPICN	0.06 (0.01) ^{E}	0.59 (0.10) ^{F}	-0.92 (0.00)	4.59 (4.09 – 5.16) ^{D}	4.65 (3.00 – 7.20) ^{AB}	±76%
grPICN	2.36 (0.23) ^{B}	13.77 (1.32) ^{B}		1.09 (0.98 – 1.23) ^{E}	4.67 (3.15 – 6.92) ^{AB}	
pZLS	0.04 (0.00) ^{E}	0.52 (0.06) ^{F}	-0.53 (0.00)	16.51 (12.18 – 22.38) ^{BC}	1.80 (1.13 – 2.86) ^{C}	±37%
grZLS	1.70 (1.11) ^{C}	10.71 (0.15) ^{C}		10.37 (8.50 – 12.65) ^{C}	2.77 (1.73 – 4.42) ^{BC}	
pLD	0.05 (0.01) ^{E}	0.46 (0.08) ^{F}	-0.61 (0.00)	17.69 (14.91 – 20.98) ^{B}	3.10 (2.05 – 4.68) ^{ABC}	±43%
grLD	1.58 (0.16) ^{C}	9.42 (0.88) ^{D}		10.09 (9.30 – 10.95) ^{C}	6.55 (4.43 – 9.67) ^{A}	
pYZ	0.12 (0.01) ^{E}	1.04 (0.10) ^{F}	-0.83 (0.00)	74.99 (63.29 – 88.85) ^{A}	3.20 (2.04 – 4.99) ^{ABC}	±72%
grYZ	3.80 (0.50) ^{A}	21.05 (2.32) ^{A}		20.67 (17.57 – 24.31) ^{B}	3.62 (2.98 – 4.40) ^{B}	

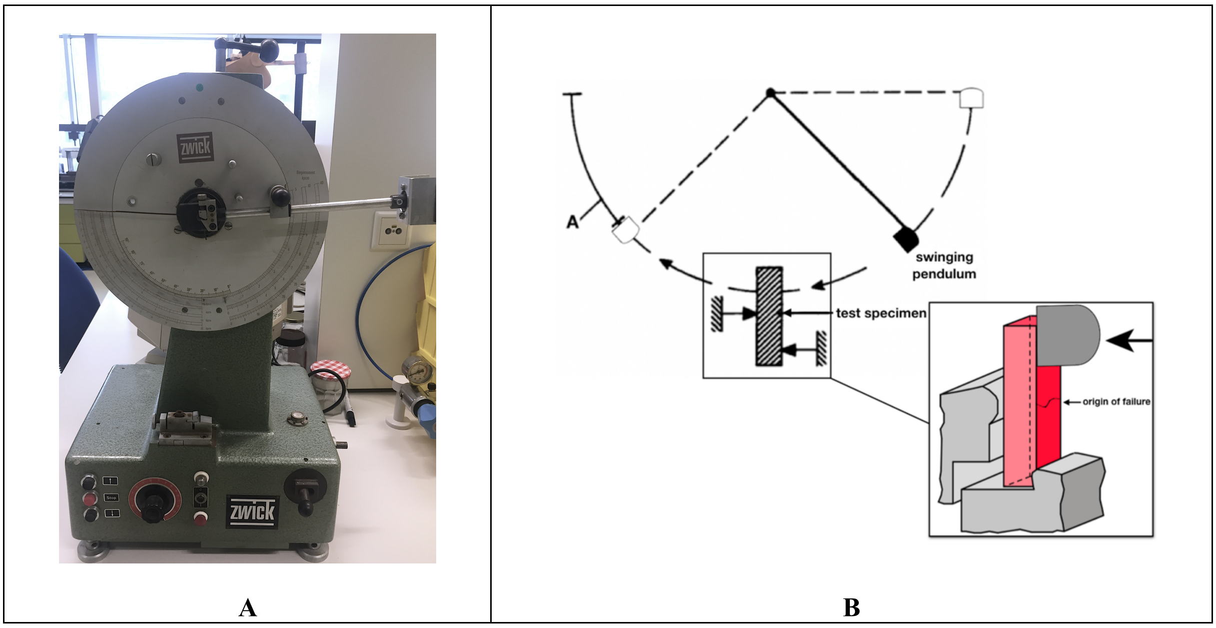
Different uppercase letters in each column indicate significant statistical difference based on the † Two-Way ANOVA and Post-hoc Tukey HSD, and based on the *f* Confidence Intervals overlapping of Weibull analysis.

Table 4 – Survival rates (in % - probability of the specimens to exceed the respective fatigue failure load and number of cycles for failure without fail) of the experimental groups and their respective standard error measurements.

Groups	Fatigue failure load (N) / Number of cycles for failure (×10^3) on the respective step*																										
	40/ 15	60/ 25	80/ 35	100/ 45	120/ 55	140/ 65	160/ 75	180/ 85	200/ 95	220/ 105	240/ 115	260/ 125	280/ 135	300/ 145	320/ 155	340/ 165	…	480/ 235	500/ 245	520/ 255	540/ 265	560/ 275	580/ 285	600/ 295	620/ 305	640/ 315	660/ 325
pFC	1	0.27 (0.1)	0.0 (0.0)	-	-	-	-	-	-	-	-	-	-	-	-	-	…	-	-	-	-	-	-	-	-	-	-
grFC	1	0.0 (0.0)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	…	-	-	-	-	-	-	-	-	-	-
pPICN	1	1	0.93 (0.06)	0.40 (0.13)	0.0 (0.0)	-	-	-	-	-	-	-	-	-	-	-	…	-	-	-	-	-	-	-	-	-	-
grPICN	1	1	0.27 (0.1)	0.0 (0.0)	-	-	-	-	-	-	-	-	-	-	-	-	…	-	-	-	-	-	-	-	-	-	-
pZLS	1	1	1	1	1	0.93 (0.06)	0.80 (0.10)	0.73 (0.1)	0.47 (0.13)	0.40 (0.13)	0.27 (0.11)	0.13 (0.09)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.0 (0.0)	…	-	-	-	-	-	-	-	-	-	-
grZLS	1	1	1	0.93 (0.06)	0.80 (0.10)	0.40 (0.13)	0.20 (0.10)	0.07 (0.06)	0.0 (0.0)	-	-	-	-	-	-	-	…	-	-	-	-	-	-	-	-	-	-
pLD	1	1	1	1	1	1	0.87 (0.09)	0.80 (0.10)	0.53 (0.13)	0.40 (0.13)	0.40 (0.13)	0.33 (0.12)	0.13 (0.09)	0.13 (0.09)	0.0 (0.0)	-	…	-	-	-	-	-	-	-	-	-	-
grLD	1	1	1	1	0.93 (0.06)	0.60 (0.1)	0.33 (0.12)	0.07 (0.06)	0.0 (0.0)	-	-	-	-	-	-	-	…	-	-	-	-	-	-	-	-	-	-
pYZ	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	…	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.87 (0.09)	0.67 (0.13)	0.40 (0.13)	0.27 (0.11)	0.07 (0.06)	0.0 (0.0)
grYZ	1	1	1	1	1	1	1	1	1	0.80 (0.10)	0.67 (0.12)	0.47 (0.13)	0.20 (0.10)	0.07 (0.06)	0.0 (0.0)	-	…	-	-	-	-	-	-	-	-	-	-

The sign '-' indicates absence of specimen being tested on the respective step. *These values are approximated. The sign '…' indicates the advance of steps without alteration on the survival probabilities.

FIGURES



A

B

Figure 1 – Impact test assembly. A) Dynstat apparatus for determination of impact strength, and B) principle of impact strength test: arc-length A is proportional to energy absorbed by breaking of the specimen. Design adapted from Wijn et al. [55] and Zwick/Roell Group for Dynstat test.

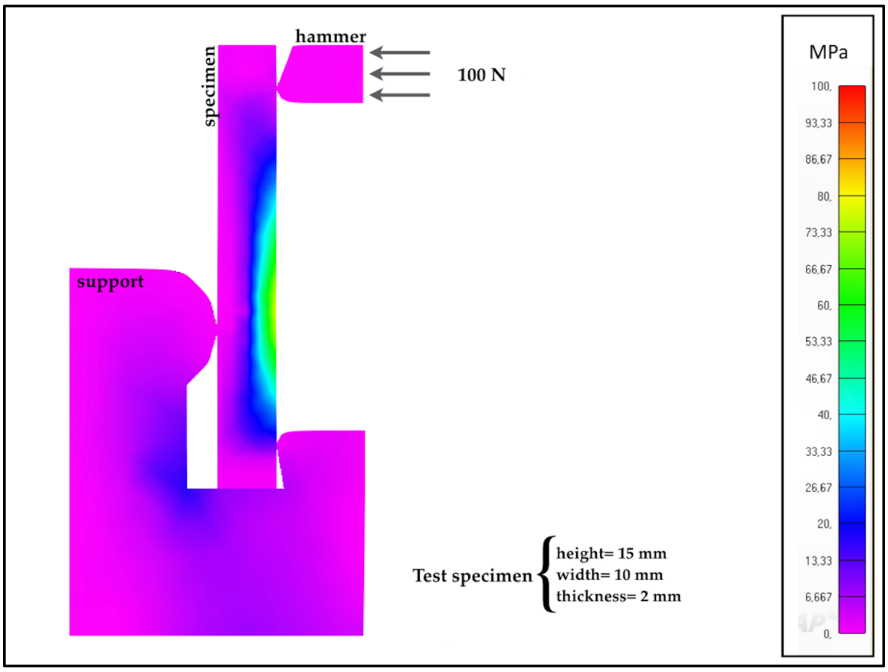
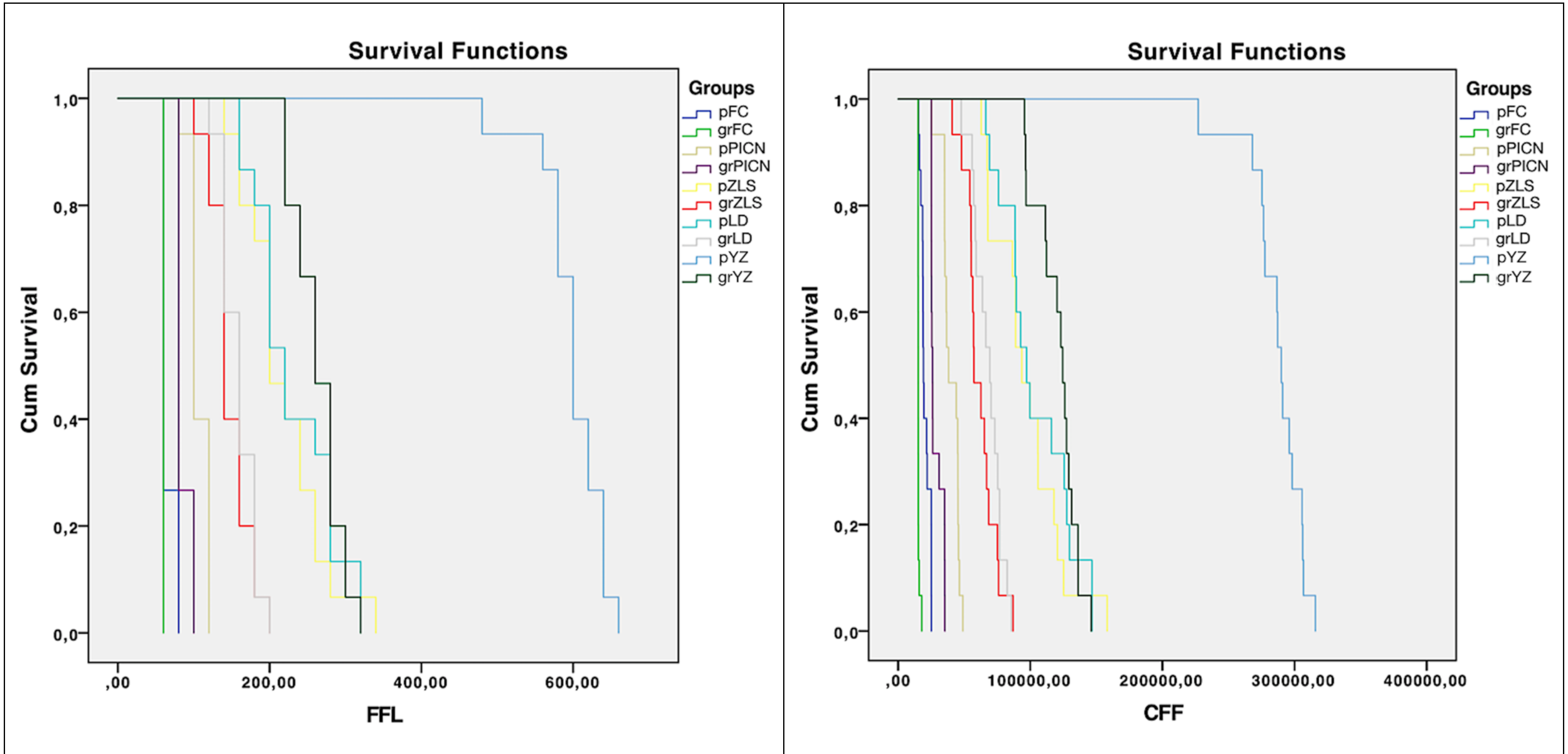


Figure 2 – Representative image of FEA analysis showing the maximum principal stress concentration at the surface when the pendulum impacts the specimen (polished feldspathic ceramic) during the impact test. This analysis was used to illustrate where it concentrates the maximum principal stress in such test which is few applied in dental ceramic studies.



A

B

Figure 3 – Survival graphs obtained by Kaplan-Meier and Log-rank (Mantel-Cox) tests for the (A) Fatigue Failure Load - FFL and (B) number of Cycles for Failure - CFF for each evaluated condition.

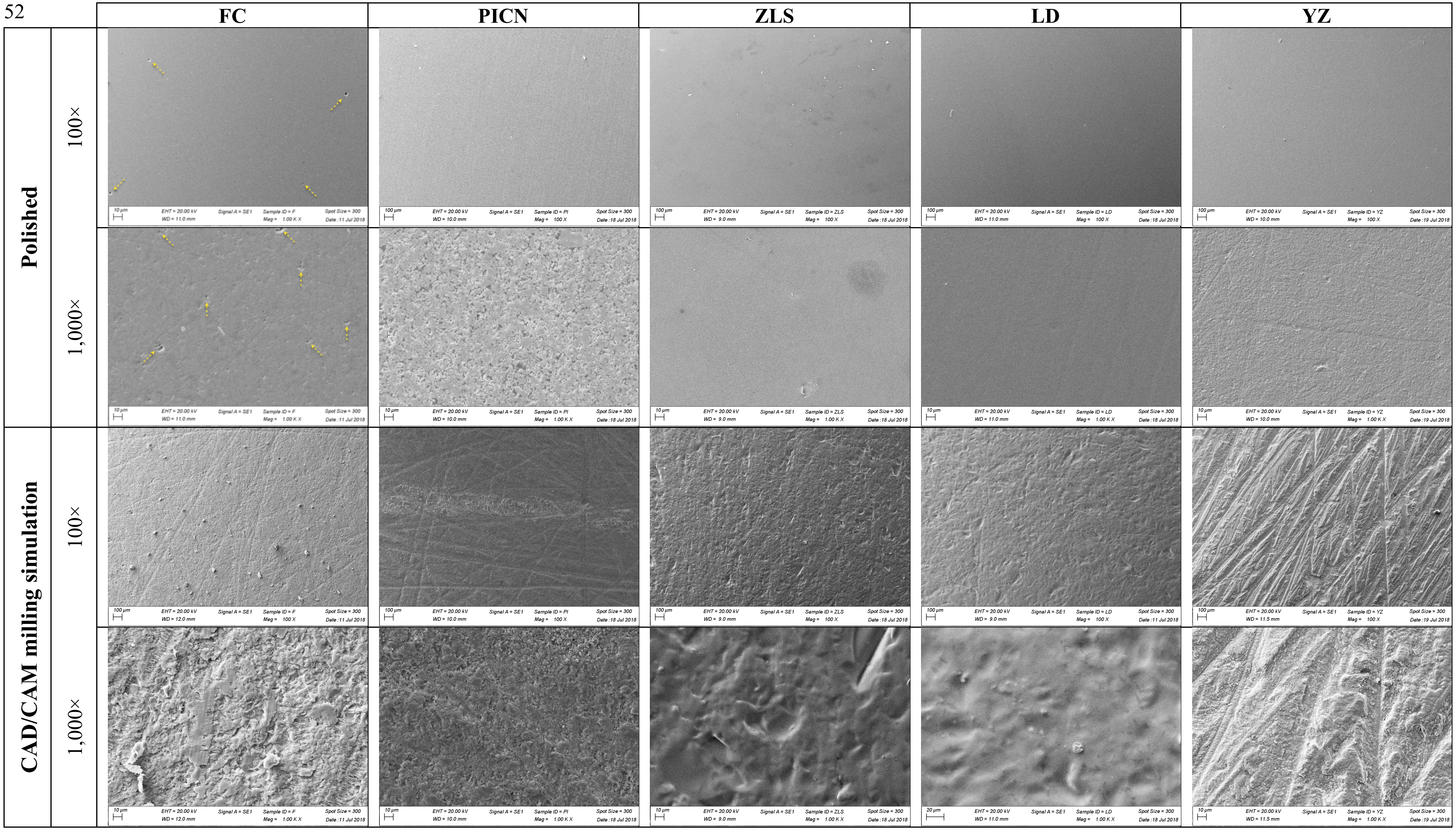


Figure 4 – Topographic images on Scanning Electron Microscopy (100× and 1,000× of magnification) of the ceramics' surfaces. Dashed arrows point to the defects (e.g., bubbles and fissures) that remain on the surface of the Feldspathic Ceramic after polishing.

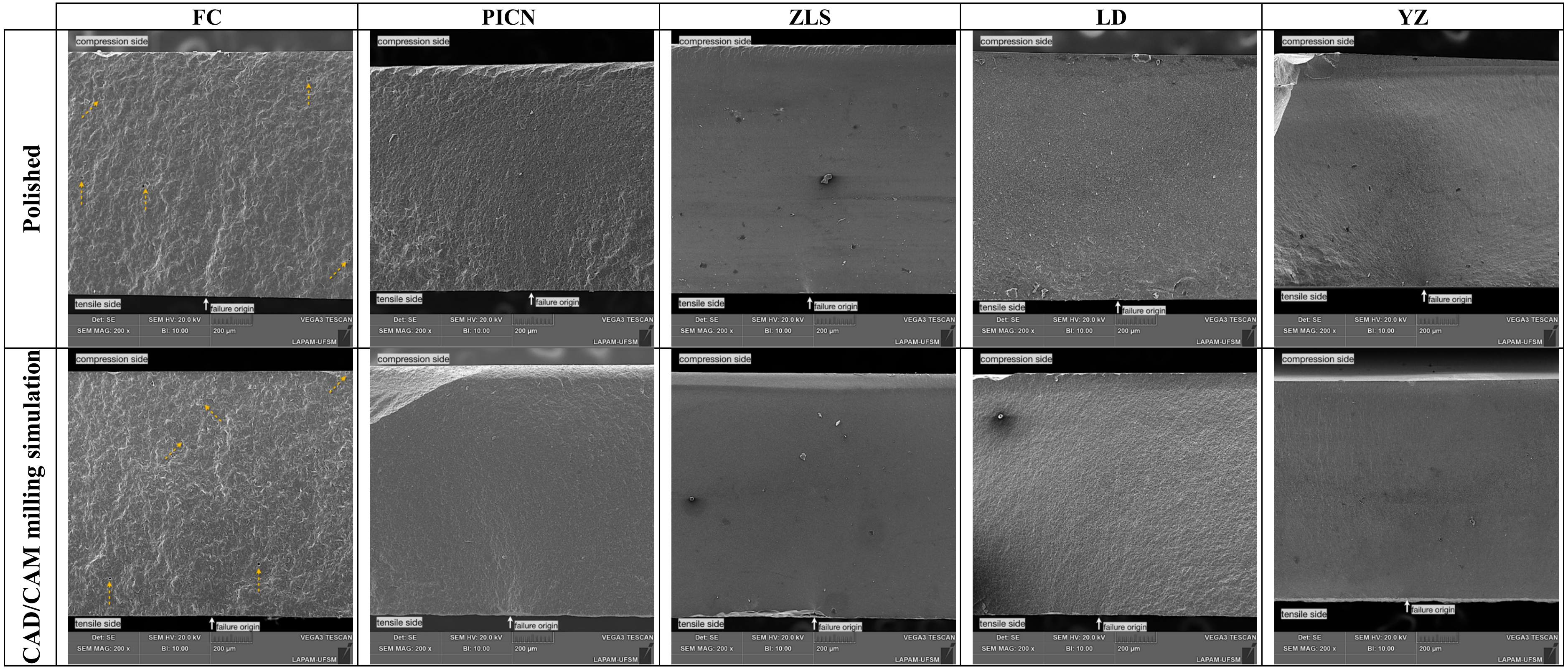


Figure 5 – Fractographic images (200× of magnification) on Scanning Electron Microscopy of the groups in the polished and CAD/CAM milling simulation conditions, showing the typical fractographic characteristics after the fatigue test. The origin of failure is located in the lower side of the micrographs, the area where tensile stress concentrates during the piston-on-three-ball fatigue test. Dashed arrows point to the bulk defects (e.g., bubbles and fissures) of the Feldspathic Ceramic inherent to its microstructure and processing.

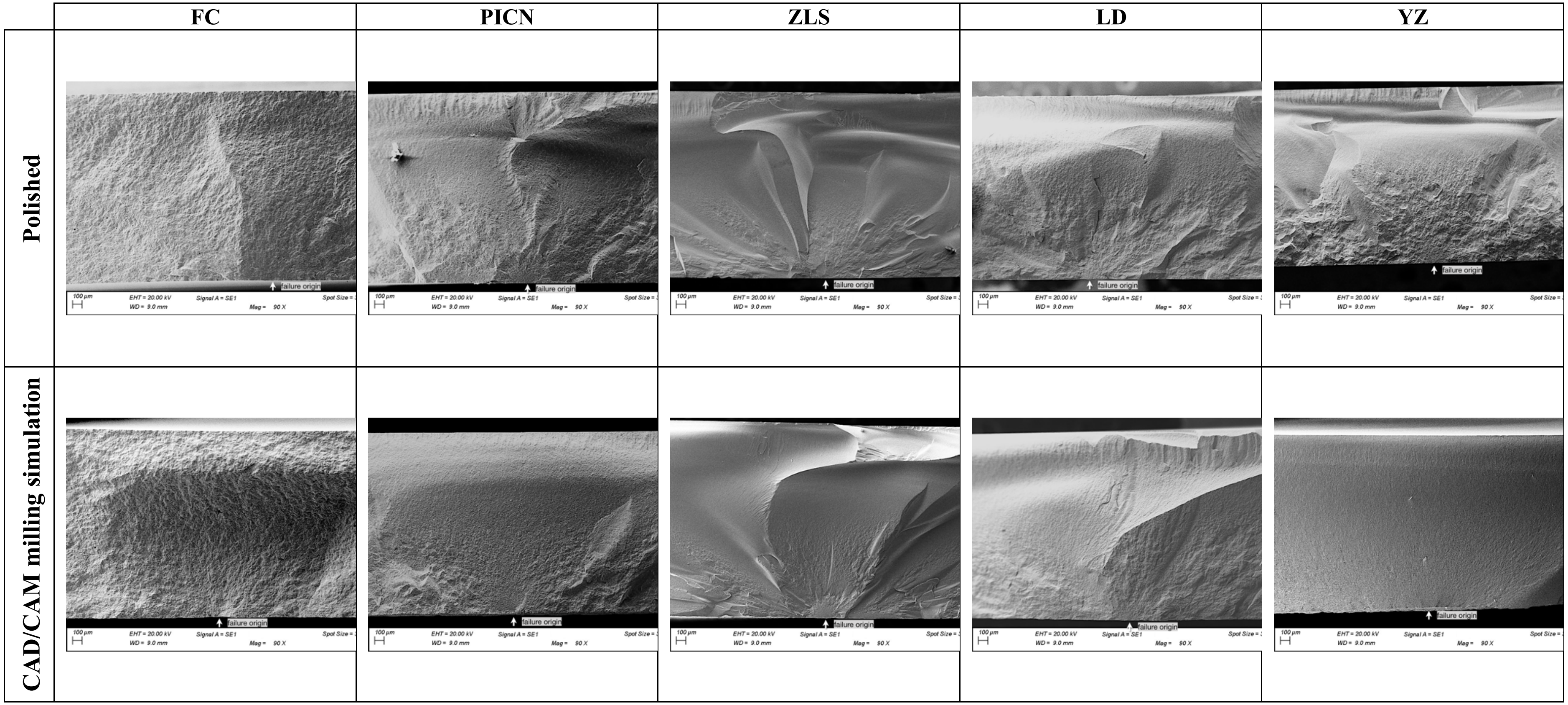


Figure 6 – Fractographic images (90× of magnification) on Scanning Electron Microscopy of the groups in the polished and CAD/CAM milling simulation conditions, showing the typical fractographic characteristics after the impact strength test. The origin of failure is located in the lower side of the micrographs, corresponding to the region of maximum tensile stress in the impact strength test, according to the FEA analysis in the Fig. 2.

3. ARTIGO 2 - FATIGUE FAILURE LOAD OF A BONDED SIMPLIFIED MONOLITHIC FELDSPATHIC CERAMIC: INFLUENCE OF HYDROFLUORIC ACID ETCHING AND THERMOCYCLING

Este artigo está aceito no periódico *Operative Dentistry*, ISSN: 1559-2863 (online), Fator de impacto = 1,180; Qualis CAPES A1. As normas para publicação estão descritas no Anexo B.

Fatigue failure load of a bonded simplified monolithic feldspathic ceramic: influence of hydrofluoric acid etching and thermocycling

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Short title: Etching and aging on fatigue of feldspathic ceramic.

ABSTRACT

Objective: To evaluate the effect of hydrofluoric acid (HF) etching and thermocycling (Tc) on fatigue failure load of feldspathic ceramic restorations cemented with two resin cements. **Methods:** Disc-shaped feldspathic ceramic (Vitablocs Mark II; Ø=10 mm, 1.0 mm thick) and G10 epoxy resin (Ø=10 mm, 2.5 mm thick) specimens were made and randomly allocated considering three factors: ‘ceramic etching’ (i.e. with vs. without 10% HF plus silane application); ‘resin cement’ (i.e. self-adhesive - RelyX U200 ‘U200’ or conventional - Multilink Automix ‘MA’); and ‘thermocycling-Tc’ (i.e. with vs. without 5-55°C/12,000 cycles). Adhesive cementation followed each manufacturer’s instructions. Fatigue test (n=20) was based on the staircase approach (250,000 cycles; 20 Hz). Contact angle, surface topography and fractography analysis were also executed. Specific statistical tests were employed for each outcome ($\alpha=0.05$). **Results:** The interaction of HF and Tc factors decreased the fatigue resistance for both cements (U200 542.63 > U200/HF-Tc 495.00; MA 544.47 > MA/HF-Tc 506.84). Comparing the cements associated with HF or Tc, there was statistical superiority for MA (U200-Tc 537.37 < MA-Tc 561.32; U200/HF 535.79 < MA/HF 557.11), and no statistical difference was detected when only cement type or its association with HF-Tc were compared (U200 542.63 = MA 544.47; U200/HF-Tc 495.00 = MA/HF-Tc 506.84). The fracture always originated from defects at the ceramic intaglio surface as radial cracks. **Conclusion:** HF-etching plus silane agent increased the ceramic surface free energy and its wettability, but it did not provide better results in terms of fatigue resistance compared to only silane agent application. The association of HF etching and aging significantly reduced the fatigue resistance of the material, regardless of the resin cement used.

Clinical Relevance: Defects introduced by hydrofluoric acid etching can propagate when the assembly is subjected to aging and fatigue stimuli, impairing its mechanical performance.

Keywords: Vitreous ceramic. Fatigue testing. Surface treatments. Reinforcement by adhesion. Aging.

Running title: Etching and aging on fatigue of feldspathic ceramic.

INTRODUCTION

Hydrofluoric acid (HF) etching followed by silanization is the most widely accepted surface treatment for glass-ceramics.[1] HF etching selectively dissolves the glassy matrix of glass-ceramic surface creating retentive micropores, pits, cracks and grooves on the conditioned surface, favoring micromechanical retention.[2,3] A silane coupling agent is applied on the etched surface to promote a chemical bond between the inorganic phase of the ceramic (i.e. silica) with the organic phase of the resin cement (i.e. polymer).[4-6]

However, this has become increasingly debatable, as some studies have also shown no need for HF etching for enhancing adhesion between resin cements and glass-ceramics,[7-9] and it has also been demonstrated that HF etching may even lead to a ceramic weakening effect.[10,11] This deleterious impact is explained by the fact that HF etching also creates and increases the flaw population at the ceramic bonding interface. By that, it might increase the ceramic susceptibillity to slow crack growth of critical defects under the constant masticatory stresses, since the ceramic fracture strength is inversely proportional to the largest or critical flaw present in the loaded restoration, as described by Griffith's law.[12] From the clinical standpoint, the above-mentioned weakening effect is particularly relevant when considering the evidence showing that the glass-ceramic fails from flaws on the ceramic intaglio surface.[13,14]

Thus, a relevant factor to be considered as a predictor of the restoration performance seems to be the capacity of the cement to completely fill in the introduced defects,[15] which has been scarcely discussed in the literature. Resin cements are indicated for adhesive luting of feldspathic ceramic restorations. They provide better adhesion (higher bond strength) to both ceramic and restorative foundations/substrates, as well as superior mechanical and optical properties (greater color selection possibilities and higher color stability), as well as high resistance to hydrolysis, and great inherent tensile strength.[16] The advent of self-adhesive resin cements in the attempt to provide simplification and easy handling techniques appeared as a promising alternative approach and these cements have shown acceptable aesthetic properties and bond strength results at least comparable to the conventional resin cements.[1]

In addition to understanding the population of defects and the fill up potential of different cements on feldspathic ceramic restorations, another important condition involved in such a scenario is the subsequent degradation of the bonding interface. According to Lu et al.,[17] aging the bonding interface in water promotes a reduced elastic modulus of the cements, as well as an apparent reduction in the bond strength. This degradation of cement properties may be sufficient to redistribute stresses in the restorative set, thus reducing the ability of restorations to tolerate masticatory loads over time.

Few studies have studied the mechanical performance of feldspathic ceramics cemented to tooth substrates or analog materials under fatigue stimuli.[18,19] According to Strasser and collaborators,[20] ceramic pre-treatments (e.g., HF etching and air-abrasion) are fundamental to provide a better and stable adhesion (i.e. reduced susceptibility to interface degradation), and consequently better performance and long-term predictability for such restorations, being that its absence may increase the risk of premature failure.

Thus, considering the above assumptions, the lack of consensus on this thematic, and the clinical appeal, this *in vitro* study problematized the following: does hydrofluoric acid etching change the flaw population on the glass-ceramic intaglio surface and may consequently affect the fatigue behavior of adhesively bonded simplified monolithic glass-ceramic restorations when subjected to aging?

From these standpoints, the study aimed to evaluate the influence of hydrofluoric acid etching and aging on the fatigue failure load of a CAD/CAM monolithic feldspathic ceramic adhesively bonded to a dentin analog material using two resin cements (a self-adhesive and a conventional). The assumed hypotheses were: (1) hydrofluoric acid etching would not promote different fatigue behavior compared to non-etching; (2) thermocycling will reduce the fatigue failure load results; and (3) no significant difference would be found between the cements.

METHODS AND MATERIALS

Study design

Restorative sets were fabricated through a simplified approach[3] using feldspathic ceramic discs (Vita Mark II) cemented to glass fiber reinforced epoxy resin discs (G10 substrate - dentin analog) with conventional (Multilink Automix) and self-adhesive (RelyX U200) resin cements. The diameter of the specimens was determined as 10.0 mm because it resembles the average diameter of the first molar occlusal surface,[21] and the final thickness of the cemented set was 3.5 mm because it is equivalent to the average thickness between the occlusal surface and the pulp chamber roof of molar teeth.[22,23] The specimens were prepared and randomly (www.random.org) distributed considering the three factors under study: ‘ceramic etching’ (i.e. with vs. without 10% hydrofluoric acid (HF) etching plus silane application); ‘resin cement’ (i.e. self-adhesive - RelyX U200 ‘U200’ or conventional - Multilink Automix ‘MA’); and ‘thermocycling-Tc’ (i.e. with vs. without; 5-55°C/12,000 cycles) (Table 1).

Production of ceramic discs

Pre-fabricated feldsphatic ceramic blocks (Vitablocs Mark II for CEREC/inLab, Vita Zahnfabrik, Bad Säckingen, Germany; LOT 45950) were rounded into cylinders with a diamond drill (internal Ø= 10 mm; Diamant Boart, Brussels, Belgium) coupled to an eletric drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) and then sectioned in a precision diamond saw (Isomet 1000; 15LC Diamond Disc, Buehler, Lake Bluff, IL, USA), resulting in disc-shaped specimens with 1.05 mm of thickness. The procedures were executed with constant and abundant water-cooling. The 'occlusal' surfaces of the discs were sequentially polished (#400-, #600-, #800-, #1200- and #2000-grit silicon carbide abrasives; Buehler) to obtain a smooth top surface, removing the defects introduced by cutting and obtaining a final thickness of 1.0 mm (±0.01 mm). The opposite surface (i.e. cementation/intaglio surface) was kept 'as-cut' to ressemble milled CAD/CAM surfaces. After, the discs were cleaned with isopropyl alcohol (78%; 10 min) in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ribeirão Preto, Brazil) to remove any processing sediment (cutting and polishing procedures).

Production of dentin analog discs

Epoxy resin cylinders (± 250 mm length × 12.7 mm Ø; NEMA grade G10. Accurate Plastics Inc; New York, USA) were rounded in a polishing machine (#200 and #600-grit silicon carbide abrasives; EcoMet/AutoMet Polisher, Buehler) to obtain cylinders with 10 mm in diameter and then cut with a precision diamond saw (Isomet 1000, Buehler), as previously described in section *2.2*, with the exception that the thickness of this material was set to 2.7 mm. Both sides of the discs were sequentially polished (#400- and #600-grit SiC abrasives) to obtain smooth surfaces, removing the defects introduced by cutting until a final thickness of 2.5 mm.

Cementation procedure

Prior to cementation procedures, the discs were randomly assigned into eight groups (n=25) according to the study factors (Table 1). Based on the two levels (with vs. without) considered in the 'HF etching' factor, half of the ceramic specimens were only submitted to an ultrasonic bath (distilled water; 5 min) and air dried for 30 s, remaining untouched until the cementation procedure; while the other half was etched with 10% hydrofluoric acid (Condac 10 Porcelana, FGM, Joinville, Brazil) for 1 min, rinsed (1 min) and submitted to the ultrasonic bath protocol to remove any precipitates generated from acid etching, and then air-dried for 30 s prior to the cementation procedure. The epoxy resin specimens were all etched with 10% HF (Condac 10 Porcelana, FGM, Joinville, Brazil) for 1 min, rinsed (1 mim) and submitted to ultrasonic bath (distilled water; 5 min) to remove the debris formed during acid etching, and air-dried for 30 s prior to the cementation procedure.

The other cementation procedures were performed according to the manufacturer's instructions of the respective cement:

- self-adhesive resin cement (RelyX U200, 3M ESPE; Seefeld, Germany) - The silane-based primer (RelyX Ceramic Primer, 3M ESPE) was scrubbed in both substrates (ceramic and epoxy resin) for 5 s and air-dried until the solvent evaporation (10 s).

- conventional resin cement (Multilink Automix, Ivoclar Vivadent; Schaan, Liechtenstein) - Ceramic conditioning: The silane-based primer (Monobond Plus, Ivoclar Vivadent) was applied on the ceramic bonding surface, scrubbed for 15 s and then kept to react for 45 s, and air-dried. Epoxy resin conditioning: after the aforementioned HF etching, Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the epoxy resin surface for 30 s, and air-dried for around 5 sec to obtain a thin film.

After the primer applications, the respective cement was mixed (1:1 ratio), applied on the epoxy resin, and the ceramic disc was seated under a load of 250 g. The cement excess was removed with microbrush and light-curing was performed (1200 mW/cm^2, Radii-cal, SDI Limited, Bayswater, Australia) for 20 s at each region (occlusal and 4 axial surfaces - 0, 90, 180 and 270°). Prior to the fatigue test, the specimens were stored in distilled water (37°C) for 4 days. The specimens that were thermocycled were stored (distilled water; 37°C) for 1 day prior to the thermocycling and for additional 4 days after thermocycling and prior to the fatigue test.

Thermocycling - Tc

'Tc' subgroups (Table 1) were subjected to aging by thermocycling to stimulate the degradation of the bond interface. The specimens were subjected to 12,000 thermal cycles in water at temperatures of 5°C and 55°C with a dwell time of 30 s and a transfer time of 4 s (Ethik Technology; Vargem Grande Paulista, São Paulo, Brazil).

Fatigue failure load test – Staircase Method

First, in order to define the fatigue test parameters for each condition, a load-to-fracture test was executed in a universal testing machine (n=5; EMIC DL 2000; São José dos Pinhais, SP, Brazil) with a crosshead speed of 1 mm/min and incremental load until the auditory perception of cracking (i.e. presence of radial cracks confirmed by light trans-illumination and visual inspection) by a single trained blinded operator (L.F.G.) (i.e. the researcher did not know the respective group at the moment of testing).

The fatigue test was run in an eletric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, USA) following the Staircase Method described by Collins.[24] To better distribute the stress during testing and to avoid contact damage (Hertzian's cone cracks - fracture by surface contact damage), an adhesive tape (110 μm) was placed on the feldsphatic top surface and a non-rigid sheet (cellophane - 2.5 μm) was placed between the piston and the specimen.[19,25,26] The specimens were placed on a flat steel base, submerged in distilled water, and a stainless-steel hemisphere of 40 mm in diameter was used to apply the load on the center of the specimens' top surface.[14,27,28]

The fatigue test (n=20; Staircase Method) was run under a frequency of 20 Hz during 250,000 load pulses in each step, with a load amplitude ranging from a minimum of 10 N to the maximum load to failure for each specimen. The first specimen from each group was tested with an initial load close to the estimated fatigue failure load (~60% of the mean of load-to-fracture test) until the fracture or survival was observed at the number of predetermined cycles (250,000). Then the next specimen was tested with a step size (~5% of initial load) higher (when the previous specimen survived) or lower (when the previous specimen failed) than the initial loading level. This procedure was repeated until at least 15 samples per group were tested after the start of the test, and according to Collins,[24] the test only starts after the first stair inversion (first different outcome obtained), with 15 specimens being required to get reliable results following this methodology.

Contact angle measurements

Additional ceramic samples (n=3) for each evaluated condition (HF etching plus silane coupling agent application; only HF application; only silane application; and baseline - no HF and no silane application) were obtained and subjected to contact angle analysis through the sessile drop technique using a goniometer (Drop Shape analysis, model DSA 30S, Krüss; Hamburg, Germany) connected to a software program (DSA3, V1.0.3-08, Krüss). A drop (11 μl) of deionized water was deposited on the ceramic treated surface using a syringe, and 5 s after dropping the contact angle was measured for 10 s (series of 30 images per second).

Topographic analysis

Additional ceramic samples (n=2) for each evaluated condition (with and without HF etching) were prepared as previously described, sputtered with a gold-palladium alloy under vacuum, and then examined under Scanning Electron Microscopy (SEM; VEGA3 Tescan, Brno-Kohoutovice, Czech Republic) to evaluate its surface topography (500× and 2,500× of magnifications) and defects created by HF etching in the cross-sectional view (500× and 3,500× of magnifications). SEM images were obtained through the use of two detectors, Secondary Electron (SE) and Back Scattering Electron (BSE).

Fractographic analysis

All the failed specimens after fatigue test were evaluated in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss, Gottingen, Germany) to determine the presence and direction of radial cracks by light transillumination. Next, these specimens were sectioned in two halves, perpendicular to the direction of the cracks, in a high-precision diamond saw (Isomet 1000, Buehler). Then the sectioned halves were reanalyzed in a stereomicroscope to determine the crack origin and its propagation direction, and the representative cracks were selected and sputtered with a gold-palladium alloy under vacuum for a descriptive analysis of higher resolution in SEM (as described for topographic analysis) with 1,000× and 2,500× of magnifications.

Data analysis

All statistical analyses were performed using the IBM SPSS Statistics Program (v24 for Windows; IBM Corp; $\alpha=0.05$).

First, a three-way analysis of variance (ANOVA) was used to determine the influence of each factor on the fatigue failure load of the restorative set, and to elucidate any presence of interaction between the independent study variables (cement, HF etching, and Tc).

One-way ANOVA and post-hoc Bonferroni tests were adopted to compare the effect of different conditions for each respective cement separately. T-tests for independent samples were used between paired conditions (U200 vs. MA; U200-Tc vs. MA-Tc; U200/HF vs. MA/HF; U200/HF-Tc vs. MA/HF-Tc) to compare and depict the statistical differences for fatigue failure load between the cements exposed to the same study factor.

RESULTS

Fatigue failure load test - Staircase Method

Based on the three-way ANOVA, there were statiscally significant influences of the factors ‘cement’ ($p=0.006$; MA > U200), ‘thermocycling - Tc’ ($p=0.000$; without Tc > with Tc), ‘hydrofluric acid etching - HF’ ($p=0.000$; without HF > with HF), and for the interaction ‘HF × Tc’ ($p=0.000$) on the fatigue load results. No statistically significant influence was detected for other associations.

Analyzing each cement separately, the One-way ANOVA and post-hoc Bonferroni analyses showed that both cements behaved similarly to the different conditions, being that the fatigue resistance was only statistically reduced when the HF and Tc factors were applied together (Table 2, Fig. 1).

Comparing the same conditions between the cements through t-tests for independent samples, the MA cement yielded better results when the specimens were submitted to HF etching or Tc factors. There was no difference between the cements at baseline (without HF and without Tc) or for the association of factors (with HF and Tc) (Table 2).

Contact angle measurements

The lowest contact angle was observed after HF etching plus silane application, indicating greater surface wettability. The highest contact angle (lowest wettability) was observed on untreated surface (baseline group). Intermediate values were observed when only hydrofluoric acid (HF) etching or only silane was used (Fig. 2).

Topographic analysis

Topographical and cross-sectioned SEM images of baseline (nonetched) and HF etched (HF 10%; 1 min) specimens are shown in Figure 3. The baseline images showed a smoother and more homogeneous surface without any relevant irregularity (Fig. 3B). Etched surfaces presented an irregular topography characterized by the presence of numerous micro irregularities, pits, grooves, and striations as a result of the glassy phase dissolution, known as honeycomb-etched pattern (Fig. 3A).

Fractographic analysis

Stereomicroscope and scanning electron microscope analysis showed that all failures were radial cracks starting from the ceramic intaglio surface (Fig. 4). Cracks by contact damage between the piston and the ceramic surface were not found.

DISCUSSION

Our findings show that the hydrofluoric acid etching condition associated with thermocycling had a significant deleterious effect on the load for fatigue failure regardless of the cement used, and HF etching prior to silanization did not enhance the fatigue resistance of the simplified ceramic restorations.

Hydrofluoric acid etching promotes a surface dissolution of the ceramic glass matrix, creating micro retentions that contribute to the mechanical bonding with dental substrate when adhesively bonded.[9,10,29] However, this has become debatable, since fatigue properties of all-ceramic systems can be related to the flaw population (size, number and distribution) of the material,[12] and some studies have demonstrated that HF etching may lead to a decrease in the feldspathic ceramics strength by introducing defects on the surface that may not be completely filled by the resin cement.[10,30,31] In our study, the first hypothesis was accepted since HF etching did not promote different fatigue failure load results compared to baseline. Regarding the thermocycling factor, the second hypothesis was partially accepted since it only reduced the fatigue failure load for the HF etched specimens.

Other authors have obtained similar results, corroborating that HF etching improves the bond strength by creating micromechanical interlocking but may lead to a weakening effect on the ceramic, thereby compromising the clinical performance of the restoration.[10,31] Moreover, recent studies corroborate these findings, showing worse or equal results for bond strength,[7] biaxial flexural strength[11] and fatigue load[19] when conditioning glass-ceramic with HF prior to bonding, proving that this subject still requires future evaluations and considerations. Additionally, hydrofluoric acid has potentially hazardous toxicity known from other applications and should also be considered when applied in Dentistry.[32]

The topographic images in Figure 3C show the micro retentions, pits and grooves created by the selective HF etching of the glass-ceramic vitreous matrix, which could lead to a decrease in the ceramic fatigue resistance, especially when the defects are not completely filled by the cement. Defects created by HF etching may also lead to stress concentration, which can result in ceramic premature fracture starting from the adhesive interface,[18,30] as observed in our study (Fig. 4). This behavior is particularly important since sharp defects (as created by HF etching; Fig. 3C) are more damaging than rounded defects. In this sense, it has to be emphasized that we used a high concentration HF etchant (10% HF for 1 min) which produces many more defects than in lower concentrations (for instance, 1 and 5%)[19] and makes the material more susceptible to the presence of unfilled defects after bonding, and, consequently, to crack initiation and propagation under fatigue loads. Clinical[33,34] and *in vitro* studies[18] on failed glass-ceramic crowns have reported that the great majority of bulk fractures start from flaws on the ceramic intaglio surface, where high tensile stress is concentrated.

Adhesive cementation significantly increases the restorative material's fracture loads.[35] Using finite element analysis and fatigue test for the lithium disilicate glass-ceramic, de Kok et al.[36] proved that proper adhesion can better distributes the stress during loading, increasing the material's resistance. In our study, the group treated with hydrofluoric acid (which provides a surface with many more defects) (Fig. 3) resulted in a statistically similar fatigue resistance to the non-etched surface (baseline) (Table 2), corroborating the results found by de Kok et al.[36] with the bonding 'protective' role overcoming the ceramic's internal roughness effect.

Regarding the resin cement filling capacity, cements with low viscosity are more prone to penetrate the ceramic than cements with high viscosity.[37] According to Gamal et al.,[38] self-adhesive resin cements have high viscosity, and consequently are less capable of spreading onto the ceramic and substrate surfaces, which may compromise its wettability. The application of hydrofluoric acid etching plus silane has been recommended to promote chemical bonding between inorganic molecules of the ceramic with organic molecules of these resin cements.[39,40]

This sequence is already well established in the literature in terms of bond strength, as these procedures increase the surface energy of the ceramic and the wettability of the resin cement, improving adhesion.[3,31] This greater wettability can be evidenced by the lower contact angle of the treated surfaces, as shown in Figure 2A. In our work, in terms of fatigue resistance, we can note that this procedure may not summarily be necessary, since the groups which were not submitted to HF etching had the same or even better results than the etched ones. However, it must be considered that this assertion is only a finding with respect to fatigue resistance. From this viewpoint, there is a need to develop novel surface conditioning methods in order to address the problem related to the bond durability and defect fill in potential,[6] land trying to eliminate the HF use in order to produce a less technique-sensitive and safer (i.e. using a less hazardous material) bonding system.

As the conventional cement performed better than the self-adhesive when the HF etching and thermocycling factors were applied separately, the third hypothesis was partially accepted. According to Gamal et al.,[38] that result could be explained by the high viscosity of the self-adhesive resin cement and less spreadability onto the substrate/ceramic surface, reducing its ability to infiltrate into surface irregularities.

When the materials are free to deform, they will expand or contract due to fluctuations in temperatures. By that, the temperature changing during thermocycling is a deleterious factor for the adhesion between ceramic and dental substrate.[41] Due to the different coefficients of linear thermal expansion between the materials in the adhesive interface (ceramic/cement/substrate), they have different degrees of contraction and expansion during thermocycling leading to micro-mechanical fatigue stresses in the adhesive interface, breaking adhesive bonds and finally reducing the adhesion quality.[41] In the present study, the aging (thermocycling 5º-55ºC/12,000 times) only significantly affected the fatigue resistance for both cements when the ceramic was previously HF-etched. This corroborates Venturini et al.[31] who hypothesized that when the micro retentions are not completely filled by the cement, this empty and unfilled space allows faster water absorption at the interface of the restoration with its consequent hydrolysis and degradation, and finally a decrease in the material fatigue resistance.

The present study implemented a fatigue test under wet environment, where constant loads with ranging intensity were applied until the failure of the specimens. This method mimics the oral environment and more closely simulates the masticatory stresses when compared to the static test.[35] However, the applied test setup (axial load) may not fully simulate all the forces to which the material is subjected in the oral environment, especially to lateral loads (sliding motion) that generate compressive, tensile, and shear stresses on the ceramic surface leading to the subsurface crack formation and propagation.[42] Another limitation may be the simplified restorative set (disc-shaped specimens) which does not completely simulate the anatomy of a molar crown.

In our study, the loads at initial radial cracks (overall mean equal to 534.83 N) exceeded the maximum bite forces during mastication (148.73 to 354.01 N),[43] but were far below the maximum bite forces reached in sleep associated bruxism (±800 N) and maximum voluntary bite forces during daytime (±1,000 N).[44] Considering the subjects discussed above, we emphasize that our results should be carefully analyzed, and more *in vitro* and clinical findings can corroborate our results.

CONCLUSION

- HF-etching process associated with a silane coupling agent increased the ceramic surface free energy, and consequently its wettability, but it did not provide better results in terms of fatigue resistance compared to only silane agent application.
- When the feldspathic ceramics were HF-etched, bonded (regardless of the cement used) and then subjected to aging, the fatigue failure load of the restorations was significantly reduced.
- Hydrofluric acid etching and thermocycling factors applied alone did not lead to degradation of the ceramic fatigue resistance, regardless of the resin cement used (conventional - Multilink Automix or self-adhesive - RelyX U200), being that the conventional cement performed better in both cases (only HF and only Tc).

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TABLES CAPTIONS

Table 1. Study experimental design.

Table 2. Results from the monotonic load-to-failure test (n=5); the parameters defined to start the fatigue test (n=20; *initial load for fatigue test* = ~60% from the mean of load-to-fracture; *step size* = ~5% of initial load for fatigue test) based on Staircase Method; and Mean load for fatigue failure (L_f and standard deviation-SD) obtained through the fatigue test.

FIGURES CAPTIONS

Figure 1. Survivals and failures patterns observed during staircase fatigue testing (250,000 cycles; 20 Hz). Horizontal lines indicate the mean load value; red marks the start of up-and-down characters, solid marks represent survival and empty marks represent failure.

Figure 2. Representative images of contact angle measurements of ceramic surfaces subjected to the following treatments: A - hydrofluoric acid etching (HF) plus Silane coupling agent application; B - only HF application; C – only Silane application; and D – baseline (no treatment).

Figure 3. SEM images of the ceramic surface with hydrofluoric acid etching (A - 500× and 2,500×) and without hydrofluoric acid etching (B - 500× and 2,500×). Figure C shows the etching pattern in cross sectioned specimens: on the left side is the hydrofluoric acid etched (HF) ceramic in 500× (top left) and 3,500× (bottom left) of magnifications; on the right side is the nonetched (NHF) ceramic in 500× (top right) and 3,500× (bottom right) of magnifications.

Figure 4. Radial crack was the failure pattern for the failed specimens in all groups: The images show the radial crack that indicates the origin and pattern of ceramic failure. A and B: Stereomicroscope images of cut representative specimens where we can clearly see the radial crack (white arrows) starting at the intaglio ceramic surface; C - 1,000× and D - 2,500× of magnification: SEM images of a cut representative specimen with white arrows pointing to the radial crack.

TABLES

Table 1. Study experimental design.

RESIN CEMENTS (manufacturers)	BASELINE		AGED*	
	No HF ^{b}	**HF ^{ab}**	**No HF ^{b}**	**HF ^{ab}**
RelyX U200 (3M ESPE)	U200	U200/HF	U200-Tc	U200/HF-Tc
Multilink Automix (Ivoclar Vivadent)	MA	MA/HF	MA-Tc	MA/HF-Tc

[a] 10% hydrofluoric acid etching for 1 min.
[b] Silane coupling agent application: RelyX Ceramic Primer for the RelyX U200 cement and Monobond Plus for the Multilink Automix cement.
[*] Thermocycling - Tc: 12,000 cycles between 5° and 55 °C.

Table 2. Results from the monotonic load-to-failure test (n=5); the parameters defined to start the fatigue test (n=20; *initial load for fatigue test* = ~60% from the mean of load-to-fracture; *step size* = ~5% of initial load for fatigue test) based on Staircase Method; and Mean load for fatigue failure (L_f and standard deviation-SD) obtained through the fatigue test.

Groups	Mean of load-to-failure test (N)	Initial load for fatigue test (N)	Step-size (N)	Mean load for fatigue failure L_f (SD)* (N)
U200	820.4	490	25	542.63 (21.88) ^{Ab}
U200-Tc	900.8	540		537.37 (21.88) ^{Ab}
U200/HF	863.9	520		535.79 (22.38) ^{Ab}
U200/HF-Tc	870.6	520		495.00 (22.05) ^{Bb}
MA	841.0	505	25	544.47 (28.03) ^{Ab}
MA-Tc	888.5	535		561.32 (41.23) ^{Aa}
MA/HF	897.8	540		557.11 (20.50) ^{Aa}
MA/HF-Tc	781.1	470		506.84 (42.79) ^{Bb}

*Different uppercase letters mean statistical differences depicted by One-way ANOVA and post-hoc Bonferroni tests considering each cement individually (α=0.05).
*Different lowercase letters mean statistical differences depicted by independent samples t-tests for paired conditions between both cements (U200 vs. MA p=0.823; U200-Tc vs. MA-Tc p=0.034; U200/HF vs. MA/HF p=0.004; U200/HF-Tc vs. MA/HF-Tc p=0.293) (α=0.05).

FIGURES

Figure 1.

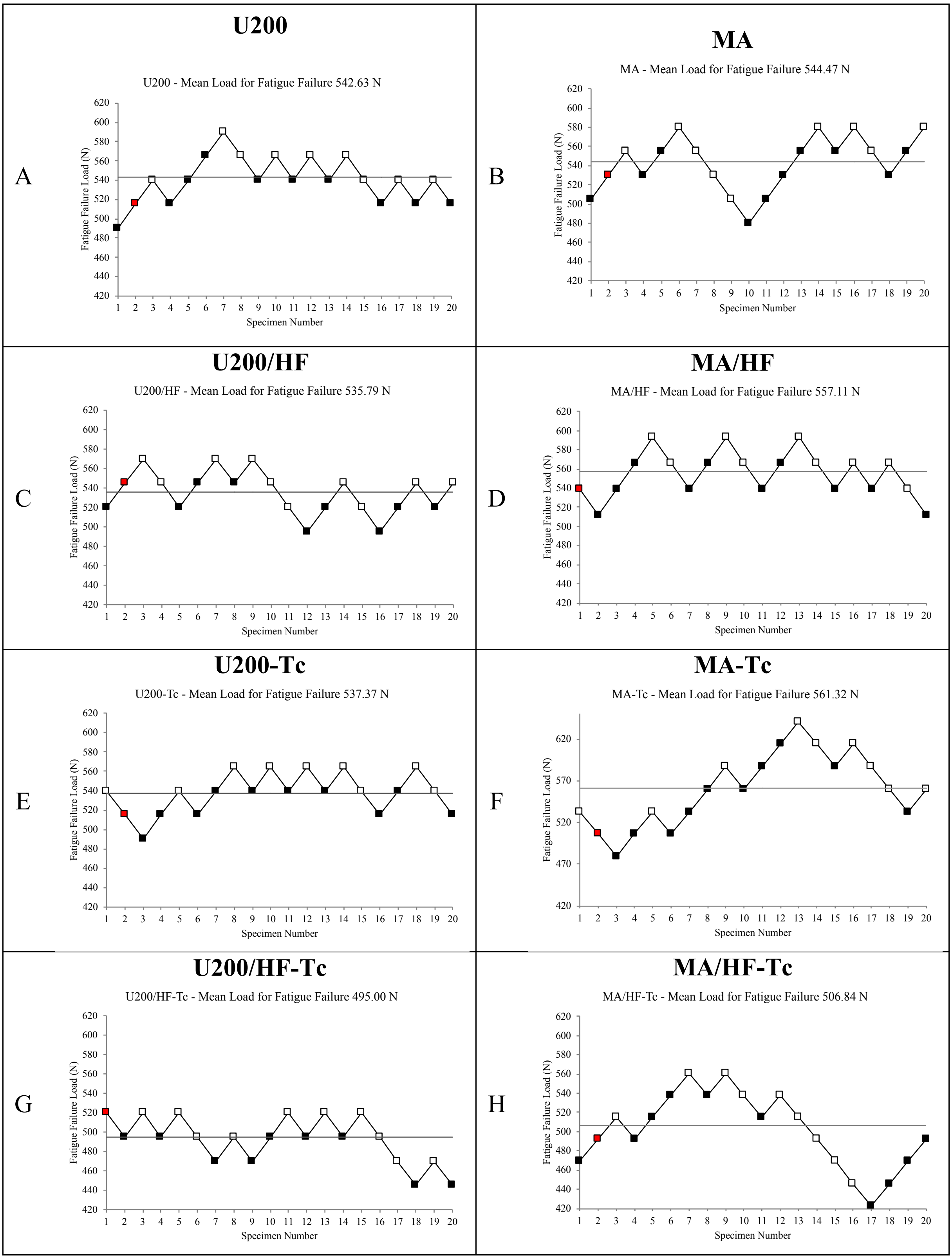
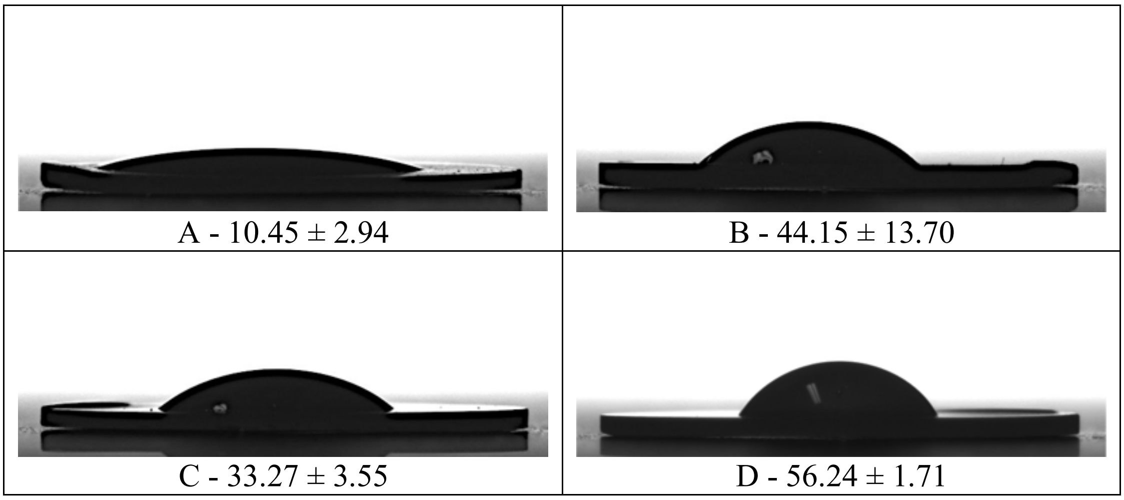


Figure 2.



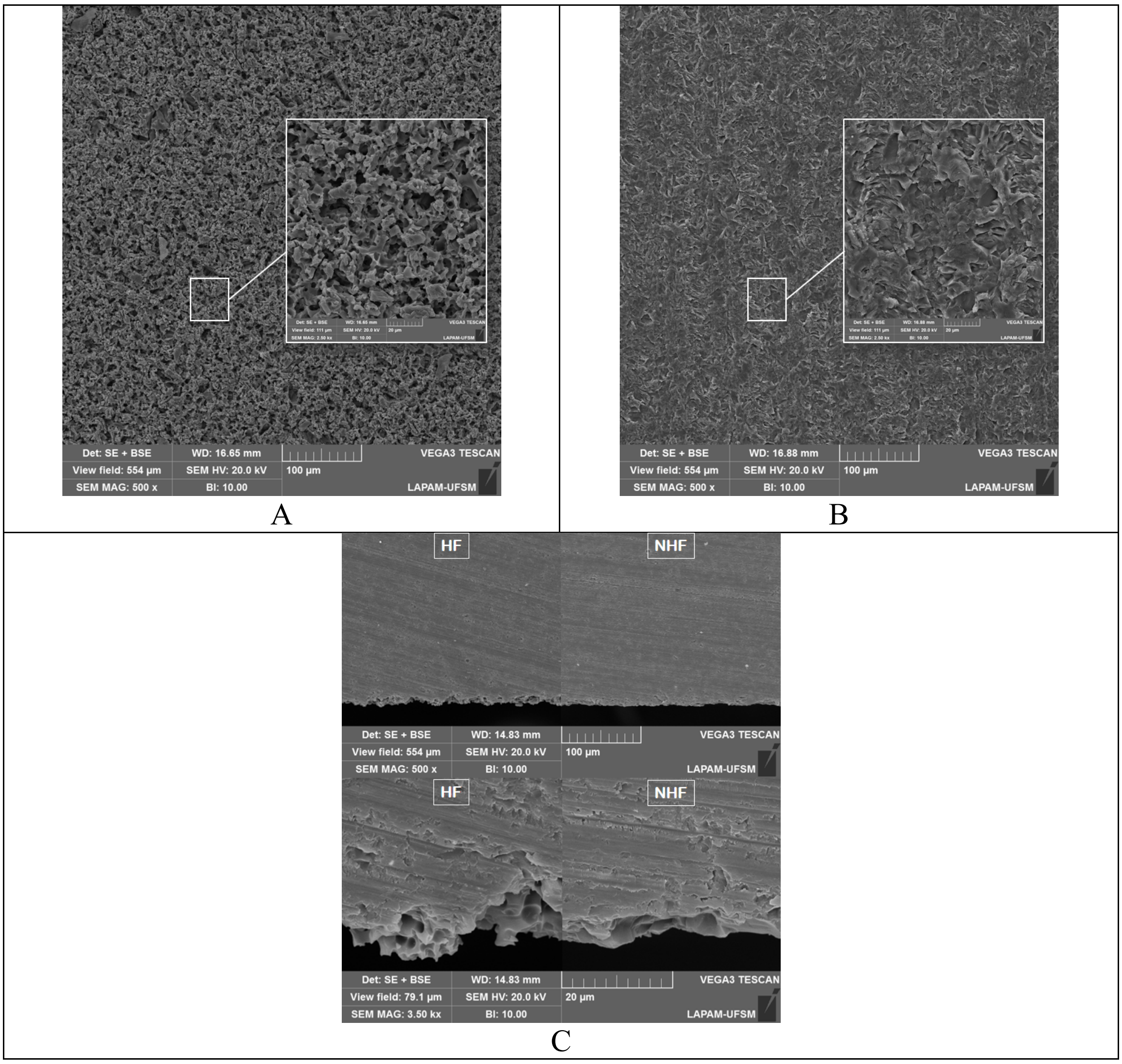
A - 10.45 ± 2.94

B - 44.15 ± 13.70

C - 33.27 ± 3.55

D - 56.24 ± 1.71

Figure 3.

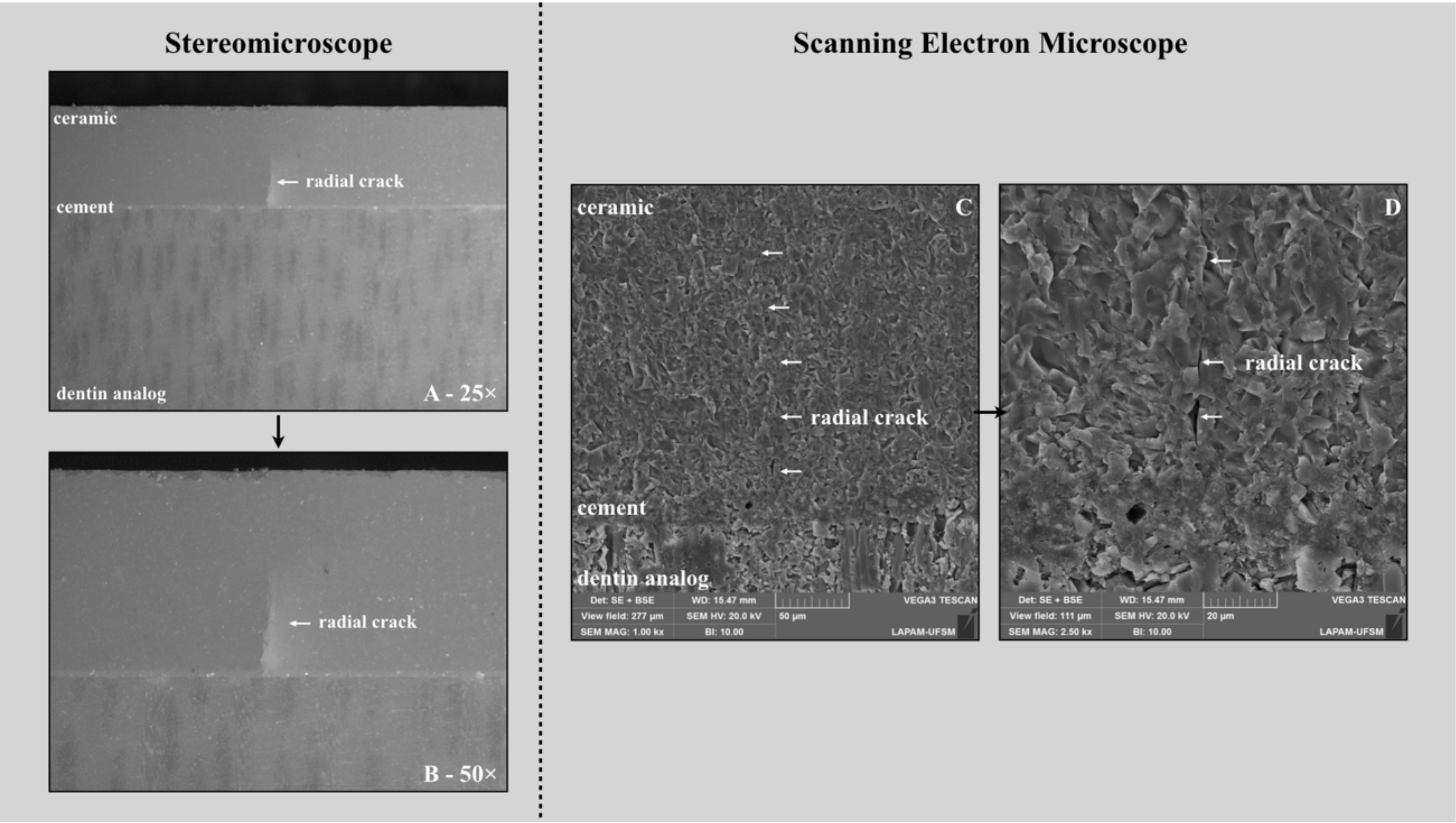


A

B

C

Figure 4.


Stereomicroscope
ceramic
radial crack
cement
dentin analog
A - 25×
radial crack
B - 50×
Scanning Electron Microscope
ceramic
C
radial crack
cement
dentin analog
Det: SE + BSE
WD: 15.47 mm
VEGA3 TESCAN
View field: 277 µm
SEM HV: 20.0 kV
50 µm
SEM MAG: 1.00 kx
BI: 10.00
LAPAM-UFSM
D
radial crack
Det: SE + BSE
WD: 15.47 mm
VEGA3 TESCAN
View field: 111 µm
SEM HV: 20.0 kV
20 µm
SEM MAG: 2.50 kx
BI: 10.00
LAPAM-UFSM

4. ARTIGO 3 - FATIGUE FAILURE LOAD OF CEMENTED SIMPLIFIED MONOLITHIC ZIRCONIA: INFLUENCE OF LUTING SYSTEM AND AGING

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Fatigue failure load of cemented simplified monolithic zirconia: influence of luting system and aging

Short title: Effect of cement and aging on fatigue of a monolithic zirconia.

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ABSTRACT

Fatigue failure load of cemented simplified monolithic zirconia: influence of luting system and aging

Short title: Effect of cement and aging on fatigue of a monolithic zirconia.

Clinical Relevance: The clinical fatigue performance of cemented monolithic zirconia can be influenced by the cement choice. Proper selection of the cement system will enhance long-term fatigue performance.

Abstract

Objective: To evaluate the fatigue failure load of simplified monolithic zirconia specimens cemented to a dentin-like substrate using different luting systems. **Methods:** Disc-shaped ceramic (Zenostar T, 10mm Ø × 0.7mm thick) and dentin-like substrate (10mm Ø × 2.8mm thick) were produced and randomly allocated into eight groups: ‘cement’ (RelyX Luting 2 – glass ionomer cement [Ion]; RelyX U200 – self-adhesive resin cement [Self]; Single Bond Universal+RelyX Ultimate – MDP-containing adhesive + resin cement [MDP-AD + RC]; ED Primer II+Panavia F 2.0 – Primer + MDP-containing resin cement [PR + MDP-RC]) and ‘aging’ (baseline or aged: TC=5°C-55°C/12,000×). Each luting system was used as recommended by the manufacturer. Staircase method (20Hz; 250,000 cycles) was applied for obtaining the fatigue failure loads. Fractographic characteristics were also assessed. **Results:** In baseline, the Ion group presented the lowest fatigue load, although statistically similar to the Self group. The MDP-containing systems presented the highest performance (MDP-AD + RC and PR + MDP-RC), but were also statistically similar to the Self group. Thermocycling influenced the groups differently. After aging, the MDP-AD + RC presented the highest mean, followed by the PR + MDP-RC and Self groups, while the Ion group had the lowest mean. Fractographic analysis depicts all failures as radial cracks starting at the zirconia intaglio surface. **Conclusion:** The luting system with MDP-containing adhesive applied prior to the resin cement presented the highest fatigue failure load after aging, presenting the best predictability of stable performance. Despite this, monolithic zirconia presents high load-bearing capability regardless of the luting agent.

Keywords: Polycrystalline Ceramic. Full-contour. Adhesion. Interface Aging. Fatigue Resistance.

INTRODUCTION

Monolithic zirconia restorations are an alternative to bilayer restorations in posterior teeth since they have not shown chipping or fractures after at least 68 months of clinical use[6,36], and are less invasive for teeth. Long-term clinical data on monolithic zirconia treatments are still scarce[49], however clinical studies with zirconia-based restorations[42,44,45,48,56,59] have shown that fracture and retention loss are their main reasons for failure.

When considering the factor "retention loss" for all-ceramic restorations, one of the aggravating characteristics is their internal relief. Unlike metal-ceramics which have a certain primary friction to the dental substrate when cemented, all-ceramic crowns are not able to withstand such tension without damage. According to Kelly[25], this friction could induce tensile stress capable of generating internal cracks in the restoration, in addition to generating the radial tension effect (*Hoop stress*) caused by dental crowns' cylindrical shape when submitted to load (e.g., luting procedure and chewing cycles). In this sense, the luting material has an important role to compensate for this lack of primary friction and to avoid the restoration debonding. Therefore, the choice of luting material should not be based on clinician preferences, but rather on specific protocols[14].

Several studies have demonstrated the benefits and importance of using techniques that not only promote a micromechanical bond, but also a strong, reliable and long-lasting chemical bond between tooth and ceramic restoration for greater longevity[23,33,38]. The quality of bonding interfaces is one of the major factors responsible for the fracture resistance of all-ceramic dental crowns since bulk fractures originate from defects on the restoration intaglio surface, and these findings have been confirmed through fractographic analyses of clinically failed restorations[27,47] and using finite element predictions[9]. Therefore, it becomes clear that the use of different cement systems can potentially influence the retention and fatigue behavior of such restorations; factors which are largely related to the restoration longevity[3]. Different mechanical and chemical treatments have been recommended to improve the adhesion between resin cements and zirconia. Among them, the most recommended based on strong laboratory and clinical evidence is the air-abrasion with small or medium (< 60 µm) aluminum oxide powders at a moderate pressure (< 2 bar) followed by the application of primers/resin cements that contain phosphate monomers, which provide long-term bonding to zirconia ceramic under the harsh oral conditions[4,15,29,38,51].

Moreover, the ability of luting systems in adequately filling the defects of the ceramic intaglio surface is another concern when cementing these restorations[54]. The fracture strength of ceramic materials is related to the size and number of defects present in their surface[20], and unfilled defects can work as starting points for slow crack growth under constant masticatory stresses, and consequently cause early failure in the restoration[19].

From these standpoints, and particularly regarding the cementation approach affecting the fatigue behavior of the restorative set-up, the present *in vitro* study aimed to investigate the fatigue performance of simplified monolithic zirconia specimens cemented to dentin-like substrate using different luting systems under standardized conditions. The alternative hypotheses tested were: (1) cement type will affect the zirconia fatigue failure load; and (2) aging will deleteriously impact the fatigue failure load of the cemented set-up, regardless of the cement system.

METHODS AND MATERIALS

Study design

A 2nd generation yttria-stablized tetragonal zirconia polycrystal (Y-TZP; 4.5 to 6.0% yttria content; Zenostar T; Wieland Dental, Ivoclar Vivadent; Schaan, Liechtenstein)[50] indicated for framework and monolithic prosthetic restorations was used in the present study. The zirconia discs were cemented on flat dentin-like substrate discs (woven glass-fiber-filled epoxy resin - National Electrical Manufacturers Association [NEMA] grade G10, Accurate Plastics Inc.; New York, USA; E_{G10}= 18.6 GPa – elastic modulus similar to wet dentin E_{dentin}= 18 GPa[28]). The final diameter of the specimens was 10 mm resembling the mean diameter of the occlusal surface of the first permanent molar[16]. The final thickness of the whole restoration set was 3.5 mm (G10 discs= 2.8 mm, zirconia discs= 0.7 mm), being equivalent to the mean thickness between the occlusal surface and the dental pulp chamber roof[22,52].

Production of specimens

Y-TZP ceramic discs

Zenostar T discs (98.5 mm Ø × 16 mm in thick) were manually cut into small blocks (12 mm × 12 mm × 16 mm) with a diamond disc coupled to a handpiece attached to an electric motor (Perfecta LA 623T, 1,000 a 40,000 rpm - W&H, Bürmoos, Austria). Next, metallic rings (Ø=12 mm) were glued to the parallel surfaces of the small blocks to guide the grinding in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) with #600 grit silicon carbide papers (SiC) and water-cooling to obtain zirconia cylinders with 12 mm of diameter.

Then, 0.94 mm thick slices were obtained by cutting under water-cooling with a diamond blade (Buehler-Series 15LC Diamond; Buehler; Lake Bluff, USA) in a precision cutting machine (Isomet 1000, Buehler), resulting in 200 discs. The discs were manually polished on both sides with SiC papers (#600 and #1200 grit) to obtain a smooth surface, free from defects and with a final thickness of 0.86 mm. They were subsequently cleaned (ultrasonic bath with distilled water for 10 min) and dried, and then sintered in a specific furnace (Heating rate of 600°C/h; Temperature 1 of 900°C with a holding time of 0.5h; Heating rate of 200°C/h; and Temperature 2 of 1450°C with a holding time of 2h; VITA Zyrcomat 6000 MS, Vita Zahnfabrik, Bad Säckingen, Germany), followed by ultrasonic cleaning in 78% isopropyl alcohol for 5 min. The final dimensions of the zirconia discs were 10.0 mm in diameter and 0.7 (±0.02) mm in thickness.

Dentin-like substrate discs

NEMA G10 round rods (±250 mm length × 12.7 mm Ø) had their diameters reduced to 10 mm and then sliced in 3.0 mm thick discs by the methodology previously described for the zirconia discs. After cutting, the discs were polished with SiC papers (#400- and #600-grit) until a final thickness of 2.8 mm, followed by ultrasonic cleaning in 78% isopropyl alcohol for 5 min.

Luting procedure – zirconia/dentin-like substrate

The intaglio surface of all zirconia discs was air-abraded for 10 s with aluminum oxide particles (Al_2O_3; 45 μm particles size) with oscillatory movements and a perpendicular angulation (90°) between the device tip and the specimen surface at a distance of 10 mm and at 2.8 bars of pressure. Next, the specimens were ultrasonically cleaned in distilled water for 5 min. All the dentin-like substrate discs were etched by 10% hydrofluoric acid for 1 min (HF etching), rinsed for 30 s, ultrasonically cleaned in distilled water for 5 min and air-dried. After, the specimens (zirconia and dentin-like substrate discs) were randomly (www.randomizer.org) allocated into 8 groups (n=25) according to the study factors (cement and aging) (Table 1). The primers for each cement system were applied to the disc surfaces and cements were handled and applied according to the manufacturers' instructions, as explained in details below:

- *RelyX Luting 2* (3M ESPE; St. Paul, USA) - *Ion and Ion/TC*

Zirconia – after air-abrasion and cleaning (above mentioned), the specimens were vigorously air-dried.

Epoxy resin – After HF etching, a silane-coupling agent (RelyX Ceramic Primer; 3M ESPE)[28] was applied for 5 s, and gentle air-dried.

- *RelyX U200* (3M ESPE) - *Self and Self/TC*

Zirconia – after air-abrasion and cleaning (above mentioned), the specimens were vigorously air-dried.

Epoxy resin – After HF etching, a silane-coupling agent (RelyX Ceramic Primer; 3M ESPE)[28] was applied for 5 s, and gentle air-dried.

- *PANAVIA™ F2.0* (Kuraray Noritake Dental Inc.; Tokyo, Japan) – *PR + MDP-RC and PR + MDP-RC/TC*

Zirconia – after air-abrasion and cleaning (above mentioned), the specimens were vigorously air-dried.

Epoxy resin – After HF etching, the Panavia system ED Primers II, liquids A and B, were mixed (ratio 1:1) and applied on the surface, the mixture was left to react for 30 s and primer excess was removed by gentle air-drying for 5 s.

- *RelyX Ultimate* (3M ESPE) – *MDP-AD + RC and MDP-AD + RC/TC*

Zirconia – after air-abrasion and cleaning (above mentioned), the specimens were vigorously air-dried. Single Bond Universal Adhesive was applied and left to react for 20 s and the excess was removed by gentle air-drying for 5 s.

Epoxy resin – After HF etching, the Single Bond Universal Adhesive (3M ESPE) was applied as mentioned for the zirconia discs.

After the primers' applications, each cement was mixed according to manufacturers' instructions (1:1 ratio) and applied on the dentin-like substrate disc. The zirconia discs were seated in their respective pairs with a uniform bonding force (250 g weight), the cement excess was removed, and the cement was light-cured (1200 mW/cm^2, 440-480 nm, Radii-cal, SDI; Bayswater, Australia) according to the manufacturer's instructions.

Artificial aging – Thermocycling 'TC'

As illustrated in Table 1, half of the specimens from each cement system underwent 12,000 thermal cycles between two water baths, 5°C and 55°C (30 s dwell time and 4 s transfer time; Ethik Technology – model 521-6D; Vargem Grande Paulista, Brazil), starting 1 day after cementation.

Prior to the fatigue tests, the specimens without thermocycling were stored (distilled water at 37ºC in a laboratory oven; Laboratory Thermo incubator, Model 502 - FANEM, São Paulo, Brazil) for 4 days, while the aged specimens were stored for 1 day before thermocycling, and for 4 days after thermocycling.

Fatigue failure load testing – Staircase Method

The specimens for each group were numbered and randomized (www.random.org) to determine their test sequence. The fatigue tests were executed in an electric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, USA) over a flat steel base and through the Staircase sensitivity method[8]. The cyclic loads (250,000 pulse cycles; 20 Hz frequency; wet testing) were applied to the center of disc surface on the zirconia side by a 40 mm Ø hemispheric stainless-steel piston (Fig. 1)[26,28]. The fatigue test parameters (initial load= ~60% of the mean of load-to-failure tests; and step-size = ~5% of the initial load) were obtained from the mean of the static load-to-failure tests (0.5 mm/min crosshead speed; EMIC DL 2000. São José dos Pinhais, Brazil) of 5 specimens until the specimen's failure (i.e. auditory perception of cracking by a single trained operator, LFG). This procedure was performed for each group. An adhesive tape (110 µm thick) was placed on the zirconia surface to improve stress spreading during load application[41,57], and a polyethylene sheet (10 µm thick) was placed between the piston and the cemented set to reduce contact stress concentration[24], both in order to avoid contact damage (Hertzian's cone cracks).

For the fatigue tests, the first specimen of each group was tested with the initial load (~60% of the mean of the load-to-failure test), and then one step-size (~5% of initial load) was added or subtracted for the next specimen depending on the previous specimen's survival (+1 step) or failure (–1 step) to the predefined cycles (250,000). The test was sequentially performed until a minimum of 15 specimens were tested after the up-and-down method had started, being (according to Collins[8]) enough to achieve an accurate fatigue measurement.

Fractographic analysis

After fatigue testing, the fractured specimens were evaluated in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss; Göttingen, Germany) to determine the crack location. The crack was marked to be cut perpendicularly in two halves in a high-precision diamond saw (Isomet 1000, Buehler). Then, representative specimens were selected for Scanning Electron Microscopy analysis (Secondary Electron Detector (SE), VEGA3 Tescan; Brno-Kohoutovice, Czech Republic) to better describe their failure characteristics. Additional SEM images were taken to analyze the radial crack in two different perspectives; a typical fractographic analysis of a debonded specimen after fracture, and an analysis of a transversal view of the crack, as above mentioned for the specimens that remained bonded after fracture.

Statistical analysis

Two-way analysis of variance (ANOVA - IBM SPSS Statistics Program (v24 for Windows; IBM Corp; $\alpha = 0.05$) was used to determine the influence of the independent variables (cement and thermocycling) and their interaction (cement + thermocycling) on the dependent variable (fatigue failure load).

The mean load for fatigue failure (L_f), standard deviation (SD), and 95% confidence interval (CI) were calculated using the Dixon and Mood method[13], which involves the maximum-likelihood estimation (overlapping confidence intervals) and assumes a normal distribution of the data[8], as described in previous studies[17,39].

RESULTS

Fatigue failure load tests

Based on two-way ANOVA, a statistically significant influence was observed for the cement ($p = 0.000$) and aging factors ($p = 0.000$), and their interaction (cement + aging; $p = 0.000$) on the fatigue failure load data.

The mean monotonic load-to-failure values, the parameters for fatigue tests and results, and the graphics of fatigue survival/failure patterns for each group were described (Table 2 and Fig. 2).

Considering the baseline condition, the Ion cement group had the lowest fatigue load (1530.00^{B}) being statistically equal to the Self group (1570.00^{AB}) and lower than PR + MDP-RC (1847.86^{A}) and MDP-AD + RC (1820.00^{A}). After aging, the MDP-AD + RC system presented higher fatigue values (MDP-AD + RC/TC 1957.50^{a}), followed by PR + MDP-RC/TC (1767.14^{b}), Self/TC (1754.17^{b}) and Ion/TC (1551.67^{c}). Aging had no deleterious effect on fatigue loads (Table 2).

Fractographic analysis

Radial crack was the fracture pattern observed for all groups (Figs. 3 and 4). Figure 4 shows the fractographic characteristics under two perspectives; in a specimen in which the fragments separated after failure (Figs. 4A and 4B), and in a transversal cut of a sectioned specimen that remained cemented after failure (Fig. 4C). No cone-cracks were observed.

DISCUSSION

The present study demonstrated that the luting protocol affects the monolithic zirconia fatigue failure load, confirming the first hypothesis, and that the aging process applied was not enough to jeopardizes the mechanical behavior of the restorative assembly, rejecting the second hypothesis. The study results showed that bonding the air-abraded monolithic zirconia using an MDP-containing universal adhesive plus an adhesive resin cement (MDP-AD + RC system) provided the best long-term fatigue failure load results. Also, the investigated zirconia ceramic (Zenostar T) can endure really high fatigue loads, even in a thin thickness (0.7 mm), thus constituting a conservative dental option for monolithic crowns in the posterior region of the mouth, being able to withstands even the highest biting forces during nocturnal bruxism, which reaches 800 N[37].

In a recent systematic review and meta-analysis, Thammajaruk et al.[55] concluded that mechanical and chemical pre-treatments are determinant on the bond strength to zirconia, particularly when MDP-containing primers are used, both with and without aging. Kern[29] reviewed and compared the best available clinical and laboratory evidence for successful bonding of dental oxide ceramic restorations and concluded that the association of air-abrasion at a moderate pressure with the use of primers and/or resin cements containing a phosphate monomer (MDP) provides long-term durable bonding to zirconia ceramic. The fatigue results of our study corroborate these findings, since better results were achieved when luting the monolithic zirconia with MDP-containing systems (Panavia F 2.0 – PR + MDP-RC and RelyX Ultimate - MDP-AD + RC systems).

The cement choice seems to be a determinant factor in the final retention of zirconia restorations as its chemical composition, viscosity, wetting capability, and mechanical properties may influence in such outcome[14,43]. The cements recommended for luting the zirconia-based ceramic are divided into two main groups: conventional (zinc phosphate and glass ionomer cements) and resinous cements. Due to the hydrophilic properties of conventional cements, they are restricted for cases where the restoration fit is satisfactory, and they require a retentive dental preparation (at least 4 mm in height and convergence angle between 6 and 15 degrees) as they have little or no chemical bonding to the dental substrate and to the ceramic. Adhesive bonding is mainly preferred in case of compromised retention[50].

Luting the zirconia ceramic with the resin-modified glass ionomer cement (RelyX Luting 2) led to the worst fatigue behavior in our study. Campos et al.[7] showed that the stress concentration at the ceramic intaglio surface is much higher when the monolithic zirconia crown is luted using a conventional cement (zinc phosphate cement). That may explain why the resin-modified glass ionomer cement (RelyX Luting 2) led to the worst fatigue behavior in our study. Furthermore, the monomer Bis-GMA present in such cement composition (Table 1) has a great impact in the cement viscosity, increasing it considerably[12], which may have affected the cement penetration in the zirconia surface irregularities, weakening the interface. Other studies have also shown better fatigue results for adhesively-cemented veneered[1] and monolithic[7] zirconia crowns compared to non-adhesively cemented crowns. Similar results were showed for adhesively-cemented feldspathic glass-ceramic crowns[35] that withstand at least twice as much load before failure than non-adhesively cemented ones. In a 16 years follow-up clinical study, Malament & Socransky[34] found that glass ceramic restorations (Dicor ceramic) cemented with resin cements exhibited a significant higher survival rate than those cemented with glass ionomer and zinc phosphate cements.

The in vitro studies should simulate the aging of the materials and of the adhesive interface[3] since the restorations are exposed to different challenges in the mouth (i.e. humidity, variations in temperature and pH)[18]. The aging can degrade the adhesive bonding through some factors, such as the cement stiffness reduction[32], hydrolytic degradation of the materials' polymer matrix by water penetration, and fatigue of the adhesive interface due to the mismatch of linear thermal expansion coefficients (different rates of shrinkage and expansion) between bonded materials during temperature changes[2], thereby affecting long-term success of the restoration[3]. According to Lu et al.[32], aging in water can degrade the bond strength and stiffness (i.e. decrease of the elastic modulus) of cement agents, leading to stress redistribution in the ceramic crown, reducing its load-bearing capacity.

In the present study, we did not observe a negative effect of thermocycling on the assembly fatigue behavior, even following the number of cycles recommended by Andreatta et al.[2] as being deleterious for bond strength values between ceramic material and resin cement. However, Zhao et al.[61] reported that slow thermal cycling is more effective than a fast-changing temperature profile to promote the aging process of the bonding interface in materials with low thermal diffusivity (e.g., zirconia). In this sense, the transfer time used in our study (only 4 s) could explain why thermal cycling did not deleteriously impact the fatigue load. In our previous study[21], the aging process did affect the zirconia fatigue behavior when the zirconia was air-abraded with aluminum oxide (45µm particle) and bonded with the Panavia F2.0 system. That could be explained since the aging process was more aggressive in such study which applied 60 days of storage in distilled water at 37°C additionally to the thermocycling protocol (12,000 thermal cycles 5°C - 55°C), which may have allowed water to penetrate and degrade the bonding interface[2].

Hygroscopic expansion is material dependent and sometimes it can exceed the amount of polymerization shrinkage, over-compensating it and leading to internal expansion stress, endangering the restoration integrity[5]. If a more severe aging protocol were applied in our study (i.e. higher transfer time between temperatures, long-term storage in water and/or higher number of thermal cycles), a deleterious effect might appear, as seen in previous studies where the adhesive interface (ceramic/MDP-containing primer) were not stable after 6 months in water[11] and a decrease in the zirconia fatigue performance was observed after thermocycling (12,000 cycles; 5°C - 55°C) and 2 months stored in water[21].

Indeed, the aging significantly increased the fatigue failure load of the MDP-AD + RC system, and according to de Oyagüe et al.[10], this could be explained by the long carbonyl chain of the acidic functional monomer present in the MDP formulation that is relatively stable to hydrolysis. For Zhao et al.[61], the presence of the methacrylate-modified polyalkenoic acid has a moisture-stabilizing effect on the Single Bond Universal adhesive, which can explain its better behavior when subjected to aging. Furthermore, only the MDP-AD + RC system received application of an adhesive at the zirconia surface, and as adhesives have lower viscosity, they are able to better wet and fill in the ceramic surface irregularities, improving the bond strength and reducing the water penetration at the interface[11]. Still, low viscosity means less filler content and higher shrinkage during polymerization, but when restorative materials absorb water, their dimensions and structural integrity may be affected, and in this case the shrinkage stress of the adhesive may be partially relieved by the water uptake, neutralizing the tensions at the adhesive interface, and better distributing the stress during loading[5], consequently increasing the fatigue failure load of the restorative set-up.

As stated by Zhang et al.[60], a post-failure fractographic analysis can provide valuable guidance to find the fracture origin and other failure characteristics. In our study, we only found radial cracks originating from the ceramic intaglio surface (Figs. 3 and 4), and no surface contact damage (Hertzian cracks) was found[26]. Since radial cracks present in monolithic ceramic crowns can propagate and result in bulk fracture (one of the most common failure modes of all-ceramic restorations)[28], the findings of our study may be clinically relevant.

The present study applied a simplified and standardized model eliminating some testing variabilities, and isolating the factors under study. It also follows some recommendations as wet testing and the use of a hemispheric piston with a minimal diameter (40 mm) to creates clinically sized contacts of 0.5 to 3 mm diameter (clinical wear facet size) at pressures of 5 to 890 MPa when applying realistic average maximum bite forces (100 to 700 N)[26]. However, a uniaxial load was applied without clinical sliding contact, so the mechanical conditions of the oral environment were only partially reproduced[58]. Furthermore, in an attempt to reduce time spent on the fatigue test and without significative changing the zirconia fatigue behavior[17], the load frequency applied (20 Hz) was much higher than a normal chewing frequency (0.94 – 2.17 Hz[40]). Care must be taken when wet-grinding and cutting the zirconia in its green body state since it may affect its densification and consequently its mechanical and optical properties[53,30]. More studies should evaluate this problem and investigate an in-lab methodology for dry-grinding and cutting the zirconia.

Considering the aforementioned aspects and that the clinical success of dental ceramic crowns is multifactorial and difficult to be simulated by in vitro studies, additional laboratory and clinical studies are needed to corroborate our results.

CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn:

- Different luting systems did affect the fatigue failure load of a monolithic zirconia cemented to a dentin-like substrate.
- Monolithic zirconia cemented with MDP-containing primer/cement systems endured higher fatigue loads.
- The aging had no damaging effect on the fatigue failure load of the monolithic zirconia specimens.
- The use of an MDP-containing adhesive associated to a resin cement promoted better fatigue results after aging.

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TABLES CAPTIONS

Table 1 – Study experimental design; Cement Systems: classification, commercial name, brand and Elastic modulus, and general composition; aging: baseline and aged (Thermocycling - TC: 12,000 cycles between 5 ºC and 55 ºC, 30 s dwell time; 4 s transfer time); and group codes.

Table 2 – Mean of monotonic load-to-failure test (n=5); fatigue test parameters: initial load for fatigue tests (~60% of mean monotonic load-to-failure), step-size (~5% of initial load). Fatigue results: mean load for fatigue failure (L_f) (standard deviation - SD) and 95% confidence interval (CI); and percentage of decreasing in load comparing the mean value of monotonic load-to-failure and the mean load for fatigue failure.

FIGURES CAPTIONS

Figure 1 - Fatigue test assembly – schematic drawing of the set and the real hemispheric stainless-steel piston (40 mm Ø) used to apply the load in the center of the specimens' occlusal surface submerged in distilled water.

Figure 2 – Staircase survival and failure patterns for each group; n=20 or a minimum of 15 specimens tested after up-and-down character have been started (red marks). Horizontal lines indicate the mean load for fatigue failure; black marks mean survived specimens; white marks mean failed ones.

Figure 3 - SEM images (250× magnification) of cut representative specimens for each experimental group, showing the fractographic characteristics of fractured specimens. Zirconia is on the top of the images and dentin-like substrate material (G10) in the bottom of the images.

Figure 4 - SEM images showing the typical fractographic characteristics after fatigue failure under two perspectives: Figures A and B show a specimen which ceramic fragments were separated after failure; Figure C shows a transverse cut of a sectioned specimen that remained cemented after failure (radial crack). White arrows point to the site of fracture origin; dashed line indicates the compression curl; white dashed arrows indicate the direction of crack propagation (dcp) (Scherrer et al. - ADM guidance-ceramics[47]).

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Table 1 – Study experimental design; Cement Systems: classification, commercial name, brand and Elastic modulus, and general composition; aging: baseline and aged (Thermocycling - TC: 12,000 cycles between 5 ºC and 55 ºC, 30 s dwell time; 4 s transfer time); and group codes.

CEMENT SYSTEMS			GROUPS	
CLASSIFICATION	**COMMERCIAL NAME (BRAND), ELASTIC MODULUS**	**GENERAL COMPOSITION[*]**	**BASELINE**	**AGED (TC)**
Resin-modified glass ionomer cement (1-step)	RelyX Luting 2 (3M ESPE) *E*= 4 GPa[*]	**Cement** - Paste A: radiopaque FAS glass, proprietary reducing agent for self-cure, HEMA, water, opacifying agent. Paste B: methacrylated polycarboxylic acid, BisGMA, HEMA, water, potassium persulfate, non-reactive zirconia silica filler.	**Ion**	**Ion/TC**
Self-adhesive resin cement (1-step)	RelyX U200 (3M ESPE) *E*= 6.6 GPa[*]	**Cement** - Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives.	**Self**	**Self/TC**
Self-etching primers + **MDP**-containing adhesive resin cement (2-steps)	ED Primer II + Panavia F 2.0 (Kuraray Noritake) *E*= 18.3 GPa[†]	**Cement** - Paste A: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica dl-Camphorquinone, catalysts; Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments. **Primers** – Liquid A: HEMA, 10-MDP, N-methacryloyl-5-aminosalicylic acid, water, accelerators. Liquid B: N-methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators.	**PR + MDP-RC**	**PR + MDP-RC/TC**
MDP-containing universal adhesive + adhesive resin cement (2-steps)	Single Bond Universal + RelyX Ultimate (3M ESPE) *E*= 7.7 GPa[*]	**Cement** – Base paste: methacrylate monomers, radiopaque, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator components, stabilizers, pigments, rheological additives, fluorescence dye, dark cure activator for scotchbond universal adhesive. **Adhesive** – MDP, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane.	**MDP-AD + RC**	**MDP-AD + RC/TC**

Abbreviations: FAS, fluoroaluminosilicate; HEMA, 2-hydroxyethylmethacrylate; BisGMA, bisphenol A glycidyl methacrylate; MDP, methacryloyloxydecyl-dihydrogen-phosphate. [*] Manufacturer's data. [†] Li et al.[32]

Table 2 – Mean of monotonic load-to-failure test (n=5); fatigue test parameters: initial load for fatigue tests (~60% of mean monotonic load-to-failure), step-size (~5% of initial load). Fatigue results: mean load for fatigue failure (L_f) (standard deviation - SD) and 95% confidence interval (CI); and percentage of decreasing in load comparing the mean value of monotonic load-to-failure and the mean load for fatigue failure.

Groups	Mean monotonic load-to-failure (N)	Initial load for fatigue tests (N)	Step-size increment (N)	Mean load for fatigue failure L_f (SD)	95% CI [f]	Load decrease (%)
Ion	1998.55	1200	60	1530.00 (286.32)	1319.23 - 1740.77^{B}	23
Ion/TC	1773.75	1060	50	1551.67 (40.60)	1518.21 - 1585.13^{c}	13
Self	2382.53	1430	70	1570.00 (294.89)	1369.02 - 1770.98^{AB}	34
Self/TC	2181.63	1310	65	1754.17 (122.98)	1661.67 - 1846.67^{b}	20
PR + MDP-RC	2160.00	1300	65	1847.86 (119.10)	1764.04 - 1931.68^{A}	14
PR + MDP-RC/TC	2124.57	1275	65	1767.14 (58.93)	1723.05 - 1811.23^{b}	17
MDP-AD + RC	2172.17	1300	65	1820.00 (55.70)	1763.77 - 1876.23^{A*}	16
MDP-AD + RC/TC	2237.54	1340	65	1957.50 (64.48)	1905.91 - 2009.09^{a*}	13

[f] Statistical analysis for fatigue test - Dixon & Mood statistical method[13] (confidence intervals overlapping): different uppercase letters represent statistically significant difference for different cement systems on baseline; different lowercase letters mean statistical difference for different cement systems after thermocycling; and asterisk (*) represents statistically significant difference between baseline and aged between the same cement system.

FIGURES

Figure 1 - Fatigue test assembly – schematic drawing of the set and the real hemispheric stainless-steel piston (40 mm Ø) used to apply the load in the center of the specimens' occlusal surface submerged in distilled water.

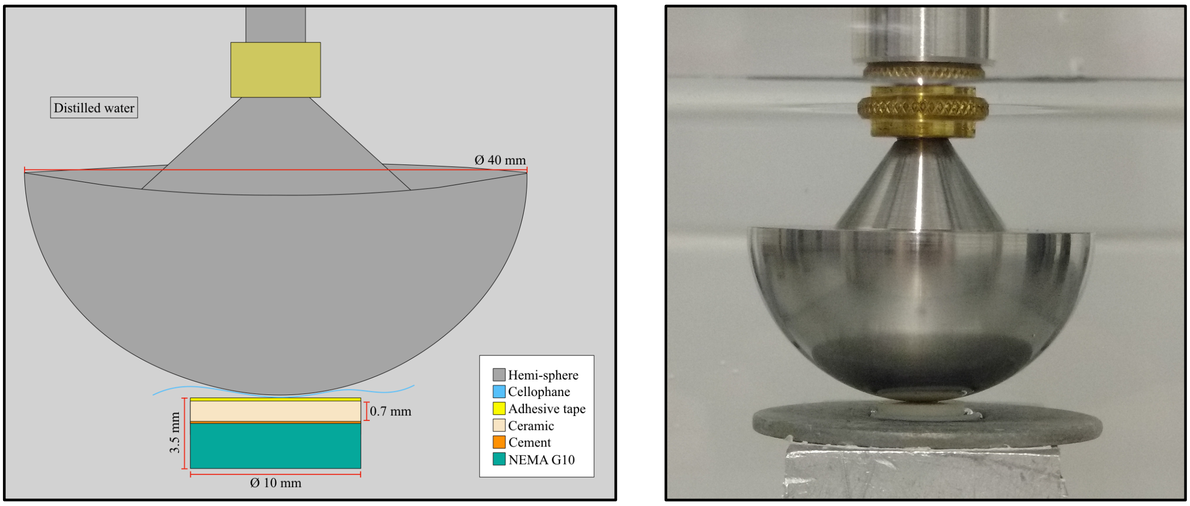


Figure 2 – Staircase survival and failure patterns for each group; n=20 or a minimum of 15 specimens tested after the up-and-down character have been started (red marks). The horizontal dashed lines represent the mean load for fatigue failure of each group; black marks mean survived specimens and white marks mean failed ones. Asterisk (*) indicates a statistically significant difference between baseline and aged groups for the same cement system based on Table 2.

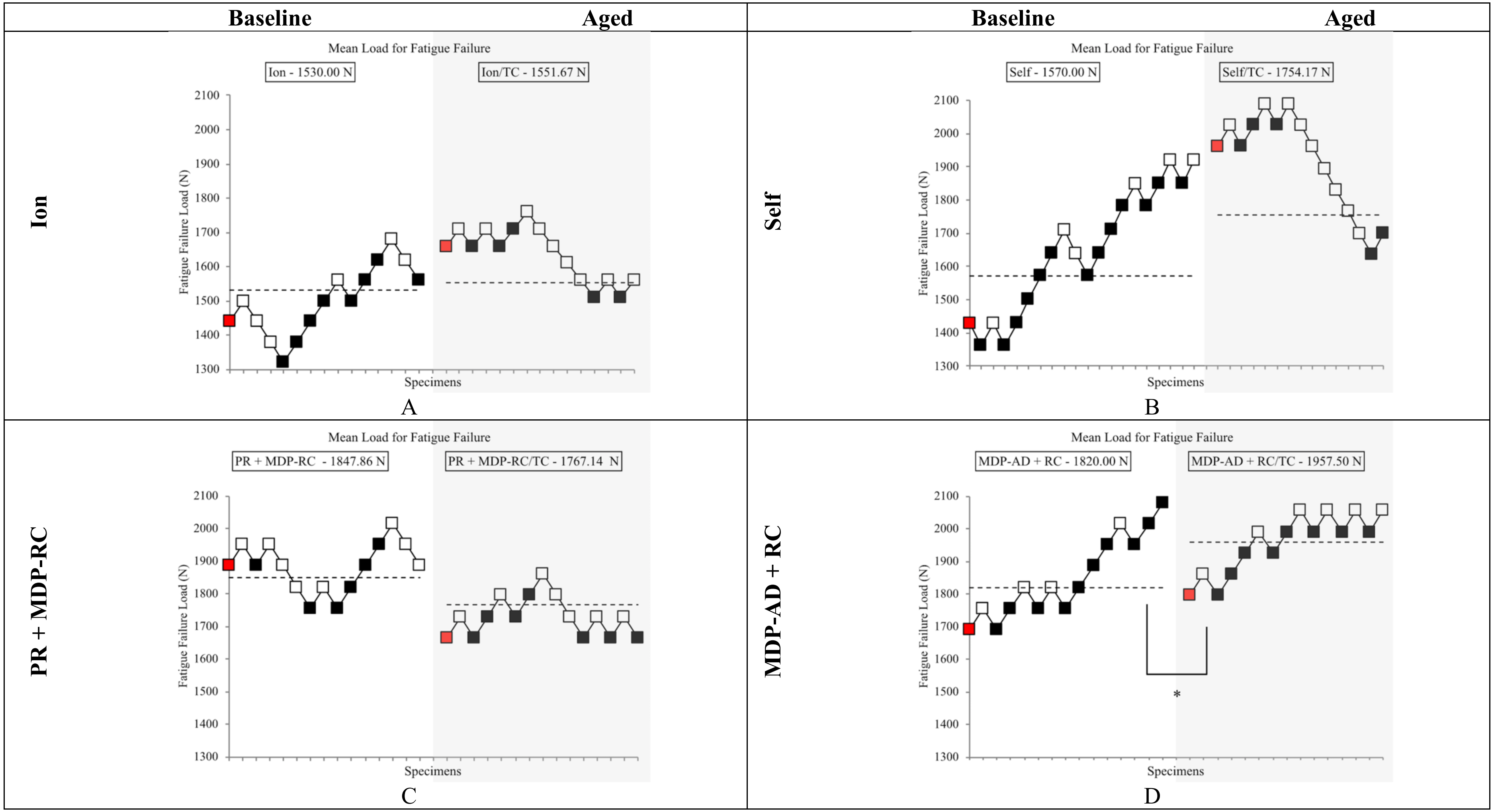
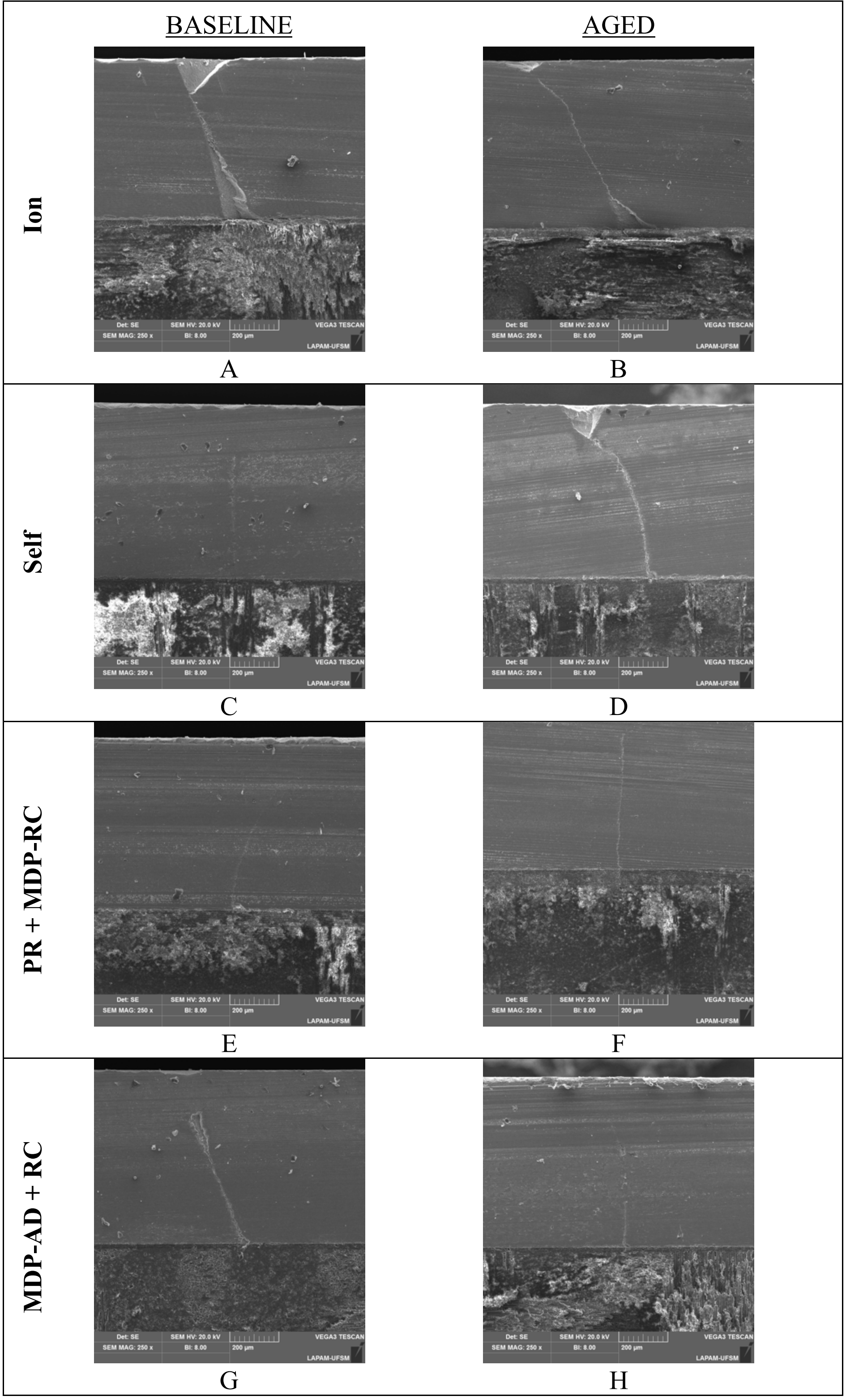


Figure 3 - SEM images (250× magnification) of cut representative specimens for each experimental group, showing the fractographic characteristics of fractured specimens. Zirconia is on the top of the images and dentin-like substrate material (G10) in the bottom of the images.



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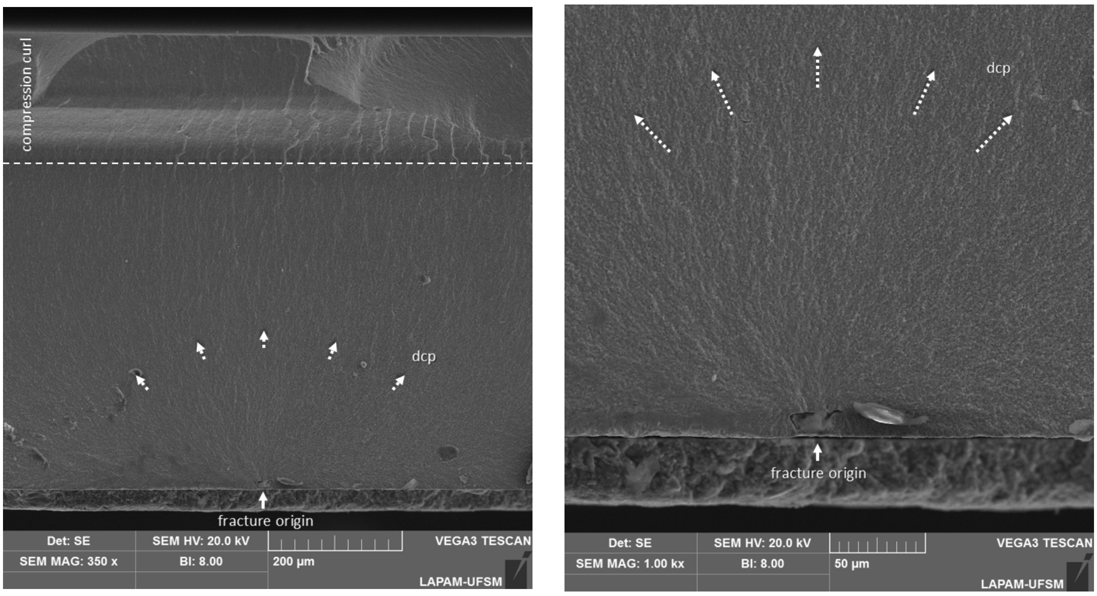
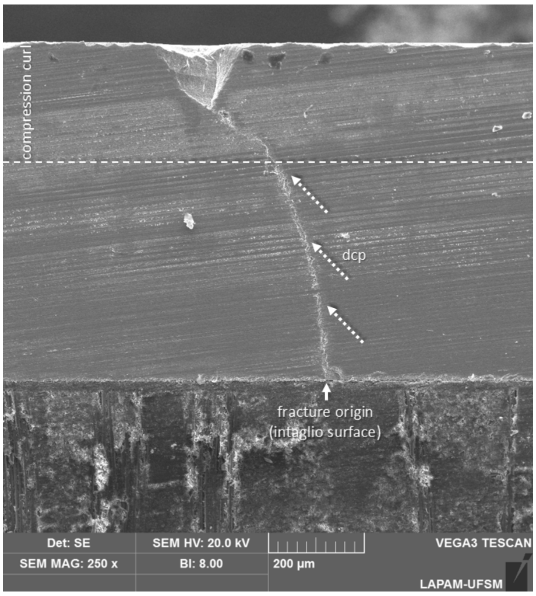
<u>Debonded representative specimen – zirconia ceramic with cement remnants</u>



A – 350×   **B** – 1,000×

<u>Cross-sectioned of a cemented specimen</u>



C – 250×

5. ARTIGO 4 - EFFECT OF ZIRCONIA SURFACE TREATMENT, RESIN CEMENT AND AGING ON THE LOAD-BEARING CAPACITY UNDER FATIGUE OF THIN SIMPLIFIED FULL-CONTOUR Y-TZP RESTORATIONS

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Effect of zirconia surface treatment, resin cement and aging on the load-bearing capacity under fatigue of thin simplified full-contour Y-TZP restorations

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Short title: Surface treatment, cement and aging on fatigue of thin monolithic Y-TZP restorations.

Abstract

This study investigated the effect of zirconia surface treatment (air-abrasion with aluminum oxide or tribochemical silica coating) and aging on the fatigue behavior of thin monolithic Y-TZP (yttria-stablized tetragonal zirconia polycrystal) restorations cemented with 2 types of resin cements, containing or not containing MDP, to a dentin-like substrate. Y-TZP ceramic (Zenostar T, diameter (Ø) 10 mm, 0.7 mm thick) and dentin-like discs (Ø 10 mm, 2.8 mm thick) were assigned into eight groups according to three factors: 'zirconia surface treatment' (aluminum oxide particles air-abrasion 'AO'; or tribochemical silica coating via silica-coated aluminum trioxide particles air-abrasion + silanization 'SC'); 'MDP-containing resin cement' (with: Panavia F2.0, 'MDP'; or without: Multilink Automix, 'nMDP'); and 'aging' (baseline; or aged - AG:12,000 thermal cycles + 60 days water storage). Y-TZP intaglio surface was conditioned and dentin-like substrate was etched with hydrofluoric acid prior to bonding. Aging was performed in half of the specimens before the fatigue testing (Staircase, 20Hz; 250,000 cycles). Fractographic and topographic characteristics were analyzed by stereomicroscope and SEM. Prior to aging, no significant difference was found between the two surface treatments, irrespective to the cement. Samples bonded with resin cement containing MDP had a significant reduction in their fatigue failure load when Y-TZP was air-abraded with aluminum oxide particles and subjected to aging (MDP-AO=2050.71^{A}; MDP-AO/AG=1756.67^{B}). Other studied conditions were not affected by aging. Topographic images revealed a rougher surface for aluminum oxide air-abrasion. Fractography supports all failures as a radial crack starting at the Y-TZP intaglio surface. Bonded thin simplified Y-TZP restorations had a high load-bearing capacity, regardless of the studied factors. The MDP-containing resin cement applied on aluminum oxide air-abraded zirconia surface was not enough to maintain the fatigue performance after aging, while higher stability to aging was achieved by treating with the tribochemical silica coating method. When using an MDP-free resin cement, the surface treatment and the aging did not impact the fatigue performance.

Keywords. Polycrystalline Ceramics. Sandblasting. 10-methacryloyloxy-decyldihydrogen-phosphate. Adhesion. Interface Aging. Fatigue Behavior.

HIGHLIGHTS

- Bonded thin monolithic Y-TZP restorations reached high load-bearing capacity under fatigue.
- For fatigue, tribochemical silica coating led to higher stability to aging when an MDP-containing resin cement was used.

- Using the resin cement without MDP, surface treatment and aging had no impact on the fatigue performance.

1. INTRODUCTION

Full-contour/monolithic dental zirconia has been widely studied and applied as a good alternative for conventional veneered zirconia restorations (Hansen et al., 2018). This approach seems to solve the weak link of zirconia-based restorations, i.e. chipping and delamination of the veneering ceramic (Raigrodski et al., 2006), and provides a less invasive approach by requiring smaller thickness in the tooth preparation, since it allows fabrication of ultrathin monolithic restorations (Souza et al., 2018), reducing the risk of asymptomatic pulp necrosis of teeth after conventional crown preparation (Kontakiotis et al., 2015).

Although clinical studies have not yet demonstrated failures for monolithic zirconia (Moscovitch et al., 2015; Bömicke et al., 2017), the loss of retention/debonding is reported as one of the most common technical complications for zirconia-based restorations (Pjetursson et al., 2007; Örtorp et al., 2012), and they still need a more reliable and long-lasting adhesion to the substrates (Xie et al., 2016). Therefore, bonding to zirconia-based ceramics still remains a challenge because of its non-reactive surface which requires different techniques than the conventional ones used for preparing silica-based ceramics, i.e. hydrofluoric acid etching plus silane agent. There is a wide range of possibilities concerning this issue and alternative methods have become a subject of great interest in trying to optimize zirconia adhesion (Thammajaruk et al., 2018). Air-abrasion, by means of aluminum oxide or with silica-coated aluminum trioxide particles, of the zirconia intaglio surface is the most acceptable mechanical treatment since it cleans the surface and makes it a more reactive surface (micro-retentions and higher surface free energy) (Strasser et al., 2018), and it increases the surface area available for bonding (Moon et al., 2016). Furthermore, the association of micromechanical and chemical pretreatments seems to be the best option for long-term durable bonding and for better clinical and *in vitro* fatigue performance (Kern, 2015; Özcan and Bernaschoni 2015; Anami et al., 2016; Campos et al., 2017; Fraga et al., 2018). In addition, the damaging effect of air-abrasion particles to the mechanical behavior of Y-TZP materials has been debated; some studies found damaging effects (Zhang et al., 2004; Zhang et al., 2006), while recent investigations demonstrated that air-abrasion with alumina or silica-alumina particles has no damaging effect to the mechanical behavior (Scherrer et al., 2011; Özcan et al., 2013; Souza et al., 2013; Oblak et al., 2014; Anami et al., 2016; Campos et al., 2017).

Regarding resin cement features, the introduction of functional monomers (e.g., 10-methacryloyloxy-decyldihydrogen-phosphate – MDP) has increased the bond strength of resin materials to zirconia through chemical bonds (P=O, OH=Zr) (Nagaoka et al., 2017; De-Paula et al., 2017), especially when combined with micromechanical air-abrasion treatment (Kern, 2015), otherwise no durable bonding is achieved (Özcan et al., 2008; May et al., 2010; Özcan et al., 2011; Yang et al., 2010; de Souza et al., 2014). The phosphate ester group of the MDP monomer bonds directly to the oxides of the zirconia surface yields a stable chemical adhesion (Attia and Kern 2011), which can resist the thermal aging and water storage (Kern and Wegner 1998; Lüthy et al., 2006; Kern, 2009). However, the long-term fatigue performance of air-abraded monolithic zirconia ceramic bonded with an MDP-containing resin cement still lacks information (Spitznagel et al., 2018).

In terms of fracture mechanics, dental ceramics tend to fracture when entering the plastic deformation region in the stress-strain curve since they are brittle. Compared to metals, this makes dental ceramic restorations much more susceptible to early fracture under cyclic loads and moisture when under an oral environment's influence which provides the critical factors for the growth of defects present in the ceramic (Wiskott et al., 1995). Mechanical tests that apply cyclical intermittent loading have been used to compare the influence of different bonding protocols on the fatigue behavior of dental ceramics since they can better simulate the clinical fatigue of these materials (Campos et al., 2017; Fraga et al., 2018). According to Zhang et al. [2013], neglecting the use of the mechanical fatigue test can lead to gross overestimation in the prediction of the ceramic survival rates. Moreover, artificial aging often significantly stresses the adhesive interface and degrades the resin/zirconia bonding (Chen et al., 2017), and its application is therefore important to evaluate the influence of adhesion on the mechanical properties of ceramic restorations (Zhang et al., 2013; Arola et al., 2017). Also, controversial reports have emerged concerning the hydrolytic stability when the bonding to zirconia ceramic is considered (Kern and Wegner 1998; Lüthy et al., 2006; Wolfart et al., 2007; Valandro et al., 2007; Kern, 2009; Moura et al., 2017).

Taken altogether and considering the authors' knowledge, some questions need to be answered: what is the fatigue behavior of thin full-contour Y-TZP restorations bonded to dentin-like substrate when its surfaces are air-abraded, the bonding is performed using two distinct resin cements, and the sets are subjected to aging (long-term thermocycling + water storage)?

The objective of this *in vitro* study was therefore to evaluate whether the type of zirconia surface treatment (alumina particles air-abrasion vs. tribochemical silica coating), type of resin cement (with or without MDP monomers), and the aging influence the fatigue failure load of a thin monolithic Y-TZP (yttria-stablized tetragonal zirconia polycrystal) restorations bonded to a dentin-like substrate.

2. MATERIAL AND METHODS

Table 1 shows the details of the products and procedures implemented in this study.

2.1 Preparation of specimens

The specimen's dimensions were based on previous studies (Kelly, 1999; Kelly et al., 2010; Chen et al., 2014) and considering the mean diameter of the occlusal surface of the first permanent molars (diameter (Ø) 10 mm) (Ferrario et al., 1999), and their mean thickness based on the distance between the occlusal surface and the dental pulp chamber roof (total thickness= 3.5 mm; G10 discs= 2.8 mm, zirconia discs= 0.7 mm) (Harris and Hicks, 1998; Sulieman et al., 2005).

2.1.1 Zirconia discs - simplified restorations

Milling discs of an yttria-stablized tetragonal zirconia polycrystal (4.5 to 6.0% content) (Ø 98.5 mm × 16.0 mm in thick) (Y-TZP, Zenostar T, LOT-U20833, Wieland Dental, Ivoclar Vivadent; Schaan, Liechtenstein) were manually cut into small blocks (12×12×16 mm^3) with a diamond disc coupled to a handpiece electric motor (Perfecta LA 623T, 1,000 a 40,000 rpm - W&H, Bürmoos, Austria). Metallic rings (Ø 12 mm) were glued on the parallel surfaces of the blocks to guide the grinding in the polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) to obtain cylinders. The grinding was executed with #600 grit silicon carbide papers (SiC, 3M, Sumare, Brazil) and constant water-cooling. Then, slices of 0.94 mm thick were obtained by cutting under water-cooling with a diamond blade (Buehler-Series 15LC Diamond; Isomet 1000, Buehler). The discs were manually polished on both sides with SiC papers (#600 and #1200 grit) to obtain a smooth surface free of defects with a final thickness of 0.86 mm, and then sintered in a specific furnace (VITA Zyrcomat 6000 MS, Vita Zahnfabrik, Bad Säckingen, Germany) using a standard sintering program for Zenostar T (Heating rate of 600°C/h; Temperature 1 of 900°C with a holding time of 0.5h; Heating rate of 200°C/h; and Temperature 2 of 1450°C with a holding time of 2h) and cleaned in ultrasonic bath with 78% isopropyl alcohol for 5 min. The final dimensions of the zirconia discs were 10 mm in diameter and 0.7 (±0.02) mm in thickness.

2.1.2 Dentin-like discs – G10

The diameter of the woven glass-fiber-filled epoxy resin bars (±250 mm length × Ø 12.7 mm; National Electrical Manufacturers Association – NEMA, grade G10, Accurate Plastics

Inc.; New York, USA) was reduced to 10 mm in the grinding machine (EcoMet/AutoMet 250, Buehler) using SiC papers (#200-, #400- and #600-grit), and then discs of 3.0 mm thick were obtained through the same methodology previously described for the zirconia discs. The discs were polished with SiC papers (#400- and #600-grit) until the final thickness of 2.8 mm, followed by ultrasonic cleaning in 78% isopropyl alcohol for 5 min.

2.2 Cementing procedure – zirconia/ dentin-like substrate

The Y-TZP and G10 discs were randomly (www.randomizer.org) allocated into 8 groups (n=20), as described in Table 1, prior to the surface treatment procedures.

For preparing the intaglio surface of the zirconia restorations, half of the specimens were air-abraded with aluminum oxide particles (AO; 45 µm particles size; aluminum oxide, Polidental, Cotia, Brazil) and the other half with silica-coated aluminum trioxide particles (SC; 30 µm particles size; Cojet Sand, 3M/ESPE, Seefeld, Germany). The air-abrasion, using a micro-etcher (DENTO-PREP microblaster, Ronvig, Daugaard, Denmark), was performed on the zirconia surface for both systems by means of oscillatory movements, at a perpendicular inclination (90°) between the micro-etcher's tip and the specimen at a distance of 10 mm, pressure of 2.8 bar, and for 10 s (Amaral et al., 2008).

After air-abrasion, the SC treated specimens received a light air spray, free of water and oil, to remove the excess dust deposited on the surface, since ultrasonic cleaning is not recommended (Nishigawa et al., 2008). AO-treated specimens were cleaned in an ultrasonic bath with distilled water for 5 min and vigorously air-dried for 20 s. All the dentin-like discs were etched with hydrofluoric acid (HF 10% for 1 min), rinsed for 30 s, ultrasonically cleaned in distilled water for 5 min and air-dried.

The primers for each cement system were applied onto the zirconia and G10 surfaces following the manufacturers' instructions, as described in Table 1, and each cement was handled and applied over the dentin-like disc. The zirconia discs were then seated in their respective G10 pairs and the cement was allowed to flow under constant pressure of 250 g, followed by cement excess removal and light-activation (1200 mW/cm^2, 440 - 480 nm, Radii-cal, SDI Limited; Bayswater, Australia) with five exposures of 20 s each (0°, 90°, 180°, 270° from lateral part of the bonded discs, and top from the occlusal side/ceramic). The samples were kept untouched for 5 min and then stored in distilled water at 37 °C for 4 days (non-aged samples) or aged, as described below, for further fatigue tests.

2.3 Artificial Aging 'AG' – thermocycling + water storage

After cementation, these samples were stored for 1 day in distilled water at 37 °C and underwent intermittent 12,000 thermal-cycles in water with an alternating temperature of 5–55

°C with 30 s dwell time at each temperature and 4 s of transfer time (Andreatta Filho et al., 2005) (Ethik Technology Limited – model 521-6D; Vargem Grande Paulista, Brazil). After thermocycling, the cemented assemblies were stored in a sealed vessel submerged in distilled water at 37 °C for 60 days in a laboratory oven (Laboratory Thermo incubator, Model 502 - FANEM, São Paulo, Brazil).

2.4 Fatigue failure load testing – Staircase Method

The Staircase fatigue test (Collins, 1993) was executed in an electric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, USA) by applying cyclic loads (250,000 pulse cycles; 20 Hz frequency; wet testing) in the center of the zirconia discs (on occlusal side of the restoration) using a 40 mm diameter hemispheric stainless-steel piston (Fig. 1; Kelly et al 1999; Kelly et al 2010; Pereira et al., 2016). The parameters for the fatigue test (initial load= ~60% of the mean of load-to-failure tests; and step-size= ~5% of the initial load) were based on the mean results of the static load-to-failure test performed in 5 additional specimens per group with a crosshead speed of 0.5 mm/min (EMIC DL 2000; São José dos Pinhais, Brazil). The first specimen of each group was tested with the initial load and one step-size was then added or subtracted for the next specimen (~5% of the initial load), depending on the survival or failure of the previous specimen to the predetermined cycles. The test was sequentially performed until a minimum of 15 specimens were tested after the up-and-down character had started. According to Collins (1993), 15 specimens are enough to achieve an accurate fatigue measurement.

An adhesive tape (110 μm thick) was placed on the zirconia surface to improve stress spreading during load application (Wachtman et al., 1972) and a sheet of polyethylene (10 μm thick) was placed between the piston and the cemented set to reduce contact stress concentration (ISO 6872:2015), both in order to avoid contact damage (Hertzian cone-cracks).

2.5 Topographic analysis

Additional ceramic specimens (n=3) were prepared for surface topography evaluation before and after air-abrasion with aluminum oxide or silica-coated aluminum trioxide particles using Scanning Electron Microscopy (Secondary Electron detector – SE; VEGA3 Tescan; Brno-Kohoutovice, Czech Republic). The specimens were first sputtered with a gold-palladium alloy to be analyzed under SEM at 2,000 and 10,000× magnification.

2.6 Fractographic and cement thickness analyses

After fatigue testing, the failed specimens were evaluated in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss; Göttingen, Germany) to determine their crack growth path. The crack was marked to be cut perpendicularly in two halves, closest to the central area where the highest tensile stress concentrates (below the load application area), in a high-precision diamond saw under water-cooling (Isomet 1000, Buehler). Then, representative specimens were selected, cleaned ultrasonically in distilled water for 10 min and subsequently sputtered with a gold-palladium alloy to be analyzed on SEM at 250, 350, and 1,000 times of magnification (VEGA3 Tescan). The cement thickness was measured for 4 specimens per cement and 3 measures per specimen during SEM fractographic analysis at 500× of magnification (VEGA3 Tescan).

2.7 Statistical analysis

Assuming a normal distribution of the data (Collins, 1993), the mean load for fatigue failure (L_f), standard deviation (SD), and 95% confidence interval (CI) were calculated using the Dixon and Mood method (1948) based on the maximum-likelihood estimation, and the overlapping confidence intervals were compared. This method is based on the least frequent event (failure vs. survival) and is well described in a previous study (Pereira et al., 2016).

3. RESULTS

3.1 Fatigue failure load tests

No significant difference was found between the two zirconia surface treatment methods regardless of the cement used (Table 2 and Fig. 2). There was no significant difference between the cements with or without MDP (Table 2 and Fig. 2). The specimens air-abraded with aluminum oxide particles and bonded with the MDP-containing resin cement (Panavia F2.0) presented a significant reduction in the fatigue failure load after aging (MDP-AO = 2050.71 N > MDP-AO/AG = 1756.67 N) (Table 2 and Fig. 2).

3.2 Topographic analysis

SEM micrographs revealed that both air-abrasion systems (AO and SC) created a more irregular topography compared to the baseline specimen, presenting scratches and grooves on the zirconia surface. It was still visually evident that air-abrasion with aluminum oxide particles created sharper defects and appeared to promote a rougher surface compared to silica-coated aluminum trioxide particles (Fig. 3).

3.3 Fractographic and cement thickness analyses

Fractography revealed that the failure pattern of all the samples was the radial crack, having the failure origin at the ceramic intaglio surface in the region underneath the load application (Fig. 4). No cone-cracks were observed.

With regard to measuring cement thickness (Fig. 5), the Panavia F2.0 cement presented a thicker film (mean = 59.29 ± 4.38 μm) than the Multilink Automix (mean = 28.20 ± 4.34 μm).

4. DISCUSSION

Many factors can contribute to the development of ceramic fracture (substrate, ceramic thickness, cement type and thickness, bonding procedures, mechanical test, ceramic surface treatment, etc.) and the study design should result in failures which are similar to the clinical ones (Kelly, 1999; Kelly et al., 2010).

The clinical performance of a ceramic restoration is based on its bonding quality and mechanical properties. Zirconia is the strongest of dental ceramics, but it is still lacking in terms of adhesion (Thammajaruk et al., 2018). The bond strength to zirconia depends on its surface free energy and on the bond system wettability. Air-abrasion is able to clean, activate and increase the zirconia surface available for adhesion (Strasser et al., 2018; Moon et al., 2016) and the bifunctional phosphate monomer 10-methacryloyloxy-decyldihydrogen-phosphate (MDP) chemically bonds to the zirconia surface oxides and to the resin cement (Kern, 2009). In this sense, this association of mechanical and chemical treatments seems to provide better long-term results (Kern, 2015; Özcan and Bernaschoni, 2015).

Concomitantly, the defects on the ceramic restoration's intaglio surface, inherent from their processing, milling or even from pre-cementation surface treatments (e.g., acid etching, grinding, air-abrasion), might work as starting points for the crack propagation (Khan et al., 2017). Air-abrasion is the most indicated mechanical treatment for the zirconia surface to improve its bond strength, and it can be applied using different protocols, varying the type and size of particles, the pressure, distance and time of its application (Özcan et al., 2013; Souza et al., 2013; Kern, 2015; Özcan and Bernaschoni, 2015). The fatigue results in the baseline groups (without aging) of our study, apart from zirconia surface treatment and resin cement (Table 2), were statistically similar, even though the micromorphological evaluations showed a rougher surface (more irregular) when the AO system was applied (Fig. 3). Nevertheless, Sato et al. (2008) found greater roughness in the zirconia air-abraded with SC system than with AO, which can be explained by the larger particle size for the SC group (125 μm) compared to the AO (70 μm) used by them.

For the damaging effect, care must be taken with regard to defects created by the air-abrasion and some concerns have been expressed about this, since these defects are a source of stress concentration and failure will consequently occur at a lower stress (Khan et al., 2017; Moon et al., 2016; Spazzin et al., 2017). But if these defects are entirely filled by the bonding system (healing effect), the flaw population is drastically reduced and the load for the ceramic to fail will need to be higher (Spazzin et al., 2016). Also, the study by Yang et al. (2010) showed that using a low-pressure air-abrasion, can be enough to produce durable bonding when proper primers containing the monomer MDP are applied, in addition to producing a surface with less defects. Campos et al. (2017) and Anami et al. (2016) showed that the fatigue load of bonded monolithic zirconia crowns was not affected by the air-abrasion of the inner surface of the crowns. Another factor that may affect the strength of Y-TZP ceramics after air-abrasion is the tetragonal to monoclinic phase transformation, resulting in the transformation toughening mechanism which creates a surface with compressive stresses. As described by Scherrer et al. (2011), fatigue performance improvements could be found when air-abrading was performed on the zirconia surface. As we did not test a control group without air-abrasion, we could not conclude if the air-abrasion jeopardized or improved the zirconia fatigue performance from our finding, although we did find high load-bearing capacity for Y-TZP restorations.

The results of the present study showed no need for using an MDP-containing resin cement (Panavia F2.0) in terms of the fatigue load standpoint, since it did not promote better results than the MDP-free resin cement (Multilink Automix) (Table 2). We have to consider that besides their different composition, cements have distinct physical and mechanical properties which may play an important role in the bonding capacity to Y-TZP ceramics and could impact the restoration strength (Spazzin et al., 2017). Different parameters such as particle size, viscosity, filler type and content, filler concentration and setting reactions may affect the physical properties and the film thickness of the resin cements (Meşe et al., 2008). The film thickness of the Panavia F2.0 (mean= 59.29 ± 4.38 μm) was two-times thicker than the Multilink Automix (mean= 28.20 ± 4.34 μm), indicating lower wettability (i.e. higher viscosity) when submitted to the same methodology during the cementation process (*Session 2.2*) (Fig. 5). Also, even though we tested simplified restorations (which means that caution must be taken in interpretreting the results), our findings pointed out that the Panavia F2.0 cement did not comply with the requirements of the International Organization for Standardization (ISO) 4049:2009 for resin-based materials (ISO 4049:2009) regarding the film thickness of luting materials, i.e. cement thickness shall be no greater than 50 μm, ultimately being more prone to the hydrolytic degradation during aging processes.

Although the reason for lower fatigue load results for the MDP-AO/AG group remains unclear, the authors suggest two explanations for this: i) the adhesion becomes very weak after aging (Özcan et al., 2008) and therefore reduces the fatigue load of the assembly; ii) based on the studies of da Silva et al. (2014) and de Souza et al. (2014), this reduction might have been caused by the higher viscosity (consequently lower wettability) of this cement (MDP-containing cement - Panavia F2.0) which leads to a lower capacity to fill the defects created by the AO air-abrasion system. Both processes probably facilitated the water from the aging process (thermocycling + storage) to more quickly infiltrate into the interface, breaking the adhesive chemical bonds, resulting in hydrolytic degradation of the cement (Blatz et al., 2004; May et al., 2010), and ultimately in a lower load for the fatigue failure to occur (Table 2; Fig. 2). Regarding the wettability of the bonding system to the zirconia ceramic surface, it can be optimized by the use of different coupling agents such as silane and ceramic primers, and by silica deposition via air-abrasion with silica-coated Al_2O_3 (da Silva et al., 2014). So, the higher viscosity of the Panavia cement was not a problem for the group treated with tribochemical silica coating (Cojet + ceramic primer) even after aging, since the silica layer encrusted onto the zirconia surface. Also, the application of the ceramic primer (Clearfill Ceramic Primer), which has a much lower viscosity and contains a silane coupling agent which strongly adheres to the silica-based materials (i.e. silica layer from the Cojet system), in addition to the proven monomer MDP for bonding to metal oxides, and therefore provided a better wettability to the zirconia surface (Yang et al., 2010), improving the adhesion and its durability (May et al., 2010; Inokoshi et al., 2013), contributing to failure load improvements.

This *in vitro* study presents inherent limitations that must be considered, such as: the use of a non-machined (CAD/CAM) surface, only axial load application, and the use of specimens with simplified flat shape. In lab (i.e. without CAD/CAM) specimens' production with a simplified flat design optimizes the material used, reducing costs. The simplified shape approach also enables precision and reproducibility of the factors under study. This design was based on the research of Kelly et al. [2010] who validated this test assembly in terms of size and shape of specimens and the use of a dentin-like material as substrate. Therefore, a dentin-like substrate with an elastic modulus similar to human dentin (E_{G10}= 18.6 GPa and E_{dentin}= 18 GPa; Kelly et al., 2010) was used in this investigation. Moreover, a 40 mm diameter hemispheric piston was used for applying the load, which resembles the contact area between the molar teeth during chewing and clenching (Kelly et al., 2010), and creates the same failure characteristics of clinically failed ceramic restorations (Kelly et al., 1990), i.e. the radial crack starting at the ceramic intaglio surface showed in the fractographic analysis (Fig. 4). Additionally, pre-sintered zirconia was wet-ground during its preparation, which has been proved to be deleterious for the material densification and consequently for its final strength and translucency (Anand et al., 2018; Kwon et al., 2018). Therefore, care should be taken during the milling of pre-sintered zirconia, and additional studies should evaluate an in vitro methodology for the application of dry-ground and the effects of wet-ground in the mechanical properties of the material.

Our findings suggest that the bond capacity (healing effect) of the tested bonding system appear to play a determinant role in fatigue behavior improvements of thin full-contour Y-TZP restorations independently of the presence or not of monomer MDP in the resin cements. Thus, from the authors' standpoint, it is crucial to choose a bonding system with already-proven high and stable bond strength to zirconia surface for load-bearing capacity improvements of these restorations. Further *in vitro* and clinical studies should evaluate this subject to corroborate our findings.

5. CONCLUSIONS

- Bonded simplified Y-TZP restorations had high load-bearing capacity under fatigue, regardless of zirconia surface treatment, resin cement and aging factors.
- The MDP-containing resin cement applied on aluminum oxide air-abraded zirconia surface was not enough to maintain the fatigue performance after aging, and higher stability to aging was achieved applying the tribochemical silica coating as surface treatment.
- The surface treatment and the aging did not impact the fatigue performance when using the MDP-free resin cement.
- A surface treatment which does not induce further surface damage would be preferable to the long-term success of bonded ceramic restorations.

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TABLES

Table 1 – Study design; Cement Systems: classification, commercial name, manufacturer, elastic modulus, LOT, and chemical composition; Zirconia and dentin-like substrate pretreatments; and Baseline and Aged group codes.

CEMENT SYSTEMS			PRETREATMENTS		GROUPS	
Classification	**Commercial name (manufacturer) Elastic modulus LOT**	**Chemical composition**	**Dentin-like substrate - G10**	**Zirconia** * aluminum oxide '**AO**' or Silica-coated aluminum trioxide '**SC**' + primers	**BASELINE**	**AGED ****
MDP-containing adhesive resin cement **(MDP)**	Panavia F 2.0 - Kuraray Noritake Dental Inc; Okayama, Japan. *E*= 18.3 GPa[†] LOT-330158	**Cement** - Paste A: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica dl-Camphorquinone, catalysts; Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments. Inorganic filler content = 78 wt%[1]	Etched with 10% HF for 1 min, rinsed for 30 s, ultrasonically cleaned (distilled water, 5 min) and air-dried. Mixed Panavia system ED Primers II A/B was applied in the surface; the mixture was left to react for 30 s; primer excess was removed by gentle air-drying for 5 s.	Air-abrasion with AO; ultrasonically cleaned (distilled water, 5 min) and air-dried.	**MDP-AO**	**MDP-AO/AG**
				Air-abrasion with SC; light air-sprayed to remove particles in excess. Clearfill Ceramic Primer (MDP/silane-based primer) was applied at the zirconia surface for 5 s and the excess was removed by gentle air-drying for 5 s.	**MDP-SC**	**MDP-SC/AG**
MDP-free self-etching resin cement **(nMDP)**	Multilink Automix - Ivoclar Vivadent Inc; Schaan, Liechtenstein. *E*= 6.2 GPa[¥] LOT-V08514	**Cement** - Base paste: BisGMA, HEMA, 2-dimethylaminoethyl methacrylate, ethyoxylated bisphenol A dimethacrylate, ytterbium trifluoride; Catalyst paste: UDMA, HEMA, ethyoxylated bisphenol A dimethacrylate, dibenzoyl peroxide, ytterbium trifluoride. Inorganic filler content = 73.4 wt% (40 vol%)[2]	Etched with 10% HF for 1 min, rinsed for 30 s, ultrasonically cleaned (distilled water, 5 min) and air-dried. Mixed Multilink N Primers A/B was scrubbed in the surface with a microbrush for 30 s; primer excess was removed by gentle air-drying for 5 s.	Air-abrasion with AO; ultrasonically cleaned (distilled water, 5 min) and air-dried.	**nMDP-AO**	**nMDP-AO/AG**
				Air-abrasion with SC; light air-sprayed to remove particles in excess. Monobond Plus (multiple bond promoter) was applied at the zirconia surface for 5 s and left to react for 60 s; the excess was removed by gentle air-drying for 5 s.	**nMDP-SC**	**nMDP-SC/AG**

[†] Li et al., 2006. [¥] Manufacturer's data for dual-curing system.

[*] Air-abrasion – Oscillatory movement, perpendicular to the specimen surface (90º), at 10 mm distance, for 10 s, 2.8 bars pressure (Aluminum oxide - 45 µm particle size; and Silica-coated aluminum trioxide - 30 µm particle size).

[**] Aging – Thermocycling 12,000 cycles between 5 and 55 ºC (30 s of dwell time; transfer time of 4 s) + 60 days stored in distilled water, 37 ºC.

Abbreviations: HEMA, 2-hydroxyethyl-methacrylate; UDMA, urethane dimethacrylate; BisGMA, bisphenol A glycidyl methacrylate; MDP, methacryloyloxydecyl-dihydrogen-phosphate; HF, hydrofluoric acid.

[1] silanated silica, silanated colloidal silica, silanated barium glass; [2] barium glass, ytterbium trifluoride, spheroid mixed oxide.

Table 2 – Mean of monotonic load-to-failure test (n=5); fatigue test parameters: initial load for fatigue tests (~60% of mean monotonic load-to-failure), step-size (~5% of initial load). Fatigue results: mean load for fatigue failure (L_f) (standard deviation - SD) and 95% confidence interval (CI); and percentage of decrease in load comparing the mean value of monotonic load-to-failure and the mean load for fatigue failure.

Groups	Mean monotonic load-to-failure (N)	Initial load for fatigue tests (N)	Step-size increment (N)	Mean load for fatigue failure L_f (SD) (N) ^{f}	95% CI	Load decrease (%)
MDP-AO	2241.04	1345	65	2050.71 (34.45) *^{Aa}	2037.48 – 2063.95	8.49
MDP-SC	2152.95	1290	65	2015.83 (52.78) ^{Aa}	1972.33 – 2059.33	6.37
MDP-AO/AG	2324.83	1395	70	1756.67 (56.84) *^{Ba}	1705.73 – 1807.60	24.44
MDP-SC/AG	2339.76	1405	70	2011.67 (141.89) ^{Aa}	1891.32 – 2132.01	14.02
nMDP-AO	2087.85	1250	60	1960.00 (178.32) ^{Aa}	1825.88 – 2094.12	6.12
nMDP-SC	2200.50	1320	65	1991.67 (131.75) ^{Aa}	1892.57 – 2090.77	9.52
nMDP-AO/AG	2212.66	1325	65	1884.00 (112.57) ^{Aa}	1790.27 – 1977.73	14.85
nMDP-SC/AG	2116.63	1270	65	1882.86 (89.01) ^{Aa}	1822.19 – 1943.52	11.08

^{f} Statistical analysis for fatigue test - Dixon & Mood method (1948) (confidence intervals overlapping): Different uppercase letters represent statistically significant difference, considering each cement system individually; Different lowercase letters represent statistical difference for paired conditions between both cements (MDP-AO vs. nMDP-AO; MDP-SC vs. nMDP-SC; MDP-AO/AG vs. nMDP-AO/AG; MDP-SC/AG vs. nMDP-SC/AG).

* Significant statistical difference.

FIGURES

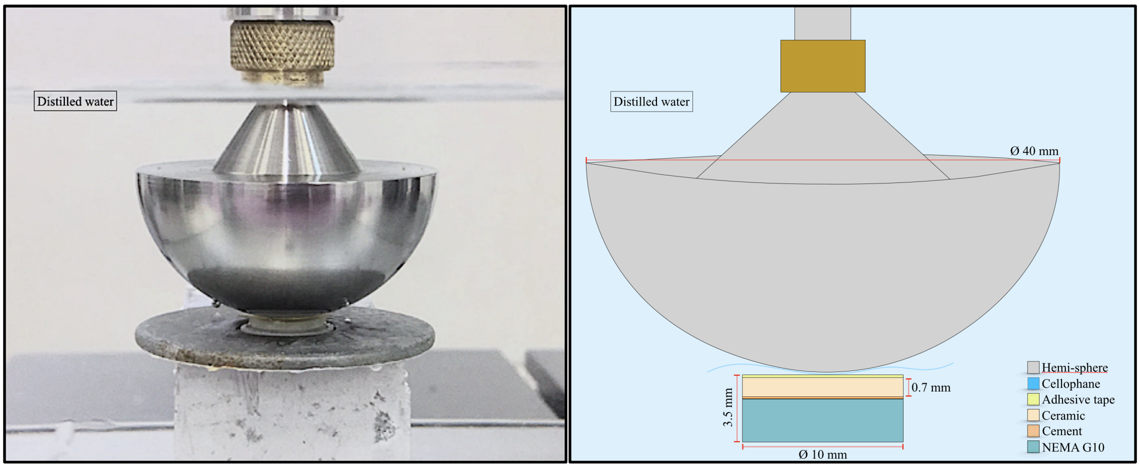


Figure 1 - Fatigue test assembly – hemispheric stainless-steel piston (Ø 40 mm), submerged in distilled water, used to apply the load in the center of the specimens' occlusal surface.

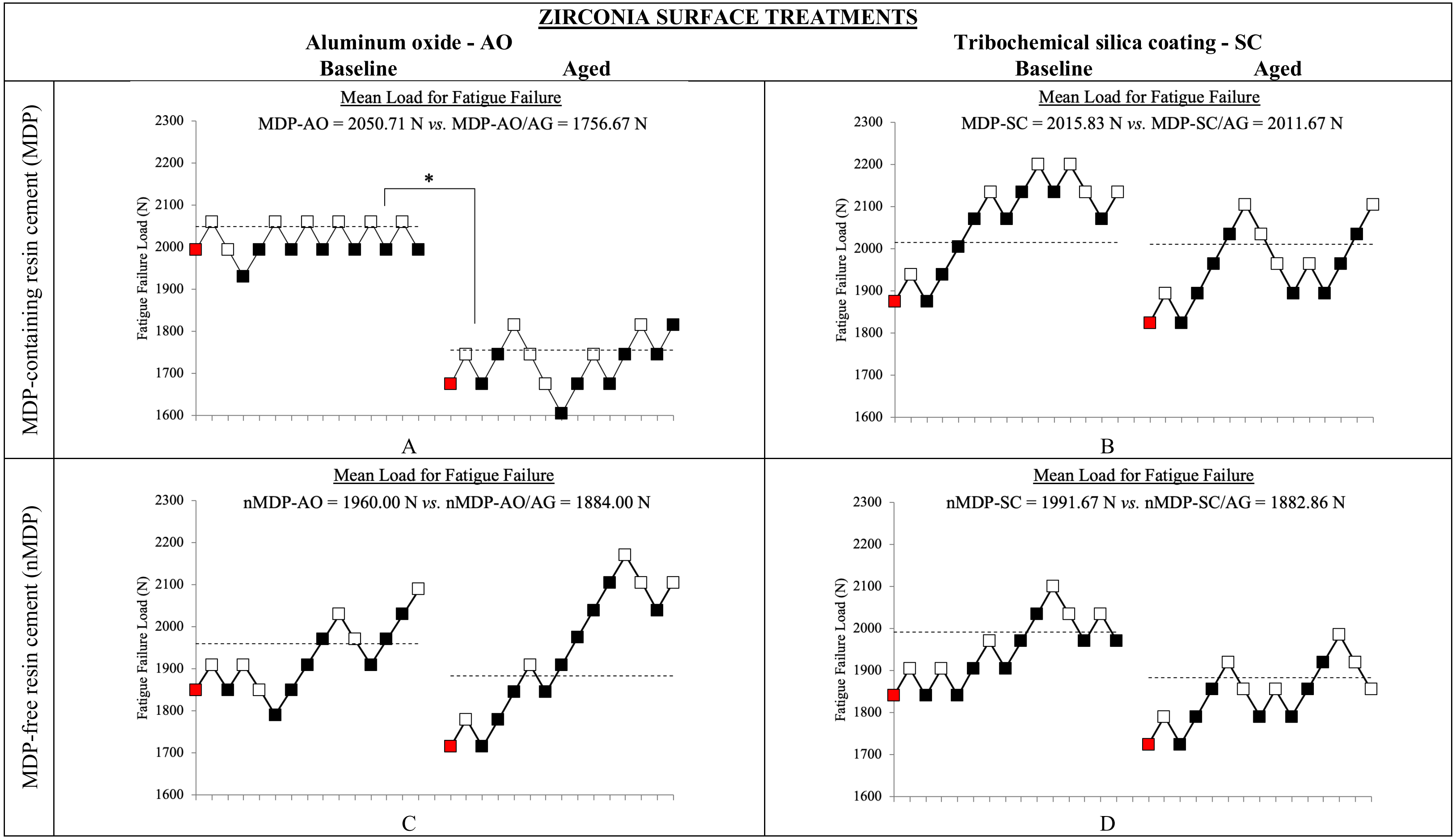


Figure 2 – Staircase survival and failure patterns for each group; n=20 or a minimum of 15 specimens tested after up-and-down character have been started (red marks). Horizontal dashed lines indicate the mean load for fatigue failure; black marks mean survived specimens; white marks mean failed ones; the asterisk (*) indicates a statistically significant difference between the groups based on Table 2.

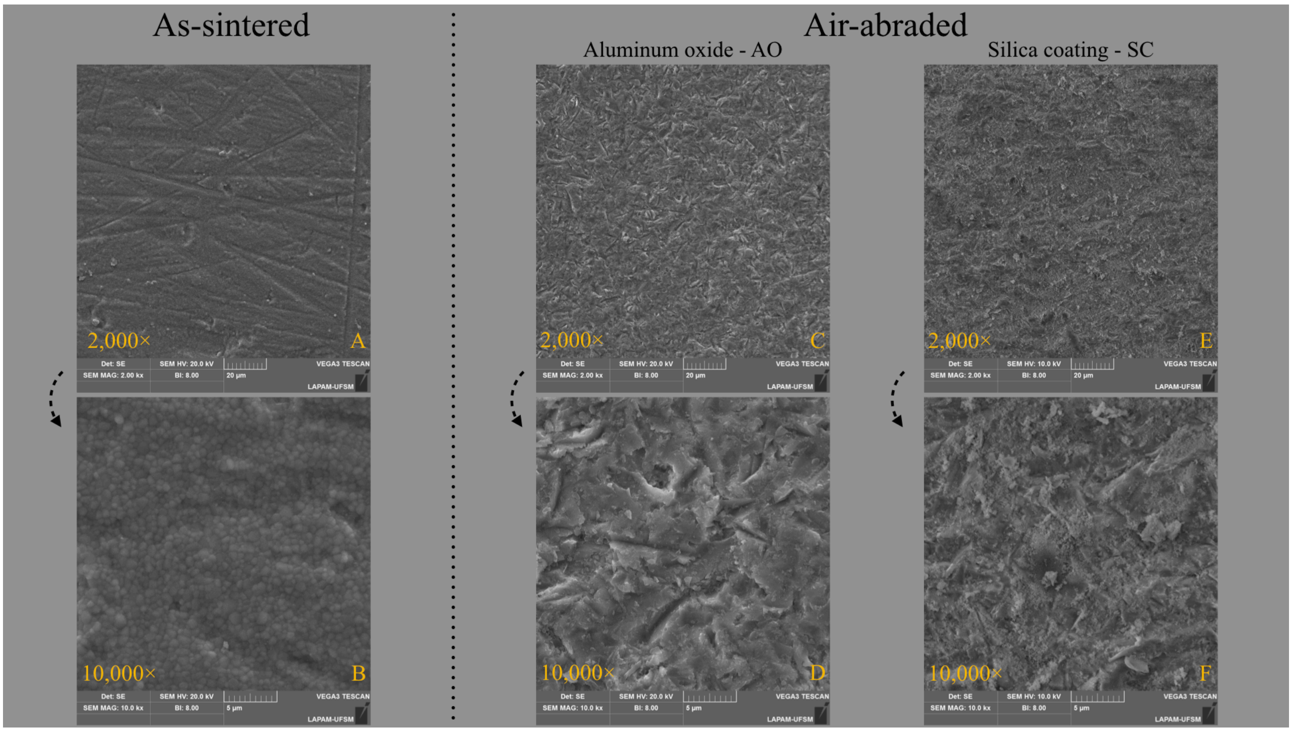
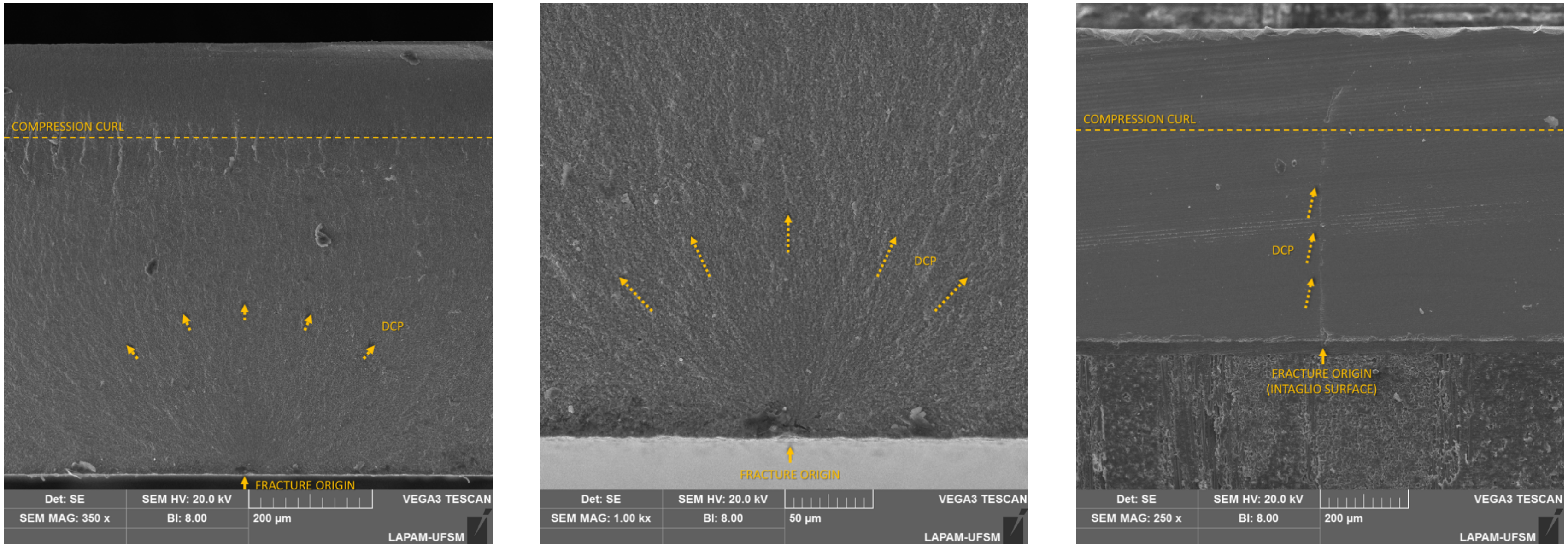


Figure 3 – Representative SEM micrographs of the zirconia ceramic surface before and after surface treatments - A and B: as-sintered (no treatment); C and D: air-abraded with Aluminum Oxide; and E and F: air-abraded with Silica-coated aluminum trioxide particles.



A **B** **C**

Figure 4 - SEM images showing the typical fractographic characteristics after fatigue failure under two perspectives: Figures A and B (350× and 1,000× magnification) show a specimen which ceramic fragments were detached after failure; Figure C (250× magnification) shows a transverse cut of a sectioned specimen that remained cemented after failure, and shows the fracture pattern presented in all failed specimens, i.e. radial crack starting at the intaglio ceramic surface and in the region bellow the application of the load with the piston. The filled arrows point to the site of fracture origin in the intaglio ceramic surface; dashed arrows indicate the direction of crack propagation (DCP); and dashed lines indicate the compression curl [Scherrer et al. 2017].

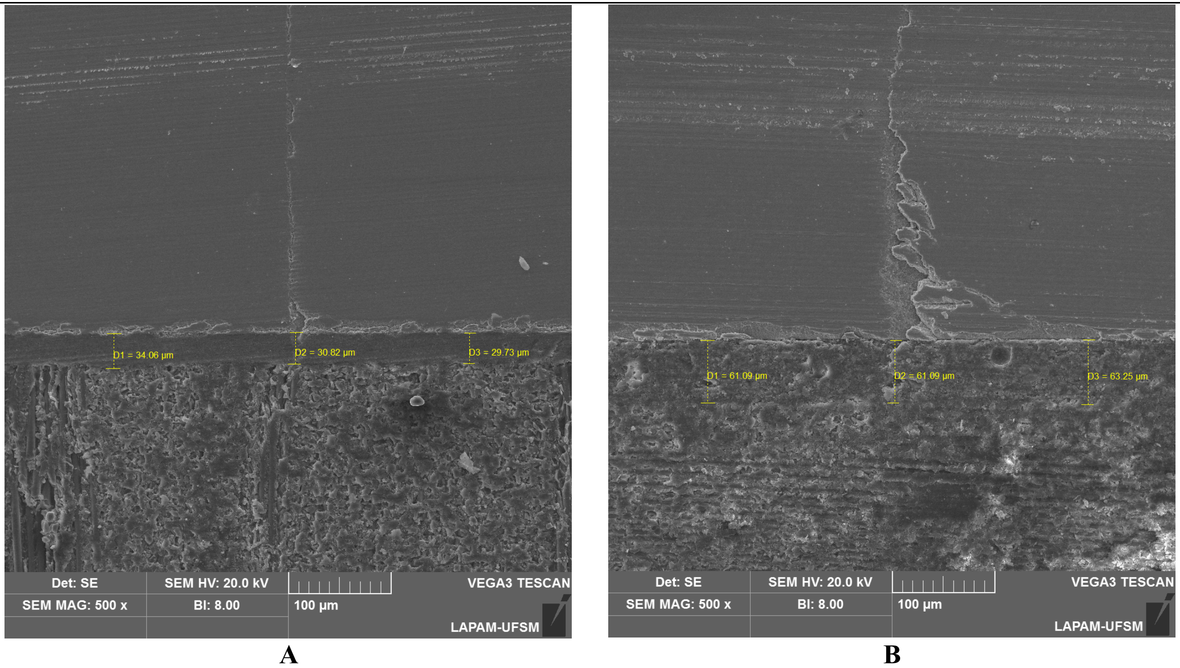


Figure 5 - SEM image (500× magnification) of cross-sectioned representative specimens showing the cement measuring obtained during fractographic analysis; A) Multilink Automix; B) Panavia F2.0.

6. DISCUSSÃO GERAL

O advento da tecnologia CAD/CAM tem gerado um avanço enorme nas restaurações totalmente cerâmicas possibilitando tratamentos muito mais conservadores e de execução mais rápida. A odontologia protética está testemunhando uma tendência para restaurações cerâmicas monolíticas. Apesar das cerâmicas odontológicas densamente sinterizadas proporcionarem restaurações mais confiáveis, do ponto de vista estrutural, elas ainda possuem capacidade relativamente pequena de absorver energia antes da fratura, ou seja, elas são friáveis (DENRY; HOLLOWAY, 2010), mesmo aquelas que apresentam elevada resistência mecânica (ex. zircônia tetragonal policristalina). Isto tem causado certa preocupação dos pesquisadores e clínicos em função do principal tipo de falha das restaurações cerâmicas ainda ser a fratura (REISS, 2006; OTTO; SCHNEIDER, 2008; GUESS et al., 2013; RAUCH et al., 2018; SAILER et al., 2015; PJETURSSON et al., 2015, 2017; MORIMOTO et al., 2016).

Neste sentido, um aspecto importante a ser considerado é que a fresagem CAD/CAM invariavelmente cria uma superfície mais rugosa (CURRAN et al., 2017), sendo que a superfície interna permanece classicamente intocada (como fresada), enquanto a superfície externa de uma restauração pode ser acabada, polida e/ou glazeada (DENRY, 2013; FRAGA et al., 2015). A esse respeito e considerando que restaurações totalmente cerâmicas podem falhar devido a trincas e falhas em sua superfície interna/de cimentação (KELLY et al., 1990; THOMPSON et al., 1994; THOMPSON; REKOW, 2004), a literatura têm apontado este efeito da usinagem com certa preocupação e cautela, uma vez que superfícies rugosas podem prejudicar as propriedades mecânicas da cerâmica por acúmulo de danos e redução da vida útil quando sob serviço clínico (carga cíclica intermitente) (KELLY et al., 2017; DE KOK et al. 2017; VENTURINI et al., 2018).

Apesar de apresentarem um efeito negativo, as superfícies rugosas desempenham um papel bastante relevante na melhoria da adesão micromecânica (SPAZZIN et al., 2016; DE KOK et al., 2017; MONTEIRO et al., 2018b). O efeito geral dessa rugosidade, a qual é inevitável na usinagem em CAD/CAM, é uma diminuição nas propriedades mecânicas das cerâmicas odontológicas, especialmente se esses defeitos não forem adequadamente "selados" durante a cimentação (SPAZZIN et al., 2016; DE KOK et al., 2017; MONTEIRO et al., 2018b). Assim, a importância de uma forte e estável adesão entre materiais cerâmicos e resinosos na vedação dos defeitos e na consequente melhoria do comportamento mecânico do conjunto deve ser destacado. Desse ponto de vista, estudos que investiguem o efeito de superfícies cerâmicas polidas ou rugosas no comportamento mecânico de cerâmicas cimentadas ao substrato devem ser conduzidos para melhor compreensão deste assunto.

As análises de sobrevivência em fadiga e de resistência ao impacto são duas análises utilizadas em odontologia para determinar o comportamento mecânico de materiais cerâmicos. Os resultados do primeiro estudo da tese mostraram que tanto o impacto quanto os resultados da fadiga foram influenciados pelo material cerâmico e pelo padrão de superfície, e, portanto, a hipótese do estudo foi aceita. A simulação da usinagem em CAD/CAM das superfícies cerâmicas reduziu significativamente o desempenho em fadiga de todos os materiais cerâmicos avaliados e também a resistência ao impacto da maioria deles.

Posteriormente, os artigos foram executados considerando-se diferentes cerâmicas (vítrea e policristalina) preparadas com diferentes tratamentos de superfície, cimentadas a um material análogo de dentina, utilizando diferentes sistemas de cimentação e submetidas ao envelhecimento, na tentativa de elucidar a importância do preenchimento de defeitos durante a cimentação adesiva na resistência final da restauração cerâmica. Muitos fatores podem contribuir para o desenvolvimento de fraturas cerâmicas (substrato, espessura da cerâmica, tipo e espessura do cimento, protocolos de cimentação, tipo de ensaio mecânico, tratamento da cerâmica, etc.) e o desenho do estudo deve resultar em falhas similares às encontradas clinicamente (KELLY, 1999; KELLY et al., 2010).

O desempenho clínico de uma restauração cerâmica é baseado em sua qualidade adesiva e em suas propriedades mecânicas. Assim, o segundo artigo da tese teve como objetivo avaliar a influência do condicionamento com ácido fluorídrico e do envelhecimento em termociclagem na resistência à fadiga de uma cerâmica feldspática cimentada com dois cimentos resinosos, um autoadesivo e outro convencional, em um material análogo de dentina (resina epóxica reforçada por fibra). Os resultados do estudo mostraram que o condicionamento com ácido fluorídrico associado à aplicação do silano aumentou a energia livre de superfície da cerâmica e sua molhabilidade pelo cimento, porém sem proporcionar melhora nos resultados de resistência à fadiga quando comparada à aplicação do agente silano apenas. Ademais, a aplicação do ácido fluorídrico seguida do envelhecimento reduziu significativamente a resistência à fadiga do material, independentemente do cimento resinoso utilizado. Outros autores obtiveram resultados semelhantes, corroborando que o condicionamento com HF melhora a força de adesão ao criar embricamentos micromecânicos, mas pode levar a um efeito enfraquecedor da cerâmica, comprometendo o desempenho clínico da restauração (ADDISON; FLEMING, 2004; VENTURINI et al., 2015). Além disso, estudos recentes corroboram esses achados, mostrando resultados piores ou iguais de resistência adesiva (PEUMANS et al., 2016), resistência à flexão biaxial (XIAOPING; DONGFENG; SILIKAS, 2014) e carga em fadiga (VENTURINI et al., 2018) ao condicionar vitrocerâmica com ácido fluorídrico previamente à cimentação, comprovando que esse assunto ainda requer avaliações e considerações. Além disso, o ácido fluorídrico tem toxicidade potencialmente perigosa conhecida de outras aplicações e também deve ser considerado quando aplicado em odontologia (ÖZCAN; ALLAHBEICKARAGHI; DÜNDAR, 2012).

Entre as cerâmicas dentárias, a zircônia é considerada a mais forte em relação à resistência, mas ainda deixa a desejar em termos de adesão (THAMMAJARUK et al., 2018). A força de união a este material depende da sua energia livre de superfície e de sua molhabilidade. O jateamento é capaz de limpar, ativar e aumentar a superfície da zircônia disponível para união (STRASSER et al., 2018; MOON et al., 2016) com os monômeros de fosfato bifuncional 10-metacriloiloxi-decildi-hidrogenofosfato (MDP), os quais se ligam quimicamente aos óxidos da superfície da zircônia e ao cimento resinoso (KERN, 2009). Nesse sentido, essa associação de tratamentos mecânicos e químicos parece propiciar melhores resultados a longo prazo (KERN, 2015; ÖZCAN; BERNASCHONI, 2015). Portanto, os dois últimos estudos da tese avaliaram o efeito de diferentes protocolos de cimentação e sistemas cimentantes na resistência à fadiga de uma zircônia monolítica fina cimentada sobre um material análogo de dentina. O primeiro deles utilizou o mesmo tipo de tratamento na superfície de cimentação da zircônia (jateamento com óxido de alumínio), variando o tipo de cimento utilizado na cimentação. Foram utilizados quatro cimentos diferentes: ionomérico, resinoso autoadesivo, resinoso com aplicação prévia de adesivo contendo MDP, e resinoso contendo MDP. Além disso, metade dos espécimes foram submetidos ao envelhecimento em termociclagem. A aplicação do cimento resinoso associado à aplicação prévia do adesivo contendo MDP proporcionou maior carga de falha em fadiga após o envelhecimento do conjunto, apresentando maior estabilidade. Apesar disso a zircônia apresentou capacidade de suportar elevadas cargas independentemente do protocolo de cimentação utilizado. O fato de que há maior concentração de tensão na superfície de cimentação das coroas de zircônia monolítica quando cimentadas com sistema convencional não-adesivo (CAMPOS et al., 2017), pode ser a explicação pela qual o cimento ionomérico teve um pior desempenho em fadiga. Outros estudos corroboram estes resultados e mostram melhores resultados em fadiga tanto para coroas de zircônia recoberta por cerâmica (ANAMI et al., 2016) quanto para coroas monolíticas de zircônia quando cimentadas adesivamente (CAMPOS et al., 2017).

Para o último trabalho, optou-se por inserir, além de diferentes cimentos e do envelhecimento, o tipo de tratamento da superfície da zircônia como fator adicional. Sendo assim, este estudo avaliou o efeito de diferentes sistemas adesivos (cimento resinoso contendo MDP ou cimento resinoso sem MDP) aplicados sobre a zircônia jateada com óxido de alumínio ou com óxido de alumínio revestido por sílica (tratamento triboquímico) na resistência à fadiga da cerâmica, com e sem envelhecimento (termociclagem + armazenamento em água por 60 dias). Os resultados do estudo mostraram que o uso do cimento resinoso contendo MDP associado ao jateamento da zircônia com óxido de alumínio não foi eficaz em manter o desempenho em fadiga após o envelhecimento do conjunto. Já o tratamento triboquímico proporcionou maior estabilidade frente ao envelhecimento. Ao usar um cimento resinoso sem MDP, o tratamento de superfície e o envelhecimento não afetaram o desempenho do material em fadiga.

Segundo Lu e colaboradores (2013), o envelhecimento da interface adesiva em água promove a redução do módulo elástico dos cimentos, bem como uma aparente redução na resistência de união da restauração. Essa degradação das propriedades do cimento pode ser suficiente para causar uma redistribuição das tensões no conjunto cimentado, reduzindo assim a capacidade das restaurações totalmente cerâmicas de tolerarem as cargas mastigatórias ao longo dos anos. Adicionalmente, no ambiente oral, as restaurações estão sujeitas à falha por fadiga, principalmente causadas pela presença de forças mastigatórias repetitivas, e associadas ao ambiente bucal úmido (ZHANG; SAILER; LAWN, 2013). A falha por fadiga pode ser definida como a fratura do material cerâmico pela introdução progressiva de trincas (crescimento lento de trincas e defeitos pré-existentes) sob tensões cíclicas de intensidade menor que a resistência normal do material (WISKOTT; NICHOLLS; BELSER, 1995).

Assim, no presente estudo não só foi avaliado o efeito do envelhecimento, como também os testes de fadiga foram realizados em ambiente úmido, onde foram aplicadas cargas constantes de intensidade variada até a ocorrência da falha dos espécimes. Esse método mimetiza o ambiente bucal e simula melhor as tensões mastigatórias se comparado ao teste estático (ATTIA et al., 2016). No entanto, a configuração do teste aplicado (carga axial) pode não simular totalmente todas as forças às quais o material é submetido no ambiente oral, especialmente para cargas laterais (movimento deslizante) que geram tensões de compressão, tração e cisalhamento na superfície da cerâmica, levando à formação e propagação de trincas na subsuperfície do material (REN; ZHANG, 2014).

Outra limitação importante é de que o conjunto restaurador foi construído de forma simplificada (espécimes em forma de disco), o qual não é capaz de simular completamente a anatomia de uma coroa de um dente molar. Talvez por isso, em nosso estudo, as cargas necessárias para causar as trincas radiais iniciais (cerâmica feldspática – Mark II, média de carga = 534,83 N) foram bem mais altas que as cargas mastigatórias normais, as quais possuem magnitude aproximada de 148 a 354 N (TAKAKI; VIEIRA; BOMMARITO, 2014). Além disso, a zircônia policristalina, mesmo em uma espessura relativamente fina (0,7 mm) e independente do tratamento de superfície ou cimento utilizado na sua cimentação, proporcionou resistências superiores às forças máximas de mordida alcançadas durante o bruxismo do sono (± 800 N) e às forças voluntárias máximas de mordida durante o dia (± 1.000 N) (NISHIGAWA; BANDO; NAKANO, 2001).

Considerando os assuntos discutidos acima, enfatizamos que nossos resultados devem ser cuidadosamente analisados, e mais estudos *in vitro* e clínicos devem ser executados para que os nossos resultados sejam corroborados.

7. CONSIDERAÇÕES FINAIS

- A simulação da usinagem em CAD/CAM aumentou significativamente a rugosidade das cerâmicas feldspática, híbrida, de silicato de lítio reforçada por zircônia, dissilicato de lítio e zircônia e consequentemente levou a um pior desempenho mecânico em fadiga e ao impacto.
- O condicionamento com ácido fluorídrico, associado à aplicação do silano aumentou a energia livre da superfície da cerâmica feldspática e sua molhabilidade pelo cimento, porém sem proporcionar melhora nos resultados de resistência à fadiga quando comparada à aplicação do agente silano apenas. A aplicação do ácido fluorídrico seguida do envelhecimento reduziu significativamente a resistência à fadiga do material, independentemente do cimento resinoso utilizado, auto-adesivo ou convencional.
- A aplicação do cimento resinoso associado à aplicação prévia do adesivo contendo MDP proporcionou maior carga de falha em fadiga após o envelhecimento do conjunto zircônia/análogo de dentina, apresentando maior estabilidade, do que os cimentos de ionômero de vidro reforçado por resina, auto-adesivo e cimento contendo MDP. Apesar disso a zircônia apresentou capacidade de suportar elevadas cargas independentemente do protocolo de cimentação utilizado.
- O uso do cimento resinoso contendo MDP associado ao jateamento da zircônia com óxido de alumínio não foi eficaz em manter o desempenho em fadiga após o envelhecimento do conjunto cerâmica/análogo de dentina. Já o tratamento triboquímico proporcionou maior estabilidade frente ao envelhecimento. Ao usar um cimento resinoso sem MDP, o tratamento de superfície (jateamento com óxido de alumínio e tratamento triboquímico) e o envelhecimento não afetaram o desempenho do material em fadiga.

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ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *JOURNAL OF PROSTHODONTIC RESEARCH*

GUIDE FOR AUTHORS

INTRODUCTION

Journal of Prosthodontic Research is published 4 times annually, in January, April, July, and October, under supervision by the Editorial Board of Japan Prosthodontic Society, which selects all materials submitted for publication.

Journal of Prosthodontic Research originated as an official journal of Japan Prosthodontic Society. It has recently developed a long-range plan to become the most prestigious Asian journal of dental research regarding all aspects of oral and occlusal rehabilitation, fixed/removable prosthodontics, oral implantology and applied oral biology and physiology. The Journal will cover all diagnostic and clinical management aspects necessary to reestablish subjective and objective harmonious oral aesthetics and function.

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Prosthodontic treatment may become necessary as a result of developmental or acquired disturbances in the orofacial region, of orofacial trauma, or of a variety of dental and oral diseases and orofacial pain conditions. The scientific content of the Journal therefore strives to reflect the best of evidence-based clinical dentistry. Modern clinical management should be based on solid scientific evidence gathered about diagnostic procedures and the properties and efficacy of the chosen intervention. The content of the Journal also includes documentation of the possible side-effects of rehabilitation, as well as prognostic perspectives of the treatment modalities chosen. The Journal focuses on presenting original research findings and original technical appraisals, generating critical reviews and relevant case stories, and stimulating commentaries and professional debates in the Letters to the Editor column. The work shall not be subsequently published in any other publication in any language without prior written consent of the publisher.

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

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

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

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

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

Case report

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

<Structure of case report>

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

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

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

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

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

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

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

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

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

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

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Journal article: multiple authors - Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin Critical Review of Oral and Biological Medicine 8(3) 306-335.

Journal article: special issue/supplement - Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry Operative Dentistry (Supplement 6) 119-144.

Abstract: Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003) Comparative study on adhesive performance of functional monomers Journal of Dental Research 82(Special Issue B) Abstract #0051 p B-19.

Corporate publication: ISO-Standards (1997) ISO 4287 Geometrical Product Specifications Surface texture: Profile method – Terms, definitions and surface texture parameters Geneve: International Organization for Standardization 1st edition 1-25.

Book: single author - Mount GJ (1990) An Atlas of Glass-ionomer Cements Martin Duntz Ltd, London.

Book: two authors - Nakabayashi N & Pashley DH (1998) Hybridization of Dental Hard Tissues Quintessence Publishing, Tokyo.

Book: chapter - Hilton TJ (1996) Direct posterior composite restorations In: Schwarts RS, Summitt JB, Robbins JW (eds) Fundamentals of Operative Dentistry Quintessence, Chicago 207-228.

Website: single author - Carlson L (2003) Web site evolution; Retrieved online July 23, 2003 from: http://www.d.umn.edu/~lcarlson/cms/evolution.html

Website: corporate publication - National Association of Social Workers (2000) NASW Practice research survey 2000. NASW Practice Research Network, 1. 3. Retrieved online September 8, 2003 from: http://www.socialworkers.org/naswprn/default

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