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Michele Mirian May

EFEITO DO PROCESSAMENTO E DO USO DE CIMENTOS RESINOSOS DE DIFERENTES VISCOSIDADES NO COMPORTAMENTO MECÂNICO DE VITROCERÂMICAS

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 a obtenção do título de **Doutora em Ciências Odontológicas.**

Orientadora: Prof.ª. Dr.ª Liliana Gressler May
Coorientador: Prof. Dr. Luiz Felipe Valandro Soares

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Aprovada em 22 de julho de 2021:

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Gabriel Kalil Rocha Pereira, Dr. (UFSM)

Santa Maria, RS
2021

DEDICATÓRIA

Aos meus filhos Eduardo e Clahra,
motivação do meu caminhar, razão da minha existência,
e ao meu marido Fabiano, companheiro de uma vida.
À minha avó emprestada Ela Alice (in memoriam),
presente de amor e afeto.
Permanecerás comigo, aonde quer que eu vá.
A eles, com todo o meu amor e minha devoção.

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(Isaac Newton)

RESUMO

EFEITO DO PROCESSAMENTO E DO USO DE CIMENTOS RESINOSOS DE DIFERENTES VISCOSIDADES NO COMPORTAMENTO MECÂNICO DE VITROCERÂMICAS

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A presente tese é constituída por três estudos que abordam como tema central o efeito do protocolo adesivo com cimentos resinosos em diferentes viscosidades no comportamento mecânico da cerâmica de dissilicato de lítio usinada. O primeiro estudo objetivou acessar a literatura científica disponível acerca do efeito que os procedimentos que antecedem a cimentação desenvolvem na resistência de restaurações vitrocerâmicas usináveis em sistema *computer-aided design; computer-aided manufacturing* (CAD-CAM). Foi conduzida uma revisão sistemática de estudos *in vitro*, acessando as bases de dados PubMed/MEDLINE, Web of Science, and Scopus. Dos 2764 potenciais artigos encontrados na busca, 42 foram selecionados para leitura completa e 12 artigos foram incluídos na metanálise. Concluiu-se que a resistência flexural de vitrocerâmicas para sistema é reduzida por procedimentos abrasivos, como desgaste com brocas diamantadas e usinagem. Além disso, a microestrutura da cerâmica, a concentração do ácido fluorídrico e o tempo de condicionamento determinam sua influência na resistência flexural e na rugosidade das vitrocerâmicas. O segundo estudo analisou se a aplicação de uma camada de cimento resinoso de alta e baixa viscosidades influenciaria a resistência à fadiga de dissilicato de lítio usinado. Para tanto, foram confeccionados discos de cerâmica obtidos por usinagem em sistema CEREC inLab, bem como a partir de blocos reduzidos em cilindros, seccionados em máquina de corte e polidos. Seis grupos experimentais foram avaliados, de acordo com a característica superficial (usinado ou polido), protocolo de cimentação (com cobertura ou sem cobertura) e o cimento utilizado (alta ou baixa viscosidade). Os corpos de prova foram submetidos a ensaio de resistência à fadiga flexural (n=15) pelo método "*step-stress*" (10.000 ciclos por passo, frequência de 20 Hz, tensão inicial 60 MPa e incrementos de 20 MPa), em configuração de ensaio *piston-on-three ball*, além de análises de rugosidade superficial, ângulo de contato, e análise topográfica e fractográfica em microscopia eletrônica de varredura. Concluiu-se que a cobertura com cimento exerceu um efeito capaz de reverter os danos causados pela usinagem no comportamento à fadiga do dissilicato de lítio. Além disso, o comportamento dos cimentos de alta e baixa viscosidade foi similar com relação ao reforço da cerâmica. O terceiro estudo avaliou a carga para falha em fadiga de discos de dissilicato de lítio usinados e polidos cimentados em material análogo à dentina. O estudo contou com quatro grupos experimentais, conforme a característica superficial (usinados ou polidos) e o cimento utilizado (alta ou baixa viscosidade). A carga e o número de ciclos para a falha foram determinados através do método "*step-stress*" (n = 15) (10.000 ciclos por passo a uma frequência de 20 Hz, carga inicial de 500 N e incrementos de 100 N por passo). Similarmente ao segundo estudo, foram conduzidas análises de rugosidade superficial antes da cimentação, topografia de superfície e análise fractográfica após os testes de fadiga. Os resultados mostraram que quando a cerâmica de dissilicato de lítio foi aderida a um substrato análogo à dentina, os efeitos deletérios da usinagem não foram revertidos pela ação do cimento resinoso. Não foram encontradas diferenças no desempenho das duas viscosidades de cimento investigadas.

Palavras-chave: Cimentação. e-max CAD. Fadiga Cíclica. Resistência. Usinagem.

ABSTRACT

PROCESSING AND COATING WITH DIFFERENT RESIN CEMENT VISCOSITIES: EFFECTS ON THE MECHANICAL BEHAVIOR OF GLASS-CERAMICS

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ADVISOR: Liliana Gressler May
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This thesis comprises three studies that address as a central theme the effect of adhesive protocol with resin cements at different viscosities on the mechanical behavior of lithium disilicate machined ceramics. The first study aimed to access the available scientific literature on the effect that procedures performed before cementation develop on the strength of computer-aided design; computer-aided manufacturing (CAD-CAM) glass-ceramics restorations. A systematic review was conducted for in vitro studies, accessing the PubMed/MEDLINE, Web of Science, and Scopus databases. Of the 2764 potential articles found in the search, 42 were selected for full reading and 12 articles were included in the meta-analysis. It was concluded that the flexural strength of CAD-CAM glass-ceramics is reduced by grinding procedures, such as machining and fitting adjustment. Furthermore, ceramic microstructure, hydrofluoric acid concentration, and etching time determined the influence of hydrofluoric acid etching on the flexural strength and surface roughness of glass-ceramic materials. The second study analyzed whether high and low viscosity resin cement coating influenced the fatigue strength of machined vs polished lithium disilicate. Ceramic discs were obtained by machining in a CEREC inLab system and from reducing CAD-CAM blocks into cylinders, slicing in a cutting machine and polishing. Six experimental groups were evaluated, according to the surface characteristic (machined or polished), cementation protocol (coated or uncoated) and the cement used (high or low viscosity). The specimens were submitted to flexural fatigue strength test (n=15) by the "step-stress" method (10,000 cycles per step, frequency of 20 Hz, initial tension 60 MPa and increments of 20 MPa), in a piston-on-three ball test configuration, as well as roughness and contact angle analysis and scanning electron microscopy topographic and fractographic analysis. It was concluded that the cement coating was able to revert the damage effect caused by machining on the fatigue behavior of lithium disilicate. Furthermore, the behavior of high and low viscosity cements was similar regarding to ceramic strengthening. The third study evaluated the fatigue failure load of machined and polished lithium disilicate discs cemented to a dentin analogue material. The study had four experimental groups, according to the surface characteristic (machined or polished) and the cement used (high or low viscosity). The fatigue failure load and number of cycles for fatigue failure were determined using the “step-stress” method (n = 15) (10,000 cycles per step at a frequency of 20 Hz, initial load of 500 N and increments per step of 100N). Similarly to the second study, surface roughness analyzes were conducted before cementation, surface topography and fractographic analysis were done after fatigue test. The results showed that when lithium disilicate ceramic is cemented to a dentin analog support substrate, the deleterious effects of machining were not reverted by the resin cement bonding. No differences were found in the fatigue performance for the two investigated cement viscosities.

Keywords: Cementation. e-max CAD. Cyclic Fatigue. Strength. Milling.

SUMÁRIO

1 INTRODUÇÃO

As restaurações totalmente cerâmicas são consideradas opções vantajosas de tratamento para a odontologia restauradora devido à sua aparência natural, propriedades biomecânicas e biocompatibilidade (KELLY, 2016; ROJPAIBOOL; LEEVAILOJ, 2017). O advento da tecnologia *Computer Assisted Design, Computer Assisted Manufacturing* (CAD-CAM) reduziu o tempo de produção e proporcionou maior confiabilidade para essas restaurações, já que os blocos cerâmicos fabricados industrialmente são mais homogêneos e têm menor probabilidade de incorporar defeitos internos (KELLY, 2016).

Uma ampla variedade de materiais cerâmicos está disponível para CAD-CAM. Eles diferem em microestrutura, comportamento mecânico e métodos de usinagem. Cerâmicas estão disponíveis para usinagem "dura", de blocos totalmente sinterizados (por exemplo, cerâmicas à base de feldspato, leucita e dissilicato de lítio) e para usinagem "macia", de blocos parcialmente sinterizados (por exemplo, zircônia tetragonal policristalina estabilizada com ítria - Y-TZP) (DENRY; KELLY, 2008; KELLY, 2016).

As cerâmicas de usinagem dura abrangem predominantemente os materiais que contêm quantidades significativas de fases vítreas e, portanto, possuem tenacidade à fratura reduzida em comparação às cerâmicas policristalinas (BORBA et al., 2011; FRAGA et al., 2017). A cerâmica à base de dissilicato de lítio é uma vitrocerâmica que apresenta alto teor cristalino (70% vol) (SANTOS et al., 2015). Os cristais de dissilicato de lítio altamente intercruzados promovem a deflexão das trincas, o que melhora sua resistência à fratura em comparação com outras cerâmicas vítreas (APEL et al., 2008; FRAGA et al., 2017). Sua resistência flexural é de cerca de 360 MPa (SONMEZ et al., 2018) e sua tenacidade à fratura 1,67 ~ 2,5 MPa $m^{-0,5}$ (SONMEZ et al., 2018; HAMPE et al., 2019). Portanto, este material é indicado para laminados, inlays, onlay, coroas anteriores, posteriores e sobre implantes (SANTOS et al., 2015; ISO 6872: 2008).

Estudos clínicos envolvendo a cerâmica de dissilicato de lítio usinada reportam alta taxa de sobrevivência (88,52 a 100%) e de sucesso (73,5 a 100%) em curto e médio prazos (até 10 anos), mas a literatura ainda carece de estudos de longo prazo (AZIZ; EL-MOWAFY; PAREDES, 2020). Apesar dos bons resultados, fraturas envolvendo estas restaurações ainda são uma realidade (REICH; SCHIERZ, 2013; RAUCH et al., 2018).

Cerâmicas são materiais de natureza frágil, nos quais a resistência à fratura é substancialmente afetada pela presença de defeitos, notadamente críticos quando colocados em zonas de concentração de tensão de tração, como a superfície interna de coroas cerâmicas

(KELLY, 1999; ADDISON; MARQUIS; FLEMING, 2008; HOOI et al. 2013; FRAGA et al., 2015, FRAGA et al., 2017). O processo padronizado de fabricação reduz os defeitos intrínsecos dos blocos CAD-CAM, no entanto a usinagem dos mesmos, introduz novas características nas restaurações cerâmicas, incluindo trincas radiais e laterais, lascamentos, defeitos na sub-superfície e tensões residuais (MARSHALL et al., 1983; REKOW E THOMPSON, 2005).

Uma redução significativa nos valores de resistência flexural de cerâmicas de diferentes microestruturas usinadas por CAD-CAM foi observada por Fraga et al. (2017) quando comparadas com suas equivalentes totalmente polidas. A maior redução na resistência à fadiga devido à usinagem foi observada na Y-TZP (40%), seguida por dissilicato de lítio (33%) e leucita (29%), sugerindo que a usinagem de corte "macio" pode ser tão prejudicial à resistência à fadiga do material cerâmico quanto a usinagem de corte "duro". Além disso, os defeitos introduzidos pela usinagem em três apresentações comerciais de dissilicato de lítio diminuíram significativamente a resistência desses materiais, não sendo eliminados por tratamentos térmicos para cristalização completa ou *annealing* (ROMANYK et al., 2019). Diante desses achados, faz-se necessário acessar a literatura disponível para investigar a influência que a usinagem e outros fatores que antecedem a cimentação desempenham na resistência das vitrocerâmicas para CAD-CAM e nas suas características superficiais.

É importante considerar que, no ambiente bucal, as restaurações em cerâmica pura estão sujeitas a muitos desafios, como cargas cíclicas, umidade, variações de temperatura e pH. Consequentemente, elas tendem a falhar devido à fadiga (WISKOTT; NICHOLLS; BELSER, 1995). As cerâmicas são suscetíveis a um crescimento de trincas lento e estável (SCG) quando submetidas a tensões abaixo do valor crítico, especialmente na presença de água. Este fenômeno pode eventualmente levar à diminuição da resistência ao longo do tempo, diminuindo a vida útil das próteses dentárias e parece estar bastante relacionado à microestrutura da cerâmica (GONZAGA et al., 2011). Daí a importância da realização de estudos in vitro em configurações que reproduzam essas condições no laboratório, quando se pretende avaliar a resistência desses materiais frente a diferentes tratamentos.

A cimentação de restaurações de cerâmica pura com agentes resinosos tem sido relacionada ao seu melhor desempenho mecânico tanto em estudos laboratoriais (MARQUIS, 1992, PAGNIANO et al., 2005, FLEMING et al., 2006, FLEMING et al., 2017, BARBON et al., 2018) quanto em estudos clínicos (VAN DIJKEN et al., 1998; MALAMENT; SOCRANSKY, 2001). Teorias para esta aparente melhoria de desempenho tem sido descritas, como a cicatrização completa ou parcial de trincas encontradas na população superficial de defeitos da cerâmica através da infiltração de resina (MARQUIS, 1992), tensões compressivas residuais geradas na interface cerâmica/cimento devido à contração de polimerização (NATHANSON, 1993; ROSENSTIEL et al., 1993; FLEMING et al., 2017), efeitos de constrição de Poisson (WANG et al., 1995; ADDISON; MARQUIS; FLEMING, 2007), e a dependência do aumento da resistência a partir de uma camada híbrida interpenetrada de cerâmica-cimento sensível às variáveis de cimentação e técnicas de assentamento clínico da peça (FLEMING et al., 2006; ADDISON; MARQUIS; FLEMING, 2008). Ressalta-se que, segundo o conhecimento do autor, não há estudo que avalie o efeito isolado do cimento resinoso sobre a resistência das cerâmicas usinadas em sistemas CAD-CAM.

A presente tese consiste em três trabalhos realizados para acessar o efeito que o protocolo adesivo com cimentos resinosos em diferentes viscosidades exerce no comportamento mecânico da cerâmica de dissilicato de lítio usinada. Inicialmente, foi investigada a literatura disponível acerca do impacto que os procedimentos que antecedem a cimentação desempenham na rugosidade superficial e na resistência de cerâmicas vítreas reforçadas por partículas disponíveis para sistema CAD-CAM. Considerando que defeitos são introduzidos pela usinagem na superfície de cimentação destas restaurações cerâmicas, sua suscetibilidade à fadiga e a evidência de que a cimentação adesiva melhora seu desempenho, os estudos subsequentes objetivaram avaliar o efeito da união ao agente cimentante resinoso sobre o comportamento à fadiga de cerâmicas à base de dissilicato de lítio usinadas por sistema CAD-CAM. O segundo estudo teve a finalidade de avaliar a capacidade do cimento resinoso em diferentes viscosidades de compensar (reverter) o resultado prejudicial da usinagem na resistência à fadiga flexural, comparando-se os efeitos em grupos usinados *vs* polidos. Posteriormente foi investigada a implicação da cimentação do dissilicato de lítio a um substrato análogo à dentina, utilizando agentes resinosos de alta e baixa viscosidade, na carga para fratura à fadiga. Neste estudo pretendeu-se simular restaurações com as superfícies internas usinadas e polidas, cimentadas a substrato dentinário, em uma configuração experimental que simula a condição clínica de maneira mais aproximada.

2. REVISÃO DE LITERATURA

2.1 RESTAURAÇÕES TOTALMENTE CERÂMICAS E A TECNOLOGIA CAD-CAM

Cerâmicas odontológicas podem ser consideradas biomateriais "compósitos", constituídas de duas ou mais fases. Podemos classificá-las em: predominantemente vítreas, vítreas reforçadas por partículas e policristalinas (KELLY, 2004).

As cerâmicas vítreas contêm uma rede tridimensional de átomos sem um padrão regular de distribuição no espaço (ângulo e distância), ou seja, uma fase amorfa com partículas cristalinas dispersas nesta matriz. Já as cerâmicas policristalinas apresentam um arranjo regular e densamente compactado de seus átomos, sem fase vítrea e com a presença de estabilizadores de fase (KELLY, 2016). Em modos gerais, cerâmicas altamente estéticas são predominantemente vítreas, enquanto cerâmicas altamente resistentes são cristalinas (KELLY, 2004).

Um dos primeiros sistemas utilizados para a fabricação de produtos odontológicos foi à base de cerâmica vítrea (SiO_2 – Al_2O_3 – K_2O) para confecção de jaquetas dentárias. Estes sistemas começaram a falhar muito rapidamente, devido à friabilidade do material. Como metais eram frequentemente usados para produzir inlays, coroas e pontes dentárias, a possibilidade de usar cerâmica para recobrir essas restaurações e, assim, conferir-lhes uma aparência natural foi aventada e cerâmicas feldspáticas sinterizadas foram desenvolvidas para este propósito. Com a cristalização de leucita ($KAlSi_2O_6$), um alto coeficiente de expansão térmica (CTE) pode ser alcançado na cerâmica, tornando-as compatíveis com os metais, consolidando as próteses metalocerâmicas (HÖLAND et al., 2009).

Devido às propriedades estéticas, a propensão de desenvolver restaurações livres de metais não foi abandonada após os primeiros insucessos. Na década de 70 cerâmicas feldspáticas sem metal foram reforçadas com componentes adicionais. Um progresso considerável no aumento da resistência desses materiais foi alcançado com esse processo. Além das cerâmicas do sistema convencional de SiO_2 – Al_2O_3 – K_2O, cerâmicas infiltradas [ZrO2 ou espinélio (MgAl2O4)] foram testadas para uso em odontologia restauradora. Estas cerâmicas eram utilizadas como infraestruturas porosas infiltradas com vidro, a fim de atenuar sua opacidade (HÖLAND et al., 2009). Nos anos 80, a introdução de cerâmicas de baixa contração e um sistema de vitrocerâmica injetável (Dicor, Dentsply) marcaram a introdução de cerâmicas com métodos de processamento inovadores (SANTOS et al., 2015). Todos esses desenvolvimentos contribuíram para a tendência em alcançar resultados estéticos excepcionais com restaurações livres de metal.

A possibilidade de desenvolvimento de materiais com qualidade controlada, redução dos custos de produção e padronização do processo de manufatura encorajaram os pesquisadores a automatizar o processo manual convencional de confecção de restaurações cerâmicas, através da introdução da tecnologia assistida por computador (CAD-CAM) na odontologia restauradora. A partir da década de 1980, Mormann e Brandestini desenvolveram o primeiro sistema operacional (CEREC I, Siemens Dental - atualmente Sirona Dental) (WITTNEBEN et al., 2009; SANTOS et al., 2015). Desde então os sistemas CAD-CAM, que usam um dispositivo de escaneamento, softwares de projeto e uma unidade fresadora, tem se desenvolvido e impulsionado o desenvolvimento de materiais cerâmicos que podem ser processados na forma de blocos para usinagem.

As vantagens de um sistema de fabricação automatizada são evidentes: materiais pré-fabricados e controlados industrialmente; maior qualidade, eficiência e reprodutibilidade; armazenamento eletrônico de dados de várias etapas de tratamento, cadeia de produção padronizada; o uso de materiais cerâmicos e de titânio de alta resistência, e a oportunidade da introdução de novos materiais na prática odontológica, bem como a possibilidade de fabricação dentro do próprio consultório (*chairside* CAD-CAM) (WITTNEBEN et al., 2009, KELLY, 2016) .

Dois métodos estão disponíveis para fresar os blocos cerâmicos: usinagem de corte "duro", a partir de blocos de cerâmica totalmente sinterizadas, e usinagem de corte "macio", baseadas em cerâmicas parcialmente sinterizadas que subsequentemente passarão por sinterização completa para adquirir sua configuração final e máxima resistência (GRIGGS, 2007; SANTOS et al., 2015). O desgaste do material parcialmente sinterizado difere do desgaste das cerâmicas totalmente densas. No primeiro, a fresagem do material envolve a quebra de fracas ligações formadas entre as partículas unidas durante o primeiro processo de sinterização. Assim, menores danos provenientes da usinagem são esperados (como trincas subsuperficiais, por exemplo) (DENRY; KELLY, 2008; KELLY, 2016). Entretanto, estudos recentes relataram uma diminuição nas propriedades mecânicas das cerâmicas após a usinagem, mesmo quando submetidas ao corte "macio" (FRAGA et al., 2017; ROMANYK et al., 2019).

2.2 CERÂMICA DE DISSILICATO DE LÍTIO

O desenvolvimento de cerâmicas altamente resistentes para próteses livres de metal representa uma transição entre o aumento dos percentuais em volume do material cristalino e o decréscimo no conteúdo vítreo (KELLY, 2004). Cerâmicas vítreas reforçadas por partículas cristalinas ou vidros de alta fusão proporcionaram uma melhora nas propriedades mecânicas e físicas, como resistência, tenacidade à fratura e coeficiente de expansão térmica em comparação às cerâmicas feldspáticas. Por outro lado, há um acréscimo na opacidade e no índice de refração da luz. Estas partículas de carga podem ser incorporadas mecanicamente ou precipitadas dentro da matriz vítrea através de um tratamento térmico chamado de ceramização. Leucita, óxido de alumínio, óxido de zircônio e dissilicato de lítio são exemplos de partículas cristalinas de reforço (KELLY, 2004; KELLY, 2016).

As cerâmicas vítreas reforçadas por dissilicato de lítio ($LiSi_2O_5$) demonstram um teor de cristais de aproximadamente 70% em volume, e sua microestrutura mostra um intertravamento entre os cristais. A vitrocerâmica reforçada por dissilicato de lítio foi inicialmente empregada para confecção de restaurações por prensagem ou injeção, através da técnica da cera perdida. Cerâmicas vítreas quimicamente duráveis, de alta resistência e alta dureza, com translucidez ajustável, foram produzidas neste sistema de materiais, indicado para a confecção de coroas cerâmicas monolíticas unitárias anteriores e posteriores, inlays, onlays, facetas e próteses parciais fixas anteriores de até três elementos, que podem se estender até pré-molares. Sua estrutura pode ser revestida com cerâmica vítrea de fluorapatita para aumentar a transmissão de luz, caso haja uma maior exigência estética (CONRAD; SEONG; PESUN, 2007; HÖLAND et al., 2009).

A versão utilizada para usinagem em CAD-CAM consiste em blocos contendo metassilicatos de cadeia dendrítica que apresentam uma cor azulada (*blue stage*). Nesse estágio a usinagem é mais fácil, porém a durabilidade química deste material intermediário é muito fraca, devendo ser tratado termicamente a 840ºC após a etapa de fresagem para a cristalização completa, transformando os cristais de metassilicato de lítio em dissilicato de lítio, mais resistentes (HÖLAND et al, 2009; LI, CHOW, MATINLINNA, 2014; KELLY, 2016). A resistência flexural desta vitrocerâmica é cerca de 359,2 MPa (SONMEZ et al., 2018) e sua tenacidade à fratura 1,67 ~ 2,15 MPa $m^{-0,5}$ (SONMEZ et al., 2018; HAMPE et al., 2019).

Em relação ao desempenho clínico das cerâmicas de dissilicato de lítio, Kern, Sasse e Wolfart (2012) relataram que a taxa de sobrevivência de próteses dentárias fixas de três elementos, monolíticas, de dissilicato de lítio prensado foi de 87,9%, num período de 10 anos. Gehrt et al. (2013) relatam uma taxa de sobrevivência de 98,4% em 8 anos, para coroas com infraestrutura de dissilicato de lítio prensada recobertas com cerâmica à base de fluorapatita.

Malament et al. (2019) reportaram uma taxa de sobrevivência cumulativa de 99.6% para coroas unitárias de dissilicato de lítio prensadas ao longo do período de 10 anos, sendo que a probabilidade de sobrevivência para restaurações monolíticas de dissilicato de lítio (n = 1410) foi de 96,5% em 10,4 anos e 100% em 7,9 anos para o grupo com infraestrutura de dissilicato de lítio recoberta (bicamada) (n = 550).

Com relação à cerâmica de dissilicato de lítio usinada, ainda há poucos estudos clínicos de longo acompanhamento. Uma revisão sistemática de literatura incluiu seis estudos em sua avaliação, destes, três tinham até 26 meses de acompanhamento, dois até 48 meses de acompanhamento e um com 120 meses de acompanhamento (AZIZ; EL-MOWAFY; PAREDES, 2020). Os autores encontraram taxas de sobrevivência de curto e médio prazo variando de 93,3% a 100% e as principais complicações foram de origem biológica (complicações endodônticas e recidiva de cárie). O estudo com maior acompanhamento é o de Rauch et al. (2018), que reportou um índice de sobrevivência de 83,5% em 10 anos para coroas unitárias monolíticas de dissilicato de lítio usinadas. Foram acompanhadas 41 coroas, sendo que as falhas ocorridas foram causadas principalmente por eventos biológicos (n = 4) e a única complicação técnica (fratura da coroa) ocorreu em uma coroa depois de 2,9 anos de estudo. Após avaliação em microscópio eletrônico de varredura das partes fraturadas, os autores referiram uma espessura de 871 µm na superfície oclusal, menor que 1 mm de espessura recomendada pelo fabricante.

2.3 DEFEITOS INTRODUZIDOS NA CERÂMICA PELA USINAGEM

Foram descritos cinco estágios para a usinagem da cerâmica por instrumentos diamantados de corte. Em um primeiro momento há a indução de um campo de concentração de tensões na cerâmica, gerado pelo impacto ocasionado pelo contato entre o instrumento de corte e a cerâmica. No segundo estágio há um acúmulo de energia desencadeado pela movimentação do instrumento e o início da formação de trincas nas regiões de alta concentração de tensão. O terceiro estágio engloba a propagação das trincas decorrentes do progresso da usinagem, que se completa em um quarto estágio, quando múltiplas microtrincas se fundem, removendo o material cerâmico e aliviando a energia armazenada. A formação de uma nova textura de superfície na cerâmica usinada, contendo danos superficiais e subsuperficiais encerra o quinto estágio (ZHANG; SATISH; KO, 1994). Sindel et al. (1998) estimaram uma extensão destes defeitos de até 60 µm para uma cerâmica feldspática usinada pelo sistema CEREC I®.

Em um estudo de flexão em barras de cerâmica à base de zircônia para infraestruturas, os autores concluíram que o dano de superfície produzido pelo procedimento de fresagem CAD-CAM reduziu significativamente sua resistência flexural (1166,3 ± 162,2 MPa para a zircônia polida e 743,3 ± 213,7 MPa para a zircônia usinada), resultando em falhas inesperadas em tensões muito abaixo do que a resistência esperada do material (WANG; ABOUSHELIB; FEILZER, 2008).

Fraga et al. (2015) avaliando a resistência flexural de uma cerâmica vítrea reforçada por leucita, demonstraram que a usinagem reduziu em cerca de 27% sua resistência quando comparada à usinagem seguida de polimento. Uma redução significativa na resistência à fadiga de cerâmicas de diferentes microestruturas submetidas à usinagem foi relatada por Fraga et al. (2017) em comparação com suas equivalentes totalmente polidas. A maior redução na resistência à fadiga devido à usinagem foi observada na Y-TZP (40%), seguida por dissilicato de lítio (33%) e leucita (29%).

Romanyk et al. (2019) analisando os efeitos da usinagem na resistência flexural biaxial de cerâmicas de dissilicato de lítio, concluíram que os defeitos introduzidos pela usinagem diminuíram significativamente a resistência desses materiais (44% para IPS e.max CAD; 46% para Celtra® Duo e 21% para Vita Suprinity), e que os tratamentos térmicos para cristalização completa ou *annealing* não foram suficientes para reparar os danos ocasionados.

Romanyk et al. (2020), avaliando a resistência flexural de uma cerâmica totalmente cristalizada (dissilicato de lítio reforçada com zircônia - Celtra® Duo) observaram que a usinagem resultou em danos significativos de limitação de resistência, que foram parcialmente mitigados por tratamento térmico pós-usinagem (redução de 53% da resistência em relação ao grupo polido para o grupo sem tratamento térmico e de 19% em relação ao grupo polido para o grupo com tratamento térmico).

2.4 COMPORTAMENTO À FADIGA DE CERÂMICAS

Cerâmicas são materiais friáveis, ou seja, suportam pouca ou nenhuma deformação plástica ou elongamento antes de fraturar. Esta propriedade, aliada à presença de defeitos internos ou superficiais que estejam localizados em áreas sujeitas a tensões de tração é especialmente crítica, porque as tensões nas pontas desses defeitos são concentradas e podem causar formação, propagação de trincas e quebra de ligações. Como não há alívio das tensões pela deformação plástica na extremidade do defeito, as trincas se desenvolvem à medida que a tensão aumenta, podendo ocorrer a fratura (WISKOTT; NICHOLLS; BELSER, 1995; ANUSAVICE, 2005; ZHANG, SAILER, LAWN, 2013).

Além dos defeitos já incorporados ao material, trincas podem ser geradas durante a fabricação e o ajuste das restaurações cerâmicas, ou após sua instalação, durante a mastigação. Podem assumir a forma de defeitos microestruturais no interior da cerâmica, provenientes da usinagem, de danos por jateamento, a partir de facetas de desgaste e danos de contato nas superfícies oclusais ou de cimentação ou, ainda, de microcontatos com objetos pontiagudos (ZHANG, SAILER, LAWN, 2013).

A fratura ocorre quando o fator de intensidade de tensão na ponta da trinca (K_I) atinge um nível crítico (K_{IC}), definido pela combinação da tensão aplicada (σ), o comprimento da trinca (α), e uma constante adimensional (Y), que depende do modo de tensão, formato e dimensões do material e a geometria da trinca (WISKOTT; NICHOLLS; BELSER, 1995; GONZAGA et al., 2011). Ou seja, sob cargas contínuas, essas microtrincas se fundem a uma trinca crescente que enfraquece a restauração de maneira insidiosa, resultando em uma fratura catastrófica a partir de um ciclo de carregamento final que excede a capacidade mecânica remanescente do material (WISKOTT; NICHOLLS; BELSER, 1995).

Outro aspecto importante sobre a presença de defeitos em materiais cerâmicos é que eles podem apresentar um crescimento lento e estável ao longo do tempo. Esse fenômeno pode levar à degradação da resistência, diminuindo o tempo de vida das restaurações cerâmicas (GONZAGA et al., 2011). Tal fenômeno é conhecido como crescimento subcrítico de trincas (*'subcritical'* ou *'slow' crack growth* - SCG), onde falhas preexistentes crescem ao longo do tempo sob a presença de um fator de intensidade de tensão abaixo do nível crítico, até que o tamanho da trinca e o nível de tensão resultem em um fator de intensidade que excede a tenacidade à fratura do material (DE AZA et al., 2002, GONZAGA et al., 2011; BONFANTE; COELHO, 2016). Dois mecanismos de crescimento subcrítico de trincas são observados - a corrosão sob tensão e a fadiga cíclica, que podem ocorrer simultaneamente ou individualmente (PARIS; ERDOGAN, 1963). Na maioria dos biomateriais cerâmicos, estes mecanismos se associam. A tensão na ponta da trinca e a presença de moléculas de água ou fluido corporal, temperatura e outras variáveis (redução da energia superficial na ponta da trinca) resultam na ruptura das ligações de óxido metálico do material, com a consequente formação de hidróxidos. Assim, como consequência da corrosão sob tensão, um defeito pode atingir seu tamanho crítico e resultar em fratura catastrófica (RITTER, 1995; DE AZA et al., 2002). O ambiente oral apresenta diversos elementos que favorecem o SCG, como água proveniente da saliva ou fluidos dentinários, cargas mastigatórias, variações de temperatura e pH (GONZAGA et al., 2011). Assim, é fundamental que os testes mecânicos simulem tais condições, para que forneçam informações mais acuradas sobre o comportamento dos materiais (BONFANTE; COELHO, 2016).

No caso da cerâmica à base de zircônia, simultaneamente com o SCG, outro fenômeno denominado degradação em baixa temperatura (*low temperature degradation* – LTD) foi relatado (CHEVALIER; CALE`S; DROUIN, 1999; KELLY; DENRY, 2008); no entanto, uma vez que a presente tese tem foco maior nas vitrocerâmicas, este evento não será discutido com detalhes.

2.5 CIMENTAÇÃO ADESIVA DE RESTAURAÇÕES TOTALMENTE CERÂMICAS

John McLean (1988) postulou que a estrutura dentária atuaria como um "núcleo reforçador" para restaurações cerâmicas quando cimentadas adesivamente, sendo o cimento resinoso responsável por uma "união sinérgica" que conferiria resistência ao sistema. Dois estudos de comparação clínica direta evidenciaram que a cimentação adesiva de restaurações monolíticas de cerâmicas vítreas é essencial para otimizar a longevidade das mesmas, demonstrando taxas de sobrevivência clínicas significativamente maiores em comparação com sistemas de cimento ácido-base (VAN DIJKEN; HÖGLUND-ABERG; OLOFSSON, 1998; MALAMENT; SOCRANSKY, 1999).

Cimentos resinosos apresentam composição e características similares aos compósitos restauradores convencionais e consistem em partículas inorgânicas de reforço embebidas em uma matriz orgânica, como por exemplo Bis-GMA, TEGDMA, UDMA. A retenção da restauração cerâmica à estrutura dental e o preenchimento das fendas marginais entre a restauração e o dente dependerão da capacidade do agente cimentante aderir à superfície da cerâmica e ao substrato dental, bem como do seu grau de escoamento (BLATZ; SADAN; KERN, 2003; THOMPSON et al., 2011).

A união entre cerâmicas dentárias e materiais resinosos tem sido extensivamente investigada na literatura odontológica, tanto no contexto de sistemas para reparo de cerâmicas (HAMMOND; SWIFT JR; BRACKETT, 2009; KOCAAĞAOĞLU; GÜRBULAK, 2015) quanto na melhoria da retenção de restaurações totalmente cerâmicas no ambiente bucal (KIM et al., 2005; VAN DEN BREEMER; GRESNIGT; CUNE, 2015). A união entre cerâmica e cimento é dependente de interações físicas e químicas. A interação física pode ser melhorada pelo aumento na rugosidade superficial da cerâmica, resultando em maior área disponível para adesão e potencial para embricamento micromecânico, e consequentemente melhor molhamento desta superfície pelo cimento resinoso (ADDISON; MARQUIS; FLEMING, 2008). Quimicamente, a interação entre a resina e a cerâmica é mediada por ligações de hidrogênio e forças de Van der Waals, mas pode ser aumentada com a ligação covalente a silanos organofuncionais aplicados na superfície da cerâmica (BLATZ; SADAN; KERN, 2003). Primers à base de silano também conferem resistência à degradação da ligação de resina-cerâmica exposta à umidade e às mudanças térmicas intraorais (MATINLINNA et al., 2004; BRENTEL et al., 2007). A modificação física e química de uma superfície cerâmica depende de sua composição, que determinará a técnica para aumentar a rugosidade de superfície utilizada e a escolha do primer a ser empregado (BLATZ; SADAN; KERN, 2003).

Cerâmicas vítreas, ácido-sensíveis (cerâmicas feldspáticas, leucíticas e dissilicato de lítio) sofrem degradação da matriz vítrea superficial pelo ácido fluorídrico (HF), produzindo um padrão topográfico que favorece a ligação micromecânica. Além disso, devido às suas características moleculares bifuncionais, a aplicação do agente de ligação silano na superfície da cerâmica favorece a união química entre a cerâmica e os materiais resinosos, promovendo maior molhabilidade do cimento na superfície cerâmica e unindo os óxidos de sílica presentes nas cerâmicas à matriz orgânica dos cimentos resinosos por meio de ligações siloxanas (DELLA BONA; SHEN; ANUSAVICE, 2004; BRENTEL et al., 2007; THOMPSON et al., 2011).

A Odontologia segue uma tendência de simplificação dos procedimentos clínicos. Buscando ampliar a versatilidade dos primers cerâmicos, os fabricantes combinam outros componentes, além do silano. Nesse sentido foram introduzidos adesivos multi-modo ou universais contendo silano, além de monômeros (hidrófilos e hidrófobos), MDP (monômeros fosfatado ácido: 10-methacryloyloxydecyl dihidrogênio fosfato), água e etanol. Está indicado como agente adesivo para esmalte e dentina e como primer cerâmico. Entretanto, o uso desses adesivos multi-modo na superfície cerâmica não se mostrou tão efetivo quanto a técnica convencional (HF seguido de silano) (YOSHIHARA et al., 2016; MURILLO-GÓMEZ; RUEGGEBERG; DE GOES, 2017).

Recentemente, um primer autocondicionante foi introduzido no mercado, à base de polifluoreto de amônio e silano trimetoxipropil metacrilato, para ser utilizado em passo único antes da cimentação. O polifluoreto de amônio tem acidez mais suave em comparação com o ácido fluorídrico, o que resulta em um padrão de condicionamento mais brando, porém com desempenho adesivo semelhante à técnica convencional (HF seguido de silano) (ROMÁN-RODRÍGUEZ et al. 2017). O novo material visa eliminar o potencial tóxico do ácido fluorídrico, reduzir o tempo necessário e a sensibilidade da técnica de condicionamento ácido com os métodos convencionais (EL-DAMANHOURY; GAINTANTZOPOULOU, 2017).

Uma consequência dos tratamentos para aumentar a rugosidade superficial da cerâmica é a introdução de uma nova textura, consistindo de uma população microscópica de defeitos sobreposta à rugosidade da superfície macroscópica. Esta modificação da população de defeitos de superfície pode impactar o desempenho de restaurações de cerâmica pura, já que os modos de falha clínica foram identificados como resultantes da propagação de um defeito crítico na superfície interna da restauração durante a função (ADDISON; MARQUIS; FLEMING, 2008; FRAGA et al., 2015, VENTURINI et al., 2018).

Clinicamente, o ambiente dos defeitos superficiais é influenciado pelo agente cimentante resinoso, que tem demonstrado ter um efeito significativo de aumento na resistência (MARQUIS, 1992, PAGNIANO et al., 2005, FLEMING et al., 2006, FLEMING et al., 2017, BARBON et al., 2018). Diversos estudos buscam esclarecer esse efeito. Marquis (1992) sugeriu que o cimento resinoso modificaria a população de falhas superficiais por um processo de cicatrização de defeitos, aumentando a resistência à fratura. Nathanson (1993) propôs que a contração de polimerização dos cimentos resinosos "tensionaria" as moléculas da cerâmica, aproximando-as e proporcionando maior resistência. Em um estudo sobre a deflexão de discos de cerâmica feldspática aderidas ao cimento resinoso e sua resistência flexural, os autores encontraram uma forte correlação positiva, indiretamente inferindo que a contração de polimerização teria um efeito de reforço deste material cerâmico (FLEMING et al., 2017). Wang et al. (1995) descrevem um fenômeno a que denominam “constrição de Poisson”: quando as resinas ficam restritas em camadas finas nas trincas, a resposta mecânica pode ser significativamente alterada. Durante a flexão biaxial, a resina dentro de uma trinca se estende perpendicularmente à face da trinca, produzindo uma contração compensatória de Poisson paralela à superfície da trinca. Essa contração é restrita pela espessura da resina em comparação com a cerâmica mais rígida, que apresenta uma menor relação de Poisson, e então observa-se um aumento efetivo na rigidez da resina. A resina na trinca, portanto, tem um comportamento mais semelhante à cerâmica. Mecanismos de aumento da resistência cerâmica independentes da severidade do defeito individual, mas sensíveis à textura da superfície, foram identificados por Addison, Marquis e Fleming (2008), que sugerem uma dependência na criação de uma camada híbrida interpenetrada de resina-cerâmica, dependente do módulo de elasticidade do cimento resinoso (módulos de elasticidade mais altos seriam favoráveis ao fortalecimento cerâmico) (ADDISON; MARQUIS; FLEMING, 2007; ADDISON; MARQUIS; FLEMING, 2008).

A pressão exercida durante o assentamento da cerâmica também foi investigada. Os autores estudaram o efeito da tensão e duração da carga aplicada durante a cimentação na resistência flexural biaxial de uma cerâmica vítrea reforçada por leucita. Foram investigadas as condições: pressão de cimentação de 30 N por 300 s (156,2 MPa), 5 N por 300 s (152,2 MPa), 30 N por 60 s (142,5 MPa) e 5 N por 60 s (163,5 MPa). O maior reforço foi observado quando uma carga de assentamento de 5 N foi aplicada por um período curto (60 s). Os autores atribuem este achado a que, durante a aplicação da carga, os defeitos da superfície da cerâmica ficam em tensão e a deformação elástica gerada atua 'abrindo' os defeitos. Também sugerem que a carga de assentamento possa afetar as características reológicas do cimento (ADDISON; SODHI; FLEMING, 2010).

May et al. (2012) investigaram o efeito da adesão ao cimento resinoso e sua espessura na resistência à fratura de coroas de cerâmica feldspática. Encontraram um efeito de reforço da adesão à restauração cerâmica, entretanto esse efeito foi perdido quando a espessura do cimento foi maior que 450 µm. Com a espessura de cimento de 50 µm as coroas que estavam aderidas ao substrato suportaram uma carga duas vezes maior (673,5 ± 88,4 N) do que as não aderidas com a mesma espessura de cimento (308,2 ± 98,8 N), bem como, o dobro da carga para fratura das coroas cimentadas com espessura de 500 µm aderidas (300,6 ± 41,5 N) ou não aderidas (233,2 ± 49,0 N). Um estudo sobre a influência da espessura de cimento resinoso no comportamento à fadiga da cerâmica feldspática, demonstrou que uma espessura de 50 µm proporcionou valores de resistência superiores do que a espessura de 500 µm (246,4 ± 22,9 N *vs* 158,9 ± 22,9 N) (MAY et al., 2015).

De Kok et al. (2017), estudaram o efeito da rugosidade da cerâmica de dissilicato de lítio para CAD-CAM em suas propriedades mecânicas. Foram comparados grupos de dissilicato de lítio polidos e jateados com óxido de alumínio em quatro situações: teste estático somente a cerâmica (jateados 254,3 ± 79,1 N; polidos 669,8 ± 96,5 N); teste estático cerâmica com cimento, sem adesão ao substrato de resina epóxica (jateados 237,1 ± 46.1 N; polidos 385,9 ± 118,3 N); teste estático cerâmica aderida ao substrato de resina epóxica (jateados 1559,1 ± 253,2 N; polidos 1632,3 ± 225,8 N), teste de fadiga cerâmica aderida ao substrato de resina epóxica (rugosos 877,5 ± 131,3 N; polidos 902,5 ± 140,9 N). Verificaram um efeito negativo de uma superfície interna rugosa quando não está adequadamente unida ao substrato. Entretanto, quando há uma união satisfatória ao substrato, a resistência à fratura aumenta significativamente e o efeito da rugosidade interna desaparece.

Barbon et al. (2018) também constataram uma melhor resistência mecânica da cerâmica feldspática disponível para o sistema CAD-CAM quando aderida ao cimento resinoso. Foram comparados discos recobertos com uma camada de cimento resinoso e discos sem cobertura (controle) na situação de envelhecimento artificial acelerado (EAA) ou sem envelhecimento. A cerâmica não recoberta submetida ao EAA (104-113 MPa) não teve diferença significante na resistência flexural biaxial comparada com o grupo controle (109-118 MPa). O recobrimento com cimento resinoso da cerâmica aumentou sua resistência flexural (139-148 MPa) em comparação com o grupo controle. Esse aumento foi ainda mais considerável quando os espécimes de cerâmica recobertos com cimento resinoso foram submetidos ao EAA (153-163 MPa).

Fraga et al. (2018) avaliaram a influência da estratégia de cimentação e de tratamentos de superfície da zircônia em seu comportamento à fadiga e verificaram que quando esta cerâmica é jateada com partículas de óxido de alumínio (2227 ±149 N) ou óxido de alumínio revestido por sílica (2133 ± 235 N) e cimentada adesivamente, as cargas de fratura à fadiga são aumentadas em comparação à zircônia cimentada adesivamente sem tratamento de superfície (1800 ± 293 N). Porém, quando cimentadas com cimento de fosfato de zinco, sua resistência à fadiga é bastante diminuída (680 ± 101 N).

Spazzin et al. (2017) utilizaram preparações experimentais de cimentos resinosos, com modificações na proporção de monômeros da matriz, alcançando diferentes módulos elásticos e investigaram sua influência no reforço da cerâmica feldspática recoberta. Nesse estudo, o aumento do módulo elástico do cimento reduziu a variabilidade da resistência e a tensão que atinge a estrutura cerâmica, e algumas vezes alterou a origem da falha dos espécimes. Cimentos com módulos elásticos alto (13.3 GPa) e intermediário (6.6 GPa) tiveram efeito semelhante no fortalecimento da cerâmica, superior ao cimento com baixo módulo elástico (2.6 GPa).

Coelho et al. (2019) avaliaram a influência do pré-aquecimento de diferentes resinas compostas na viscosidade e no fortalecimento de uma cerâmica feldspática. Eles encontraram efeito de reforço da cerâmica semelhante entre os compósitos pré-aquecidos avaliados e o cimento resinoso usado como controle, independentemente do módulo de elasticidade. No entanto, a espessura e a viscosidade dos compósitos pré-aquecidos foram maiores do que o cimento resinoso, e dependentes de cada material.

Barbon et al (2019) investigaram a influência de agentes cimentantes resinosos experimentais com diferentes teores de carga inorgânica na resistência de uma cerâmica feldspática. Os autores verificaram que quanto maior o conteúdo inorgânico, maior o módulo de elasticidade, a viscosidade, a espessura do filme e o efeito de fortalecimento da cerâmica. Entretanto, a resistência de união foi menor caso um sistema adesivo não fosse empregado conjuntamente.

Como demonstrado nessa revisão de literatura, diversos estudos avaliaram diferentes propriedades físicas do cimento no fortalecimento das cerâmicas, entretanto a viscosidade foi apenas um dos fatores abordados, mas seu papel isolado não foi investigado. Espera-se que cimentos com menor viscosidade, mais fluidos, consigam penetrar melhor nas irregularidades superficiais, formando uma película mais fina, gerando menor contração de polimerização, reduzindo assim a possibilidade da criação de regiões sem suporte sob a cerâmica e fendas marginais (DI FRANCESCANTONIO et al., 2013; MAY et al., 2012).

3 ARTIGO 1 – EFFECT OF MILLING, FITTING ADJUSTMENTS, AND HYDROFLUORIC ACID ETCHING ON THE STRENGTH AND ROUGHNESS OF CAD-CAM GLASS-CERAMICS: A SYSTEMATIC REVIEW AND META-ANALYSIS.

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Effect of milling, fitting adjustments, and hydrofluoric acid etching on the strength and roughness of CAD-CAM glass-ceramics: A systematic review and meta-analysis.

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ABSTRACT

Statement of problem. Whether procedures performed before the cementation of computer-aided design and computer-aided manufacturing (CAD-CAM) glass-ceramic restorations, including milling, fitting adjustment, and hydrofluoric acid etching, introduce defects on the ceramic surface that affect the mechanical and surface properties is unclear.

Purpose. A systematic review and meta-analysis were conducted to assess the effect of milling, fitting adjustments, and hydrofluoric acid etching (HF) on the flexural strength and roughness (Ra) of CAD-CAM glass-ceramics.

Material and methods. Literature searches were performed up to June 2020 in the PubMed/MEDLINE, Web of Science, and Scopus databases, with no publication year or language limits. The focused question was "Do milling, fitting adjustments, and hydrofluoric acid etching affect the flexural strength and roughness of CAD-CAM glass-ceramics?" For the meta-analysis, flexural strength and Ra data on milling, fitting adjustment, and HF etching versus control (polishing) were analyzed globally. A subgroup analysis assessed the effect of etching parameters (HF concentration and time) on the flexural strength and roughness of CAD-CAM glass-ceramics with different microstructures. Comparisons were performed with random-effect models at 5% significance.

Results. Fourteen studies from 2764 potentially relevant records were included in the qualitative syntheses, and 12 in the meta-analysis. Milling and fitting adjustments increased roughness and reduced the flexural strength of CAD-CAM glass-ceramics. The effect of HF etching was dependent on the glass-ceramic microstructure, HF concentration, and etching time. For feldspathic and leucite-reinforced ceramics, HF 5% applied for between 30 and 120 seconds increased roughness without affecting flexural strength. For lithium disilicate glass-ceramics, HF concentrations above 4.9% used for 20 seconds or more reduced the strength without affecting the surface roughness.

Conclusions. The flexural strength of CAD-CAM glass-ceramic is reduced by grinding procedures such as milling and fitting adjustment. Ceramic microstructure, HF concentration, and etching time determined the effect of hydrofluoric acid etching on the flexural strength and surface roughness of glass-ceramic materials.

CLINICAL IMPLICATIONS

Treatment of the intaglio surface of ceramic restorations before cementation may affect mechanical and surface properties. Grinding glass-ceramics with diamond tools, such as in CAD-CAM milling and in fitting adjustments, reduces the flexural strength of the glass-ceramic. The effect of the hydrofluoric acid etching on flexural strength and roughness depends on the material microstructure, HF concentration, and etching time.

INTRODUCTION

Glass-ceramics are extensively used for indirect restorations, especially in esthetic situations, because of their ability to mimic dental tissues, biocompatibility, and biomechanical properties.[1-3] The composition of dental glass-ceramics has been improved by adding crystalline particles such as aluminum oxide, leucite, and lithium disilicate to increase strength and fracture toughness.[4,5] Computer-aided design and computer-aided manufacturing (CAD-CAM) systems use standardized ceramic blocks, resulting in a more homogeneous and reliable material.[2,6]

Brittle ceramics support little or no plastic deformation before fracturing,[7] with their strength strongly dependent on stress-concentrating defects.[8,9] Finite element analysis has shown that a load applied on the occlusal surface of a single crown produces tensile stresses on the cementation surface.[10] Fractographic analysis of clinically failed crowns showed that defects located at the cementation zone have been responsible for starting a fracture.[8,9,11,12] The fabrication of ceramic restorations and surface treatments before cementation can induce defects in this critical area, affecting the roughness and strength of the restoration.[13-16]

CAD-CAM milling is a subtractive process in which the prefabricated ceramic block is shaped in the desired design through the cutting edges of the relatively hard particles present on the grinding tool surface.[17] This process induces strain at the ceramic surface which precedes crack propagation and chip formation, resulting in a rougher surface[18-21] and creating surface and subsurface damage.[13,22] Glass-ceramics are susceptible to such damage accumulation as they have relatively low fracture toughness and low damage tolerance.[4,7] Furthermore, a ceramic restoration may require clinical adjustment, typically performed with a diamond rotary instrument in order to improve fit.[23] As in CAD-CAM milling, the abrasive diamond particles may introduce defects on the ceramic surface.[23-28] As a result, the strength of the milled or ground restoration may significantly differ from the true strength of the ceramic substrate since the distribution and the character of the surface and subsurface defects, stress concentrators, and transient and residual stress states influence this characteristic.[7,13,15,16,29,30]

The clinical success and longevity of ceramic restorations relies on a suitable bond between the ceramic and dental substrate.[31] Adequate surface roughness is necessary for cement interlocking and enhanced bonding.[32-35] Hydrofluoric acid selectively etches the glassy phase of vitreous ceramic, exposing the silicon oxides, thereby promoting an increase in the surface energy before application of the silane agent (chemical bond) and in turn increasing the actual surface area for mechanical interlocking with a resin-based cement.[36,37] Substrate constitution, acid concentration, and etching time may influence the impact of acid etching on the mechanical and surface properties of glass-ceramics.[15,38-45]

This systematic review and meta-analysis evaluated the effect of procedures performed on the intaglio surface of an indirect restoration before cementation, specifically milling, fitting adjustments, and hydrofluoric acid etching, on the flexural strength and roughness of CAD-CAM glass-ceramics. The null hypotheses were that CAD-CAM milling, fitting adjustments, and hydrofluoric acid etching would not influence the flexural strength and roughness of CAD-CAM glass-ceramics.

MATERIAL AND METHODS

This systematic review was designed according to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) recommendations.[46] A protocol was determined before the literature search and registered on the Open Science Framework (OSF), available at osf.io/957ef.

The population, intervention, comparison, and outcome (PICO) question was: Do milling, fitting adjustments, and hydrofluoric acid etching affect the flexural strength and roughness of CAD-CAM glass-ceramics? The population compromised CAD-CAM glass-ceramics (feldspathic-based ceramic, leucite- and lithium disilicate-reinforced glass-ceramic, and zirconia-reinforced lithium silicate). The intervention was defined as the procedures performed on the intaglio surface of an indirect restoration before cementation, specifically CAD-CAM milling, fitting adjustments, and hydrofluoric acid etching. Polished specimens were used as the comparison. The outcomes of flexural strength and roughness (Ra) were evaluated.

The PubMed/MEDLINE, Web of Science, and Scopus databases were used to identify potentially relevant studies up to June 2020 (last search update). The search strategy was independently conducted by 2 of the authors (M.M.M., S.F.) and compromised specific medical subject headings (MeSH) and key words (“free-text words”) The search strategy, described in

Table 1, was adapted for the Web of Science and Scopus databases, and the results were cross-checked to eliminate duplicates.

The search strategy and the study selection were independently performed by 2 reviewers (M.M.M., S.F.). In the first step, the studies were selected by title and abstract based on the following inclusion criteria: in vitro studies which evaluated the effect of the procedures performed before cementation, specifically milling, fitting adjustments, and hydrofluoric acid etching, on the flexural strength and roughness (Ra) of CAD-CAM glass-ceramics. In the second step, the studies previously identified by title and abstract were fully analyzed and excluded according to the criteria: did not use CAD-CAM glass-ceramic; did not have a proper control group (polished specimens); did not evaluate the interventions (milling, fitting adjustment, and hydrofluoric acid etching) or the main outcomes (flexural strength and roughness) investigated in this systematic review; evaluated ceramic specimens cemented to a substrate; and the roughness was reported by a parameter other than Ra.

A manual search in the reference list of the included studies was also performed. Disagreements between the reviewers were resolved through discussion and judgment by a third reviewer (L.G.M.). The interexaminer agreement (Kappa coefficient) was calculated for the first (0.84) and the second (0.80) steps.

The risk of bias of the included studies was evaluated based on previous studies[47-49] and considered the following parameters: sample size calculation; randomization of the specimens; specimen preparation described in a reproducible way; polishing protocol clearly specified; intervention protocol (milling, fitting adjustment, and hydrofluoric acid etching) clearly described; specimens prepared by a single operator; and adequate description of the test parameters used to evaluate the outcomes (flexural strength and roughness). Values from 0 to 2 were attributed for each parameter: 0, if the parameter was clearly described; 1, if the parameter execution was reported but the execution accuracy was unclear; and 2, if the parameter was not reported. The risk of bias was considered low when the sum of the values ranged between 0 to 4, medium between 5 to 9, and high between 10 to 14.[49]

Data from the eligible studies were independently collected by 2 reviewers (M.M.M., S.F.), and then compiled and discussed for consensus. Distinct meta-analyses were performed to evaluate the single effect of milling, fitting adjustment, and hydrofluoric acid etching on the flexural strength and roughness of CAD-CAM glass-ceramics by using polished groups as the comparison. Regarding hydrofluoric acid etching, subgroup analyses were conducted to understand the effect of HF concentration and etching times on the outcomes.

The analyses were carried out in a software program (Review Manager, v5.3; Cochrane Collaboration) by using random effects at a significance level of 5% (Z test). Pooled effect estimates were obtained by comparing the means of each of the flexural strength and roughness values, and these were expressed as raw mean differences among the groups. The heterogeneity among studies was evaluated by the Cochrane Q test, in which $P<.1$ was considered statistically significant, and by the inconsistency I^2 test, where values higher than 50% were considered indicative of substantial heterogeneity.[50]

In the case of studies which did not provide mean and standard deviation values and sample size, the corresponding authors were contacted up to 3 times by e-mail. These studies were then only descriptively analyzed if no answer was obtained.

RESULTS

The search strategy identified 4552 potentially relevant records. After removing duplicates, 2764 studies were analyzed by title and abstract. From these, 2722 did not meet the inclusion criteria, and 42 were deemed eligible for full-text analysis. Fourteen studies were included in the qualitative syntheses, and 12 in the meta-analysis. Manual searching through the reference lists of included studies resulted in 1 additional article (Fig. 1).

Data analyses were performed qualitatively (descriptive summary) and quantitatively (meta-analyses) when appropriate for each of the procedures (milling, fitting adjustment, and hydrofluoric acid etching) and the outcomes (flexural strength and roughness - Ra). Studies which evaluated more than 1 glass-ceramic material, fitting adjustment protocol, or hydrofluoric acid etching condition were inserted more than once in each meta-analysis.[49]

Table 2 describes the studies which evaluated the effect of milling and fitting adjustment on the flexural strength and/or roughness of CAD-CAM glass-ceramics.[15,16,18,19,21,26-28,30] The specimens (disks and bars) in most of the studies were produced by CAD-CAM milling in a CEREC system (Dentsply Sirona).[15,16,18,19,21,30] Other studies simulated fitting adjustments with diamond rotary instruments.[26-28]

Four studies[15,16,26,30] on the effect of milling and fitting adjustment on the flexural strength of CAD-CAM glass-ceramics were included in the meta-analyses. The global analysis showed that grinding with diamond rotary instruments had a negative impact on the flexural strength of glass-ceramics (Fig. 2), with high heterogeneity (I^2=97%) among the studies. Only the studies regarding CAD-CAM milling are reported in Fig. 3, which also presented high heterogeneity.

The meta-analyses concerning the effect of grinding on the ceramic roughness included 5 studies[15,16,18,19,30] which used CAD-CAM milling to produce the ceramic specimens. Some articles were excluded because they did not present numerical values for the Ra mean or standard deviation[27,28] or the sample size.[21] The global analysis for Ra showed a significant difference between milling and polishing, favoring the control (Fig. 4), with high heterogeneity among the studies (I^2=98%).

Table 3 describes the studies which evaluated the effect of hydrofluoric acid etching on the flexural strength and/or roughness of CAD-CAM glass-ceramics.[15,39-44] The HF concentration ranged from 1% and 10% with the application time of 20 seconds up to 180 seconds, depending on the ceramic material.

Seven studies[15,39-44] were included in the meta-analysis regarding the effect of the HF etching on the flexural strength of CAD-CAM glass-ceramic. The global analysis indicated that the hydrofluoric acid etching had a negative impact on the flexural strength of glass-ceramics (Fig. 5). High heterogeneity (I^2=95%) was reported in this analysis.

Despite the global analysis having indicated a negative effect of HF etching on the flexural strength, subgroup analyses showed that HF concentrations above 5% applied for between 30 and 120 seconds did not affect the material strength for feldspathic or leucite-reinforced glass-ceramics (Fig. 6). However, HF concentrations above 4.9% applied for 20 seconds or more reduced the flexural strength of lithium disilicate glass-ceramics (Fig. 7).

Five studies[15,39-42] showing a significant difference between HF etching and polishing, favoring the control (Fig. 8), with high heterogeneity among the studies (I^2=100%) were included in the global analysis for roughness (Ra). In the subgroup analysis, all the variances in HF concentration and etching time resulted in higher Ra values for feldspathic and leucite glass-ceramic (Fig. 9) when compared with the control. Regarding lithium disilicate (Fig. 10), HF 4.9% to 5% applied for 20 seconds did not significantly increase Ra values when compared with those of the polishing groups.

Most of the studies presented a medium risk of bias, while a high and a low risk of bias were observed in 1 study each (Table 4). The sample size calculation was not reported in any of the studies, and many did not report how randomization had been performed.

DISCUSSION

This systematic review showed that procedures performed before the cementation of an indirect glass-ceramic restoration – milling, fitting adjustments, and hydrofluoric acid etching

– may increase surface roughness and reduce the fracture strength of CAD-CAM glass-ceramics. Therefore, the null hypotheses were rejected.

CAD-CAM milling creates a series of surface and subsurface damage in the cementation zone of the restoration,[22] increasing roughness[15,16,18,19,30] and reducing the ceramic strength.[15,16,30] The effect of CAD-CAM milling on the mechanical and surface properties of dental ceramics may depend on the microstructure of the material,[16] the diamond rotary instruments,[13,15] the milling order,[15] and/or the balance between crack population and the compressive stress layer generated around the crack groove.[20] These issues contribute to the high heterogeneity seen in the meta-analysis for flexural strength and roughness.

Defects on the intaglio surface of indirect ceramic restorations may also be introduced by clinical adjustments, often carried out to improve restoration fitting.[23] Internal adjustments with medium, fine, or extra fine diamond rotary instruments have been reported to reduce the fatigue failure load of lithium disilicate ceramic restorations adhesively cemented to a dentin-like material.[23] A systematic review[49] concluded that the diamond grit size of the grinding tool influences the mechanical behavior of an yttria-stabilized zirconia (Y-TZP) and that a fine grit (less than 50 µm) increases Y-TZP strength while medium and higher grits decrease strength. In the present systematic review, a subgroup analysis to evaluate the effect of the diamond size of the rotary instrument on the strength of glass-ceramics was not possible since only 1 of the included studies evaluated this condition.[26]

After CAD-CAM milling and fitting adjustments (if necessary), the internal surface of glass-ceramic restorations is commonly treated with hydrofluoric acid etching and silane agent to enhance resin cement bonding.[36,37] Recommendations for HF concentration and etching time depends on the glass-ceramic microstructure. For the feldspathic-based and the leucite-based ceramics mentioned in this systematic review, the manufacturers indicate 4.5% to 5% HF for 60 seconds.[51,52] The common recommendation for lithium disilicate and some zirconia-reinforced lithium silicate is to use 4.5% HF for 20 seconds.[52,53]

HF etching is a dynamic process controlled by surface chemical reactions, which are influenced by etching time and concentration, and by the glass-ceramic composition.[38] The subgroup analysis indicated that HF protocols of 5% for 60 seconds or more and 9% to 10% for 60 seconds significantly increased surface roughness for feldspathic- and leucite-based glass-ceramics, without affecting flexural strength. All the acid etching protocols for lithium disilicate glass-ceramics evaluated in the subgroup analysis reduced their flexural strength. No difference was found in the Ra values between the protocol indicated by the manufacturer (4.5%

HF for 20 seconds) and the polishing group. However, these values should be interpreted with caution since only a few studies with high heterogeneity were included in the analysis.

High heterogeneity was also seen in the global meta-analyses regarding the influence of the hydrofluoric acid etching on the flexural strength and roughness of CAD-CAM glass-ceramics. This may be explained by the different etching protocols and ceramic compositions. The heterogeneity was reduced to acceptable values (less than 50%, Fig. 7) in the lithium disilicate subgroup analysis performed with similar HF concentrations and etching times.

The risk of bias assessment of in vitro studies is challenging because of the absence of a defined protocol and the variability among the studies. The evaluation of the quality of the included studies was based on an adapted protocol from previous systematic reviews.[47-49] The majority of the included studies had medium risk of bias, which might imply that these studies did not control all the variables involved or did not report them properly. These results are consistent with other systematic reviews,[49,54-56] indicating that poor methodological reporting is a common problem.[57] However, poor reporting does not necessarily denote unsatisfactory quality of the study, but it does restrict the assessment and reliability of the methodological quality.[58]

Limitations of the present systematic review include the high heterogeneity of the global meta-analysis and the low number of papers included in the subgroup analysis, requiring caution in interpreting the results. Despite these limitations, the findings should suggest future studies on the effects of milling, fitting adjustment, and HF etching on the surface roughness and strength of CAD-CAM glass-ceramics.

CONCLUSIONS

Based on the findings of this systematic review and meta-analysis, the following conclusions were drawn:

1. Milling and fitting adjustments increased the roughness and reduced the flexural strength of CAD-CAM glass-ceramics.
2. HF concentrations above 5% used between 30 and 120 seconds did not affect the strength and increased the roughness of feldspathic and leucite glass-ceramics.
3. HF concentrations above 4.9% applied for 20 seconds or more reduced the flexural strength of lithium disilicate glass-ceramic without affecting the surface roughness.

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TABLES

Table 1. Search strategy used to identify relevant studies in PubMed/MEDLINE database

MESH and free text words
(((((((((((computer aided design) OR computer aided machining) OR computer aided manufacturing) OR cad-cam) OR cad/cam) OR cadcam) OR (((((((((ceramic) OR glass) OR leucite) OR lithium disilicate) OR feldspathic) OR feldspar) OR silica) OR zirconia reinforced lithium silicate)) OR (("Ceramics"[Mesh]) OR "Computer-Aided Design"[Mesh])))) AND ((((((((((((machin*) OR grind*) OR ground) OR bur) OR bur adjustment) OR mill) OR milled) OR cut) OR etch*) OR acid) OR hydrofluoric acid) OR (("Hydrofluoric Acid"[Mesh]))))) AND ((((((((((strength) OR resistance) OR fracture) OR fatigue) OR uniaxial) OR biaxial) OR roughness) OR Ra)) OR "Flexural Strength"[Mesh]))) AND polish*)

Table 2. Milling and fitting adjustments versus polishing: characteristics of included studies

Outcome	Study/year	Material	Brand	Grinding system/protocol	Flexural testing arrangement	Sample size
Flexural strength and roughness (Ra)	Fraga et al, 2015[15]	Leucite-reinforced glass-ceramic	IPS Empress CAD (Ivoclar Vivadent AG)	CAD-CAM milling, CEREC (Dentsply Sirona)	Biaxial (piston-on-three ball)	24
	Fraga et al, 2017[16]	Leucite-reinforced glass-ceramic	IPS Empress CAD (Ivoclar Vivadent AG)	CAD-CAM milling, CEREC (Dentsply Sirona)	Biaxial (piston-on-three ball)	20 (Ra for leucite and lithium disilicate)
		Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)			5 (flexural strength for lithium disilicate)
	Romanyk et al, 2019[30]	Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG);	CAD-CAM milling, CEREC (Dentsply Sirona)	Biaxial (ball-on-ring)	15
		Zirconia reinforced lithium silicate	Vita Suprinity (Vita Zahnfabrik); Celtra Duo (Dentsply Sirona)			

Flexural strength	Coldea et al, 2015[26]	Feldspathic-based ceramic Lithium disilicate-reinforced glass-ceramic	VITABLOCS Mark II (VITA Zahnfabrik) IPS e.max CAD (Ivoclar Vivadent AG);	Grinding with coarse (107-181 µm), medium (64-126 µm) or extra-fine (10-36 µm) diamond rotary instrument, for 2s in both transversal and longitudinal directions.	Uniaxial (three-point bending)	10
Roughness (Ra)	Kou et al, 2006[27]*	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	Grinding with 107-126 µm, 76 µm and 46 µm diamond rotary instruments for 30s each	-	5
	Al-Shammery et al, 2007[19]	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	CAD-CAM milling, CEREC (Dentsply Sirona)	-	5
	Aykent et al, 2010[28]*	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	Grinding with 46 µm and 25 µm diamond rotary instruments for 30s each	-	10

	Alao et al, 2017[21]*	Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	CAD-CAM milling, CEREC (Dentsply Sirona)	-	not reported
	Mota et al, 2017[18]	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	CAD-CAM milling, CEREC (Dentsply Sirona)	-	10
		Leucite-reinforced glass-ceramic	IPS Empress CAD (Ivoclar Vivadent AG)			
		Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)			
		Zirconia reinforced lithium silicate	Suprinity (VITA Zahnfabrik)			

*It was excluded from the meta-analysis because it did not present numerical values of Ra mean and standard deviation[27,28] or sample size.[21]

Table 3. Hydrofluoric acid etching versus polishing: characteristics of included studies

Outcome	Study/year	Material	Brand	HF%	HF time (s)	Flexural testing arrangement	Sample size
Flexural strength and roughness (Ra)	Zogheib et al. 2011[39]	Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	4.9%	20s 60s 90s 180s	Uniaxial (three-point bending)	15
	Fraga et al. 2015[15]	Leucite-reinforced glass-ceramic	IPS Empress CAD (Ivoclar Vivadent AG)	10%	60s	Biaxial (piston-on-three ball)	24
	Liu et al. 2015[40]	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	5%	30s 60s 120s	Biaxial (piston-on-three ball)	10
	Venturini et al. 2015[41]	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	1% 3% 5% 10%	60s	Uniaxial (three-point bending)	30
	Prochnow et al. 2017[42]	Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	1% 3% 5% 10%	20s	Uniaxial (three-point bending)	23
Flexural strength	Yi, Kelly 2011[43]	Feldspathic-based ceramic	VITABLOCS Mark II (VITA Zahnfabrik)	9%	60s	Biaxial (piston-on-three ball)	10

	Menees et al. 2014[44]	Lithium disilicate-reinforced glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	5% 9.5%	20s 120s	Uniaxial (three-point bending)	10

Table 4. Risk of bias of included studies

Study	Sample size calculation	Random	Specimen preparation	Polishing protocol	Intervention protocol	Operator	Test parameters	Total	Risk of bias
Fraga et al, 2015[15]	2	1	0	1	0	1	0	5	medium
Fraga et al, 2017[16]	2	0	0	0	0	0	0	2	low
Romanyk et al, 2019[30]	2	2	0	0	0	2	0	6	medium
Coldea et al, 2015[26]	2	2	0	1	0	2	0	7	medium
Kou et al, 2006[27]	2	1	1	1	0	0	0	5	medium
Al-Shammery et al, 2007[19]	2	1	1	0	0	1	0	5	medium
Aykent et al, 2010[28]	2	2	1	1	0	0	1	7	medium
Alao et al, 2017[21]	2	2	1	0	0	1	0	6	medium
Mota et al, 2017[18]	2	2	1	0	0	0	0	5	medium
Zogheib et al, 2011[39]	2	1	1	0	0	2	0	6	medium
Liu et al, 2015[40]	2	1	0	1	1	2	1	8	medium
Venturini et al, 2015[41]	2	1	0	1	0	2	0	6	medium
Prochnow et al, 2017[42]	2	1	0	0	0	2	0	5	medium
Yi e Kelly 2011[43]	2	1	0	1	1	2	0	7	medium
Menees et al, 2014[44]	2	2	1	1	1	2	1	10	high

FIGURES

Figure 1. Selection of study procedures according to PRISMA.

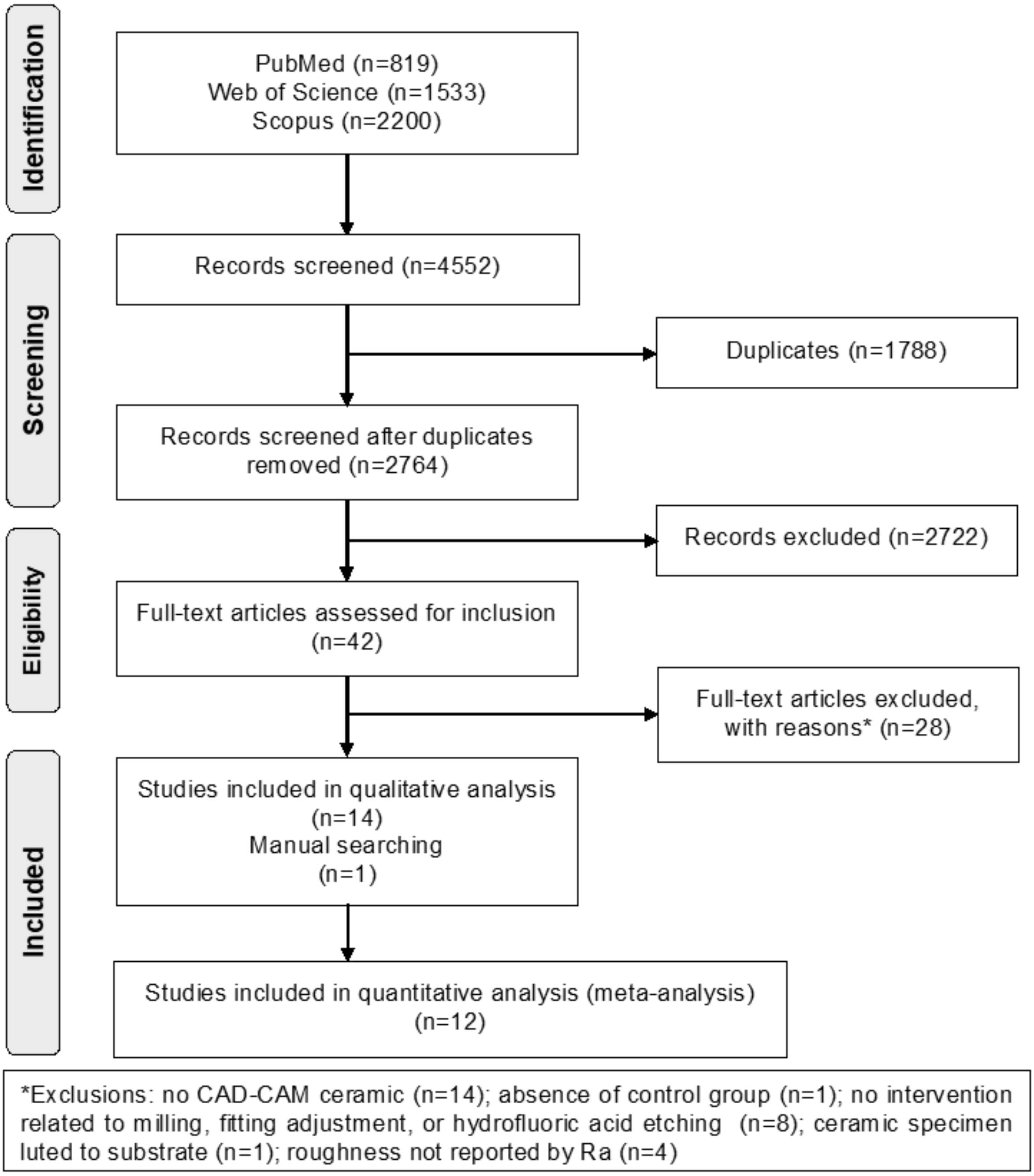


Figure 2. Forest plot for flexural strength analysis of CAD-CAM milling and fitting adjustment versus polishing (control) for glass-ceramics. F, feldspathic; L, leucite; LD, lithium disilicate; ZLS, zirconia-reinforced lithium silicate.

	Milling/fit adjustment			Polishing (control)				Mean Difference
Study or Subgroup	**Mean**	**SD**	**Total**	**Mean**	**SD**	**Total**	**Weight**	**IV, Random, 95% CI**
Coldea et al, 2015_F_coarse diamond_fit	83.83	12.4	10	122.18	6.36	10	10.7%	-38.35 [-46.99, -29.71]
Coldea et al, 2015_F_extra fine diamond_fit	93.18	15.67	10	122.18	6.36	10	10.7%	-29.00 [-39.48, -18.52]
Coldea et al, 2015_F_medium diamond_fit	87.79	17.41	10	122.18	6.36	10	10.6%	-34.39 [-45.88, -22.90]
Coldea et al, 2015_LD_coarse diamond_fit	194.23	48.06	10	397.23	56.63	10	8.8%	-203.00 [-249.04, -156.96]
Coldea et al, 2015_LD_extra fine diamond_fit	256.4	94.03	10	397.23	56.63	10	7.2%	-140.83 [-208.86, -72.80]
Coldea et al, 2015_LD_medium diamond_fit	198.27	55.91	10	397.23	56.63	10	8.5%	-198.96 [-248.28, -149.64]
Fraga et al, 2015_L_milling	122.12	14.11	24	166.48	25.81	24	10.6%	-44.36 [-56.13, -32.59]
Fraga et al, 2017_LD_milling	269.52	26.27	5	479.39	58.88	5	8.0%	-209.87 [-266.38, -153.36]
Romanyk et al, 2019_LD_milling	362	24	15	643	86	15	8.8%	-281.00 [-326.18, -235.82]
Romanyk et al, 2019_ZLS_Celtra_milling	220	16	15	409	81	15	9.1%	-189.00 [-230.78, -147.22]
Romanyk et al, 2019_ZLS_Suprinity_milling	321	65	15	405	121	15	7.0%	-84.00 [-153.51, -14.49]
Total (95% CI)			**134**			**134**	**100.0%**	**-124.95 [-156.26, -93.64]**

Heterogeneity: Tau²=2363.12; Chi²=288.25, df=10 (*P*<.001); I²=97%
Test for overall effect: Z=7.82 (*P*<.001)

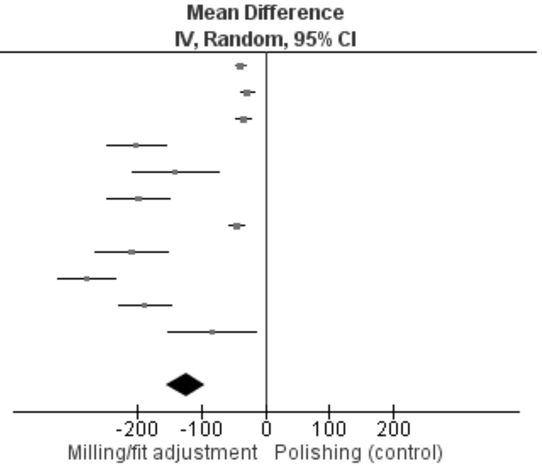


Figure 3. Forest plot for flexural strength analysis including just CAD-CAM milling studies for glass-ceramics. L, leucite; LD, lithium disilicate; ZLS, zirconia-reinforced lithium silicate.

Study or Subgroup	Milling Mean	Milling SD	Milling Total	Polishing (control) Mean	Polishing (control) SD	Polishing (control) Total	Weight	Mean Difference IV, Random, 95% CI
Fraga et al, 2015_L_milling	122.12	14.11	24	166.48	25.81	24	20.8%	-44.36 [-56.13, -32.59]
Fraga et al, 2017_LD_milling	269.52	26.27	5	479.39	58.88	5	19.7%	-209.87 [-266.38, -153.36]
Romanyk et al, 2019_LD_milling	362	24	15	643	86	15	20.1%	-281.00 [-326.18, -235.82]
Romanyk et al, 2019_ZLS_Celtra_milling	220	16	15	409	81	15	20.2%	-189.00 [-230.78, -147.22]
Romanyk et al, 2019_ZLS_Suprinity_milling	321	65	15	405	121	15	19.2%	-84.00 [-153.51, -14.49]
Total (95% CI)			**74**			**74**	**100.0%**	**-161.39 [-267.94, -54.85]**

Heterogeneity: Tau² = 14164.75; Chi² = 156.37, df = 4 (*P* < .001); I² = 97%
Test for overall effect: Z = 2.97 (*P* = .003)

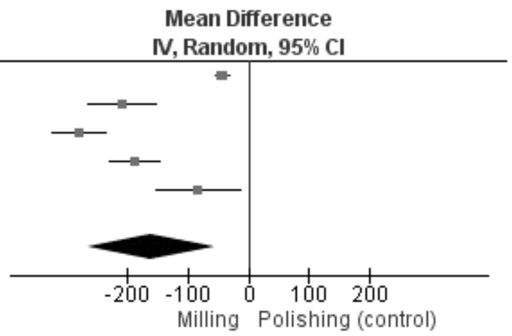


Figure 4. Forest plot for roughness (Ra) analysis of CAD-CAM milling versus polishing (control) for glass-ceramics. F, feldspathic; L, leucite; LD, lithium disilicate; ZLS, zirconia-reinforced lithium silicate.

Study or Subgroup	Milling Mean	Milling SD	Milling Total	Polishing (control) Mean	Polishing (control) SD	Polishing (control) Total	Weight	Mean Difference IV, Random, 95% CI
Al-Shammery et al, 2007_F_milling	4.45	2.01	5	1.5	0.59	5	2.7%	2.95 [1.11, 4.79]
Fraga et al, 2015_L_milling	1.37	0.18	24	0.04	0.01	24	10.3%	1.33 [1.26, 1.40]
Fraga et al, 2017_L_milling	1.59	0.17	20	0.08	0.03	20	10.3%	1.51 [1.43, 1.59]
Fraga et al, 2017_LD_milling	1.84	0.18	20	0.13	0.06	20	10.3%	1.71 [1.63, 1.79]
Mota et al, 2017_F_milling	1.88	0.41	10	0.54	0.09	10	9.8%	1.34 [1.08, 1.60]
Mota et al, 2017_L_milling	1.79	0.49	10	0.54	0.15	10	9.5%	1.25 [0.93, 1.57]
Mota et al, 2017_LD_milling	2.71	0.39	10	0.81	0.11	10	9.8%	1.90 [1.65, 2.15]
Mota et al, 2017_ZLS_milling	2.52	0.29	10	0.95	0.12	10	10.0%	1.57 [1.38, 1.76]
Romanyk et al, 2019_LD_milling	4.141	0.887	15	0.059	0.025	15	8.9%	4.08 [3.63, 4.53]
Romanyk et al, 2019_ZLS_Celtra_milling	4.566	0.642	15	0.062	0.022	15	9.5%	4.50 [4.18, 4.83]
Romanyk et al, 2019_ZLS_Suprinity_milling	3.191	0.866	15	0.046	0.021	15	8.9%	3.15 [2.71, 3.58]
Total (95% CI)			**154**			**154**	**100.0%**	**2.21 [1.86, 2.57]**

Heterogeneity: Tau²=.32; Chi²=553.47, df=10 (*P*<.001); I²=98%
Test for overall effect: Z=12.25 (*P*<.001)

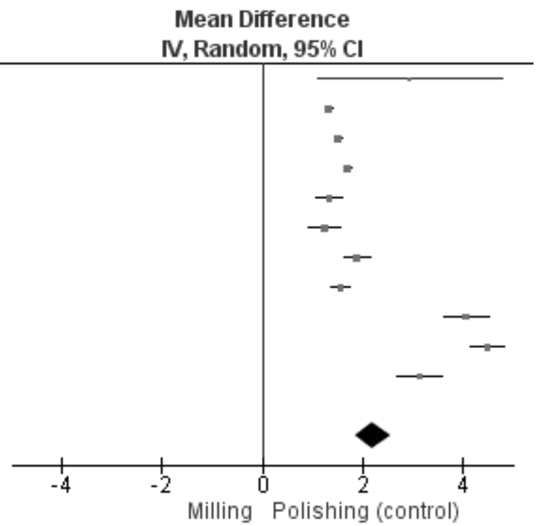


Figure 5. Forest plot for flexural strength analysis of hydrofluoric acid etching versus polishing (control) for glass-ceramics. F, feldspathic; L, leucite; LD, lithium disilicate.

Study or Subgroup	Hydrofluoric acid etching Mean	SD	Total	Polishing (control) Mean	SD	Total	Weight	Mean Difference IV, Random, 95% CI
Fraga et al, 2015_HF10%_60s_L	161.47	16.3	24	166.48	25.81	24	6.5%	-5.01 [-17.22, 7.20]
Liu et al, 2015_HF5%_120s_F	86.8	9.6	10	90	5.5	10	7.0%	-3.20 [-10.06, 3.66]
Liu et al, 2015_HF5%_30s_F	89	4.8	10	90	5.5	10	7.1%	-1.00 [-5.52, 3.52]
Liu et al, 2015_HF5%_60s_F	86.3	7.4	10	90	5.5	10	7.1%	-3.70 [-9.41, 2.01]
Menees et al, 2014_HF5%_120s_LD	333.8	46	10	376.5	80	10	2.2%	-42.70 [-99.90, 14.50]
Menees et al, 2014_HF5%_20s_LD	313.7	62	10	376.5	80	10	1.9%	-62.80 [-125.53, -0.07]
Menees et al, 2014_HF9.5%_120s_LD	353.5	67	10	376.5	80	10	1.9%	-23.00 [-87.68, 41.68]
Menees et al, 2014_HF9.5%_20s_LD	303.5	39	10	376.5	80	10	2.3%	-73.00 [-128.16, -17.84]
Prochnow et al, 2017_HF1%_20s_LD	333.71	59.7	23	321.88	57.6	23	4.0%	11.83 [-22.07, 45.73]
Prochnow et al, 2017_HF10%_20s_LD	291.48	40.7	23	321.88	57.6	23	4.6%	-30.40 [-59.22, -1.58]
Prochnow et al, 2017_HF3%_20s_LD	326.85	59.2	23	321.88	57.6	23	4.0%	4.97 [-28.79, 38.73]
Prochnow et al, 2017_HF5%_20s_LD	308.36	59.1	23	321.88	57.6	23	4.0%	-13.52 [-47.25, 20.21]
Venturini et al, 2015_HF1%_60s_F	106.47	8.4	30	143.3	12.1	30	7.1%	-36.83 [-42.10, -31.56]
Venturini et al, 2015_HF10%_60s_F	102.64	8.7	30	143.3	12.1	30	7.1%	-40.66 [-45.99, -35.33]
Venturini et al, 2015_HF3%_60s_F	105.54	10.4	30	143.3	12.1	30	7.1%	-37.76 [-43.47, -32.05]
Venturini et al, 2015_HF5%_60s_F	102.02	5.3	30	143.3	12.1	30	7.1%	-41.28 [-46.01, -36.55]
Yi, Kelly 2011_HF9%_60s_F	81.36	4.45	10	91.59	11.48	10	6.9%	-10.23 [-17.86, -2.60]
Zogheib et al, 2011_HF4.9%_180s_LD	314	62	15	417	55	15	3.3%	-103.00 [-144.94, -61.06]
Zogheib et al, 2011_HF4.9%_20s_LD	367	68	15	417	55	15	3.1%	-50.00 [-94.26, -5.74]
Zogheib et al, 2011_HF4.9%_60s_LD	363	84	15	417	55	15	2.6%	-54.00 [-104.81, -3.19]
Zogheib et al, 2011_HF4.9%_90s_LD	329	70	15	417	55	15	3.0%	-88.00 [-133.05, -42.95]
Total (95% CI)			**376**			**376**	**100.0%**	**-27.20 [-37.45, -16.96]**

Heterogeneity: Tau²=378.99; Chi²=374.67, df=20 (*P*<.001); I²=95%
Test for overall effect: Z=5.20 (*P*<.001)

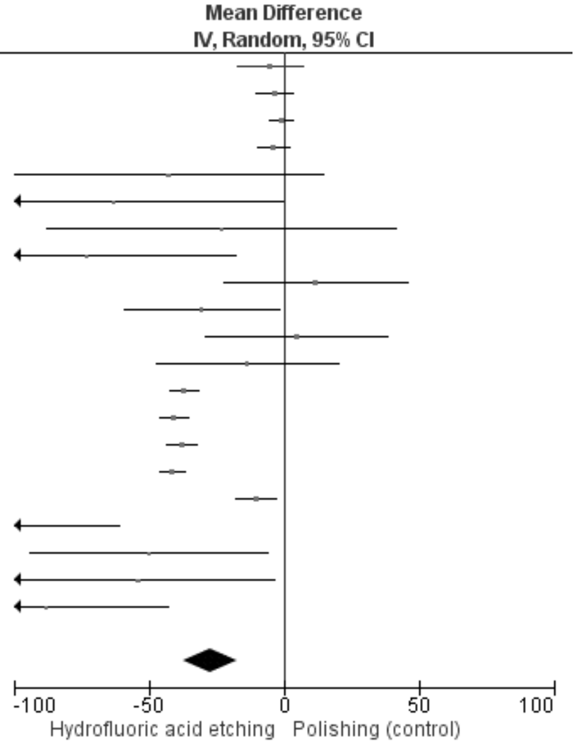


Figure 6. Subgroup analysis for flexural strength analysis of hydrofluoric acid etching versus polishing (control) for feldspathic (F) and leucite (L) glass-ceramics.

	Hydrofluoric acid etching			Polishing (control)				Mean Difference
Study or Subgroup	**Mean**	**SD**	**Total**	**Mean**	**SD**	**Total**	**Weight**	**IV, Random, 95% CI**
HF5%_30s or more								
Liu et al, 2015_HF5%_120s_F	86.8	9.6	10	90	5.5	10	14.3%	-3.20 [-10.06, 3.66]
Liu et al, 2015_HF5%_30s_F	89	4.8	10	90	5.5	10	14.6%	-1.00 [-5.52, 3.52]
Liu et al, 2015_HF5%_60s_F	86.3	7.4	10	90	5.5	10	14.4%	-3.70 [-9.41, 2.01]
Venturini et al, 2015_HF5%_60s_F	102.02	5.3	30	143.3	12.1	30	14.5%	-41.28 [-46.01, -36.55]
Subtotal (95% CI)			**60**			**60**	**57.9%**	**-12.34 [-33.07, 8.40]**
Heterogeneity: Tau²=439.62; Chi²=181.62, df=3 (*P*<.001); I²=98%								
Test for overall effect: Z=1.17 (*P*=.24)								
HF9%-10%_60s								
Fraga et al, 2015_HF10%_60s_L	161.47	16.3	24	166.48	25.81	24	13.5%	-5.01 [-17.22, 7.20]
Venturini et al, 2015_HF10%_60s_F	102.64	8.7	30	143.3	12.1	30	14.5%	-40.66 [-45.99, -35.33]
Yi, Kelly 2011_HF9%_60s_F	81.36	4.45	10	91.59	11.48	10	14.2%	-10.23 [-17.86, -2.60]
Subtotal (95% CI)			**64**			**64**	**42.1%**	**-18.99 [-43.14, 5.15]**
Heterogeneity: Tau²=435.29; Chi²=56.08, df=2 (*P*<.001); I²=96%								
Test for overall effect: Z=1.54 (*P*=.12)								
Total (95% CI)			**124**			**124**	**100.0%**	**-15.16 [-30.27, -0.04]**
Heterogeneity: Tau²=403.08; Chi²=267.02, df=6 (*P*<.001); I²=98%								
Test for overall effect: Z=1.97 (*P*=.05)								
Test for subgroup differences: Chi²=.17, df=1 (*P*=.68), I²=0%								

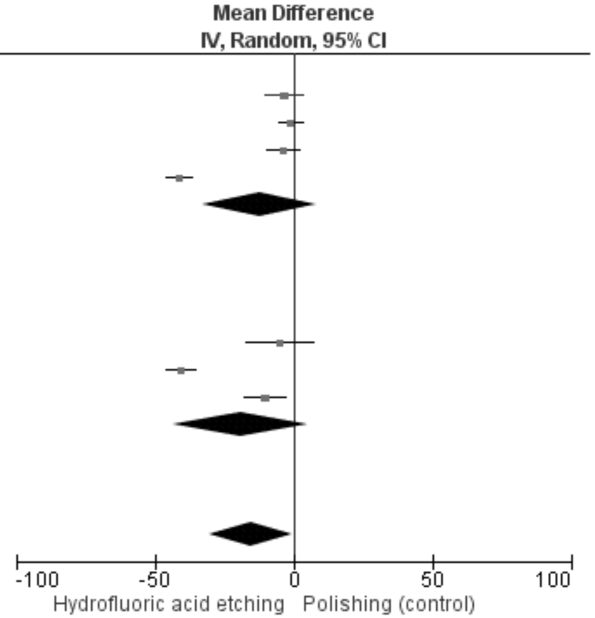


Figure 7. Subgroup analysis for flexural strength analysis of hydrofluoric acid etching versus polishing (control) for lithium disilicate (LD) glass-ceramic.

	Hydrofluoric acid etching			Polishing (control)				Mean Difference
Study or Subgroup	**Mean**	**SD**	**Total**	**Mean**	**SD**	**Total**	**Weight**	**IV, Random, 95% CI**
HF4.9%-5%_20s								
Menees et al, 2014_HF5%_20s_LD	313.7	62	10	376.5	80	10	7.5%	-62.80 [-125.53, -0.07]
Prochnow et al, 2017_HF5%_20s_LD	308.36	59.1	23	321.88	57.6	23	14.5%	-13.52 [-47.25, 20.21]
Zogheib et al, 2011_HF4.9%_20s_LD	367	68	15	417	55	15	11.4%	-50.00 [-94.26, -5.74]
Subtotal (95% CI)			**48**			**48**	**33.4%**	**-35.03 [-64.90, -5.17]**
Heterogeneity: Tau²=190.32; Chi²=2.71, df=2 (*P*=.26); I²=26%								
Test for overall effect: Z=2.30 (*P*=.02)								
HF9%-10%_20s								
Menees et al, 2014_HF9.5%_20s_LD	303.5	39	10	376.5	80	10	8.9%	-73.00 [-128.16, -17.84]
Prochnow et al, 2017_HF10%_20s_LD	291.48	40.7	23	321.88	57.6	23	16.1%	-30.40 [-59.22, -1.58]
Subtotal (95% CI)			**33**			**33**	**25.0%**	**-44.94 [-84.53, -5.35]**
Heterogeneity: Tau²=403.19; Chi²=1.80, df=1 (*P*=.18); I²=44%								
Test for overall effect: Z=2.22 (*P*=.03)								
HF4.9%-5%_60s or more								
Menees et al, 2014_HF5%_120s_LD	333.8	46	10	376.5	80	10	8.5%	-42.70 [-99.90, 14.50]
Zogheib et al, 2011_HF4.9%_180s_LD	314	62	15	417	55	15	12.0%	-103.00 [-144.94, -61.06]
Zogheib et al, 2011_HF4.9%_60s_LD	363	84	15	417	55	15	9.8%	-54.00 [-104.81, -3.19]
Zogheib et al, 2011_HF4.9%_90s_LD	329	70	15	417	55	15	11.2%	-88.00 [-133.05, -42.95]
Subtotal (95% CI)			**55**			**55**	**41.6%**	**-76.25 [-103.56, -48.94]**
Heterogeneity: Tau²=175.67; Chi²=3.87, df=3 (*P*=.28); I²=23%								
Test for overall effect: Z=5.47 (*P*<.001)								
Total (95% CI)			**136**			**136**	**100.0%**	**-54.97 [-75.96, -33.99]**
Heterogeneity: Tau²=495.27; Chi²=16.04, df=8 (*P*=.04); I²=50%								
Test for overall effect: Z=5.13 (*P*<.001)								
Test for subgroup differences: Chi²=4.29, df=2 (*P*=.12), I²=53.4%								

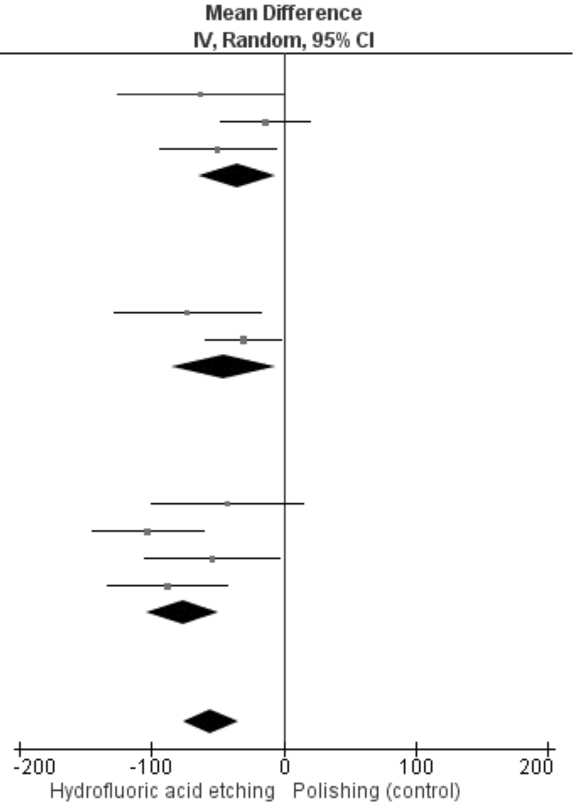


Figure 8. Forest plot for roughness (Ra) analysis of hydrofluoric acid etching versus polishing (control) for glass-ceramics. F, feldspathic; L, leucite; LD, lithium disilicate.

	Hydrofluoric acid etching			Polishing (control)				Mean Difference
Study or Subgroup	**Mean**	**SD**	**Total**	**Mean**	**SD**	**Total**	**Weight**	**IV, Random, 95% CI**
Fraga et al, 2015_HF10%_60s_L	0.61	0.06	24	0.04	0.01	24	6.3%	0.57 [0.55, 0.59]
Liu et al, 2015_HF5%_120s_F	0.52	0.09	8	0.11	0.02	8	6.2%	0.41 [0.35, 0.47]
Liu et al, 2015_HF5%_30s_F	0.25	0.04	8	0.11	0.02	8	6.3%	0.14 [0.11, 0.17]
Liu et al, 2015_HF5%_60s_F	0.5	0.07	8	0.11	0.02	8	6.3%	0.39 [0.34, 0.44]
Prochnow et al, 2017_HF1%_20s_LD	0.1291	0.46	23	0.1676	0.08	23	5.8%	-0.04 [-0.23, 0.15]
Prochnow et al, 2017_HF10%_20s_LD	0.1457	0.04	23	0.1676	0.08	23	6.3%	-0.02 [-0.06, 0.01]
Prochnow et al, 2017_HF3%_20s_LD	0.1282	0.05	23	0.1676	0.08	23	6.3%	-0.04 [-0.08, -0.00]
Prochnow et al, 2017_HF5%_20s_LD	0.1372	0.07	23	0.1676	0.08	23	6.3%	-0.03 [-0.07, 0.01]
Venturini et al, 2015_HF1%_60s_F	0.34	0.04	30	0.17	0.06	30	6.3%	0.17 [0.14, 0.20]
Venturini et al, 2015_HF10%_60s_F	1.39	0.1	30	0.17	0.06	30	6.3%	1.22 [1.18, 1.26]
Venturini et al, 2015_HF3%_60s_F	0.61	0.07	30	0.17	0.06	30	6.3%	0.44 [0.41, 0.47]
Venturini et al, 2015_HF5%_60s_F	0.82	0.07	30	0.17	0.06	30	6.3%	0.65 [0.62, 0.68]
Zogheib et al, 2011_HF4.9%_180s_LD	0.16	0.1	15	0.06	0.01	15	6.3%	0.10 [0.05, 0.15]
Zogheib et al, 2011_HF4.9%_20s_LD	0.09	0.05	15	0.06	0.01	15	6.3%	0.03 [0.00, 0.06]
Zogheib et al, 2011_HF4.9%_60s_LD	0.12	0.05	15	0.06	0.01	15	6.3%	0.06 [0.03, 0.09]
Zogheib et al, 2011_HF4.9%_90s_LD	0.14	0.06	15	0.06	0.01	15	6.3%	0.08 [0.05, 0.11]
Total (95% CI)			**320**			**320**	**100.0%**	**0.26 [0.10, 0.41]**

Heterogeneity: Tau²=.10; Chi²=4804.40, df=15 (*P*<.001); I²=100%
Test for overall effect: Z=3.28 (*P*=.001)

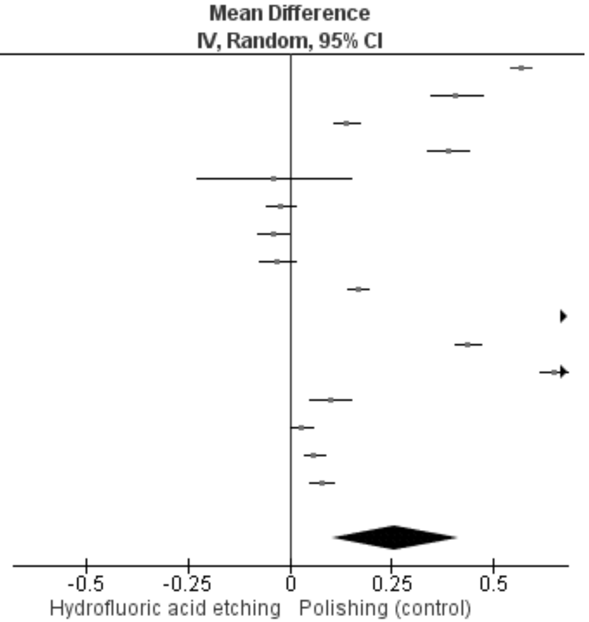


Figure 9. Subgroup analysis for roughness (Ra) analysis of hydrofluoric acid etching versus polishing (control) for feldspathic (F) and leucite (L) glass-ceramics.

Study or Subgroup	Hydrofluoric acid etching Mean	SD	Total	Polishing (control) Mean	SD	Total	Weight	Mean Difference IV, Random, 95% CI
HF5%_60s or more								
Liu et al, 2015_HF5%_120s_F	0.52	0.09	8	0.11	0.02	8	19.9%	0.41 [0.35, 0.47]
Liu et al, 2015_HF5%_60s_F	0.5	0.07	8	0.11	0.02	8	20.0%	0.39 [0.34, 0.44]
Venturini et al, 2015_HF5%_60s_F	0.82	0.07	30	0.17	0.06	30	20.1%	0.65 [0.62, 0.68]
Subtotal (95% CI)			**46**			**46**	**59.9%**	**0.48 [0.30, 0.67]**
Heterogeneity: Tau²=.03; Chi²=92.31, df=2 (*P*<.001); I²=98%								
Test for overall effect: Z=5.06 (*P*<.001)								
HF9%-10%_60s								
Venturini et al, 2015_HF10%_60s_F	1.39	0.1	30	0.17	0.06	30	20.0%	1.22 [1.18, 1.26]
Fraga et al, 2015_HF10%_60s_L	0.61	0.06	24	0.04	0.01	24	20.1%	0.57 [0.55, 0.59]
Subtotal (95% CI)			**54**			**54**	**40.1%**	**0.89 [0.26, 1.53]**
Heterogeneity: Tau²=.21; Chi²=695.47, df=1 (*P*<.001); I²=100%								
Test for overall effect: Z=2.75 (*P*=.006)								
Total (95% CI)			**100**			**100**	**100.0%**	**0.65 [0.39, 0.91]**

Heterogeneity: Tau²=.09; Chi²=913.47, df=4 (*P*<.001); I²=100%
Test for overall effect: Z=4.89 (*P*<.001)
Test for subgroup differences: Chi²=1.47, df=1 (*P*=.23), I²=31.8%

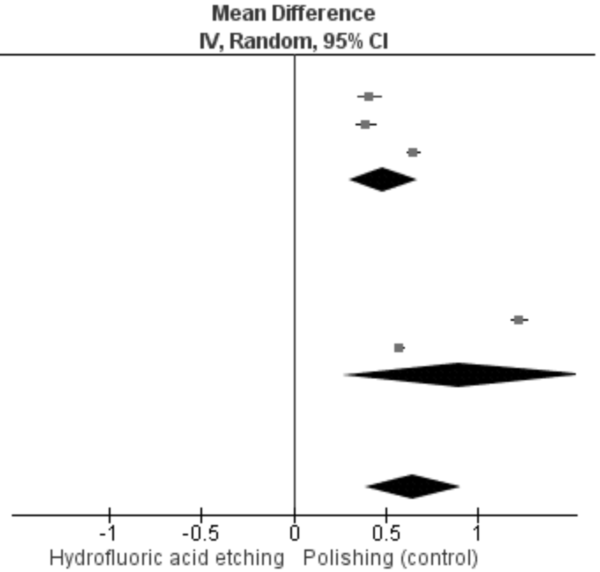
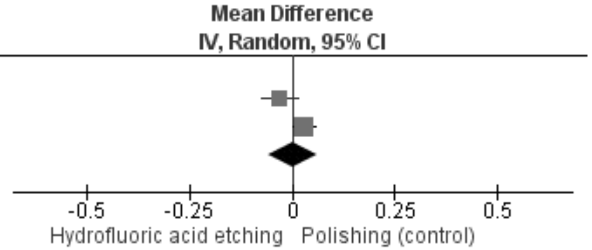


Figure 10. Subgroup analysis for roughness (Ra) analysis of hydrofluoric acid etching versus polishing (control) for lithium disilicate (LD) glass-ceramic.

Study or Subgroup	Hydrofluoric acid etching Mean	SD	Total	Polishing (control) Mean	SD	Total	Weight	Mean Difference IV, Random, 95% CI
HF4.9%-5%_20s								
Prochnow et al, 2017_HF5%_20s_LD	0.1372	0.07	23	0.1676	0.08	23	45.6%	-0.03 [-0.07, 0.01]
Zogheib et al, 2011_HF4.9%_20s_LD	0.09	0.05	15	0.06	0.01	15	54.4%	0.03 [0.00, 0.06]
Total (95% CI)			**38**			**38**	**100.0%**	**0.00 [-0.06, 0.06]**

Heterogeneity: Tau²=.00; Chi²=5.49, df=1 (*P*=.02); I²=82%
Test for overall effect: Z=.08 (*P*=.94)



4 ARTIGO 2 – EFFECT OF RESIN CEMENT COATING ON REVERTING THE IMPACT OF MACHINING ON THE FLEXURAL FATIGUE STRENGTH OF LITHIUM DISILICATE GLASS-CERAMIC.

Este artigo científico será submetido ao periódico Dental Materials, Elsevier, ISSN: 0109-5641, Fator de impacto = 4.070; Qualis A1. As normas para publicação estão descritas no Anexo B.

Effect of resin cement coating on reverting the impact of machining on the flexural fatigue strength of lithium disilicate glass-ceramic

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Abstract

Objectives. To assess the effect of resin-cement coating – with high and low viscosities – on the flexural fatigue strength of machined lithium disilicate glass-ceramic.

Methods. Disc-shaped samples (IPS e.max CAD) were prepared and divided according to surface condition (machining (M) – CEREC inLab; and polishing (P) – laboratory procedures), resin cement coating (yes or no) and cement viscosity (high (H) and low (L)). Cement-coated specimens received primer application (Monobond Etch & Prime) followed by resin cement (Variolink N Base + High or Low viscosity catalyst), which was pressed over a glass sheet and photopolymerized. Biaxial flexural fatigue strength was evaluated on a piston-on-three-ball set by step-test method (n = 15) (initial stress: 60 MPa; incremental steps: 20 MPa; 10,000 cycles/step, at 20 Hz). Weibull statistics was used for fatigue data. Contact angle, topographic and fractographic analysis were also performed.

Results. Machining produced statistically lower contact angle than polishing and a significant deleterious effect on the ceramic fatigue behavior (σ_0 M 247.2 [246.9 – 268.3]; P 337.4 [297.8 – 382.4]). Machined lithium disilicate groups followed by resin cement coating (σ_0 MH 297.9 [276.0 – 321.5]; ML 301.2 [277.1 – 327.4]) behaved similarly to the polished and coated ones (σ_0 PH 342.0 [308.9 – 378.5]; PL 357.3 [324.7 – 393.1]), irrespective of the cement viscosity. Weibull modulus of fatigue strength was higher in machined groups.

Significance. Cement coating was able to revert the impact of machining on the fatigue strength of lithium disilicate glass-ceramic. High and low viscosity cements behave similarly on the improvement of CAD-CAM lithium disilicate fatigue strength.

Key words: Computer-aided design, computer-aided manufacturing, dental ceramics, milling, cement coating, fatigue flexural strength, strengthening

1. Introduction

Lithium disilicate glass-ceramics are widely used in dentistry due to aesthetics and bioinertness properties [1]. Their mechanical behavior is improved by the crystallization heat-treatment, which creates an interlocked microstructure containing ~70 vol% of crystals [2]. Lithium disilicate is a versatile ceramic, being indicated for veneers, anterior and posterior monolithic crowns, inlays, onlays, hybrid abutments and three-unit bridges (up to second premolar) [2,3].

Computer-aided design and computer-aided manufacturing (CAD-CAM) systems use standardized ceramic blocks, which are homogenous with minimal flaws, resulting in ceramic restorations with superior biomechanical properties [4]. However, ceramics are brittle materials, supporting little or no plastic deformation before fracturing [5], and their strength is substantially affected by the presence of defects, notably critical when located in areas of tensile stress concentration, such as the internal surface of ceramic crowns [1,6–8].

The machining process can induce defects in these critical areas, including radial and lateral cracks, chipping, sub-surface defects and residual stresses [9–11] reducing the strength of CAD-CAM ceramics [12–15]. Fractographic studies also showed machining defects as the fracture origin of clinically failed all-ceramic restorations [16].

Resin coating has been related to an all-ceramic strengthening mechanism [17–20]. This effect has been associated with crack healing by resin infiltration [21], crack-closure stresses promoted by the polymerization shrinkage [22–24] and Poisson constraint effects [25]. Addison et al (2007) [26] proposed that strengthening depends on the establishment of a resin-ceramic 'hybrid layer', insensitive to individual defect size [27], but sensitive to macroscopic surface texture and elastic modulus of the resin cement [26,28].

Distinct physical properties of the resin luting agents might impact the strengthening of ceramics on cementation [19]. Modifications in resin cement viscosity have been made by manufacturers to allow a wider range of clinical applications. Low viscosity versions formed a minor pellicle thickness following the restoration placement, that could impact the polymerization shrinkage, gaps formation and premature marginal leakage [29] and maybe could penetrate the restoration surface irregularities more easily. To the author's knowledge, the comparative performance of resin-based luting agents with distinct viscosities on strengthening of glass-ceramic materials has received little attention.

It is important to consider that, in the oral environment, all-ceramic restorations are subject to many challenges, such as cyclic loads, moisture, pH and temperature variations. Consequently, ceramics tend to fail due to fatigue [30], which means they are subjected to stresses below the critical value and are susceptible to slow crack growth (SCG), resulting from a chemical reaction between water and ceramic crack tips, that breaks oxide bonds. Thus, the crack grows until it reaches a critical size for fracture, leading to catastrophic failure [31].

Studies have shown the negative effect of machining on the monotonic or fatigue flexural strength of glass-ceramics [12–14], however, there is no information about the role of an adhesive cement coating on the partial or total reversion of the negative effect of machining.

Based on the above, this study has the aim of evaluating the effect of resin cement coating – with high and low viscosities – on the fatigue behavior of a lithium disilicate ceramic machined by a CAD-CAM system. The first hypothesis is that the resin cement coating increases the fatigue strength of ceramic in comparison with the uncovered one. The second hypothesis is that there is no difference between the fatigue flexural strength of polished and machined ceramics, when they are coated with cement. The third hypothesis is that there is no difference in the fatigue behavior of lithium disilicate with low and high cement viscosity covering.

2. Materials e methods

The materials used in this study, their compositions, commercial names and manufacturers are described in Table 1.

A pilot study (n=5) was carried out for evaluation of the flexural fatigue strength of lithium disilicate discs, according surface condition and cement viscosity (Polishing/high, polishing/low, machining/high and machining/low). Machining was simulated by manual abrasion with 60-grit silica carbide papers (Norton Saint-Gobain, Guarulhos, Sao Paulo, Brazil) reproducing the average roughness of machined specimens. Means (standard deviation) were 308.30 (56.76) MPa for polishing/high, 299.48 (47.24) MPa for polishing/low, 251.60 (14.50) MPa for machining/high and 261.23 (11.83) MPa for machining/low. These results were used for estimation of sample size (n=15), using an open-source calculator (openepi.com) (95% confidence interval and power of 80%).

2.1. Specimen preparation

Disc-shaped specimens (Ø= 13.5 mm; thickness= 1.3 mm; IPS e.max CAD, Ivoclar Vivadent) were prepared in two different ways: a Computer Aided Design-Computer Aided Manufacturing (CAD–CAM) process, that reproduces machined restoration surfaces, and a laboratory process, that generates specimens with polished surfaces [14].

The machined specimens were produced by machining in a CEREC inLab MC XL milling unit (Sirona Dental Systems Gmbh, Germany). The discs were designed using a CAD software, that uses the Non-uniform rational basis spline (NURBS) language (Rhinoceros 6.0SR8, McNell North America, Seattle, WA). A 0.1 mm chamfer was planned to avoid sharp edges during machining. The three-dimensional models were exported in Standard Tessellation Language (STL) format to CAM software (inLab 18.1, Dentsply Sirona, Charlotte, NC, EUA) and the type of restoration was defined as "crown" mode. One of the faces of the disc was indicated as the occlusal region of the crown to allow the software establish a trajectory and machining strategy. Three sets of diamond burs, one stepped and one cylindrical, were used to machining the specimens and were replaced after machining with 25th disc or when a bur failed [12]. Immediately after machining, the discs measured approximately 13.5 mm in diameter and 1.5 mm in thickness.

Machining left a small residual sprue attachment on the lateral circumference of the discs, continuous with the upper surface. The sprue position was marked at the opposite side, to guide on which surface the adjustments were conducted and allow orientation of the specimens during the roughness measurements. Subsequently the sprue was manually removed using a diamond bur at low speed.

To produce the polished discs specimens, the pre-fabricated ceramic blocks were shaped into cylinders (13.5 mm diameter) by abrading with a coarse diamond grinding disc (Apex CGD, PSA, Green 240 µm, 8in, Buehler, Lake Bluff, IL, USA) followed by 240, 400 and 600-grit silica carbide papers (Norton Saint-Gobain, Guarulhos, Sao Paulo, Brazil) in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, IL, EUA) under water. Next, cylinders were sectioned with a diamond saw using a precision cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA), with a blade speed of 300 rpm and water lubrication, producing slices with 1.5 ± 0.10 mm thickness. Then, the bottom surface of the discs was polished with 240, 400, 600 and 1200 granulation silicon carbide sandpaper in a polishing machine under water [12].

The final thickness of all specimens, machined and polished (1.3 ± 0.01 mm thickness) was adjusted on the upper surface of the discs, using 240, 400, 600, and 1200 grit silicon carbide paper (Norton Saint-Gobain, Guarulhos, São Paulo, Brazil) in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, IL, EUA) with constant water irrigation [12]. Polishing/adjustment procedures were performed by a single trained operator. A digital micrometer (Mitutoyo absolute 500-196-20 Digital Caliper; Takatsu- Ku, Kawasaki, Kanagawa, Japan) was used for thickness control.

Succeeding these procedures, the lithium disilicate specimens were crystallized in a Vita Vacumat 6000 MP oven (Vita Zahnfabrik, Germany), following the manufacturer's protocol (pre-drying temperature 403 °C for 6 min; heating rate 90 °C/min (under vacuum from 550–820 ºC); firing temperature 820 °C for 0.1 min; heating rate 30 °C/min (under vacuum from 820–840 ºC); firing temperature 840 °C for 7 min; long-term cooling temperature 700 °C) [32]. After crystallization, the final dimensions of the discs were measured with a digital micrometer (Mitutoyo absolute 500-196-20 Digital Caliper; Takatsu- Ku, Kawasaki, Kanagawa, Japan) and were 13.5 mm in diameter and 1.3 ± 0.01 mm in thickness.

2.1.1 Surface roughness, contact angle and topographic analysis

A contact profilometer (SJ-410, Mitutoyo, Japan) was used to measure the roughness of the bottom surface of each specimen prior to cementation procedures, in the conditions machined and polished. After randomization (section 2.2), three measurements were carried out, transversal to the machining path, which was oriented by the aforementioned sprue position mark, considering Ra (average surface roughness) and Rz (arithmetic mean peak-to-valley height) parameters (cut-off of 5; λC of 0.8 mm; λS of 2.5 μm), according to the ISO 4287:1997 [33]. After Shapiro-Wilk and Levene tests, One Way ANOVA followed by Tukey's post hoc tests were performed for evaluation of roughness (Ra and Rz parameters), with a significance level of 0.05. Machining groups - M, MH, ML - showed statistically higher roughness values (Ra 1.44 (0.16), 1.50 (0.16) and 1.47 (0.17); Rz 8.45 (0.96), 8.92 (0.77) and 8.75 (0.77)) than polishing groups - P, PH, PL - (Ra 0.03 (0.01), 0.03 (0.01) and 0.03 (0.00); Rz 0.17 (0.04), 0.17 (0.04) and 0.17 (0.02)). Roughness values were similar, when comparing groups with the same surface characteristic ($P>0.05$).

To determine the contact angles, extra-samples of machining and polishing groups (n=5) were conditioned with a self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent) (scrubbed for 20 s, left to react for 40 s, air-water sprayed for 30 s and air dried for 10 s). Next, the contact angle was assessed via sessile drop technique at room temperature (±24°C) using a goniometer (Drop Shape analysis, model DSA 30S, Kruss GmbH, Hamburg, Germany) connected to a dedicated software (DSA3, V1 .0.3-08, Kruss). Utilizing a needle, one drop (11 μl) of distilled water was deposited at the center of the lithium disilicate surface and the contact angle was measured after 5 seconds.

Representative specimens of machined and polished groups were ultrasonically cleaned with 78% isopropyl alcohol bath for 5 min (1440 D, 50/60 Hz, Odontobras), alloy sputtered and examined by scanning electron microscope (SEM - Vega3, Tescan, Brno, Czech Republic) at 200× and 1,500× of magnification, under a high vacuum with 20.00 kV at a working distance of approximately 13.5 mm for topographic analysis.

2.2 Experimental groups

The discs of each surface processing (machined and polished) were randomly divided into three groups using the software Random Allocator (www.random.org), according to cement coating (yes; no-control) and cement viscosity (high; low). Following the allocation, six experimental groups were formed: (1) machining, no coating (M); (2) machining followed by high viscosity resin cement coating (MH); (3) machining followed by low viscosity resin cement coating (ML); (4) polishing, no coating (P); (5) polishing followed by high viscosity resin cement coating (PH) and (6) polishing followed by low viscosity resin cement coating (PL). Polished groups served as controls. The experimental design is shown in Table 2.

2.3 Resin-coating of ceramic discs

Specimens of the groups that should receive resin cement coating were ultrasonically cleaned (1440 D, 50/60 Hz, Odontobras, Ind. And Com. Equip. Odonto. LTDA, Ribeirão Preto, São Paulo, Brazil) with 78% isopropyl alcohol for 5 min. After drying, a ceramic self-etching primer (Monobond Etch & Prime, Ivoclar Vivadent) was actively rubbed on the bottom surface of the ceramic with a microbrush for 20 s, left to react for 40 s, removed with air-water spray for 30 s and air dried for 10 s, following the manufacturer recommendation [34]. Pastes of the resin-based luting agent Variolink N (base + catalyst High Viscosity or base + catalyst Low Viscosity) (Ivoclar Vivadent; Schaan, Liechtenstein) were dispensed with a standardized length of 10 mm each paste on the spatulation block (in a 1:1 ratio of base and catalyst), manipulated following the manufacturer's instructions [35] by the same operator, and applied onto the treated surface of the ceramic discs. The cement-coated specimens were immediately placed on top of a glass slide covered with a thin acetate sheet, centrally oriented on a loading platform [18]. Then, the upper ceramic surface was loaded with a 2.5 N load for 60 s. Excess luting agent was removed using a microbrush, and the assembly was light-cured (Radii-cal LED curing light, SDI, Bayswater, Australia) for five exposures of 20 s each position (0º, 90º, 180º, 270º, and on top). The assembly thickness was measured using a digital micrometer (Mitutoyo absolute 500-196-20 Digital Caliper; Takatsu- Ku, Kawasaki, Kanagawa, Japan) and the film thickness was calculated as the difference from the coated specimen and the ceramic disc before cementation [36]. The mean of the cement thickness was 0.06 (± 0.02) mm for low viscosity cement, and 0.10 (± 0.01) mm for high viscosity cement. All specimens were dry stored at 37 ºC for 24 h to 72 h in a dark container prior to flexural fatigue testing [18].

2.4 Flexural fatigue test

The specimens (n=15) were submitted to a biaxial flexural fatigue strength test, using a piston-on-three ball assembly, according to the ISO 6872:2015 [37] evaluated via step-stress methodology [38], in an electrodynamic machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, United States) under water. The bottom surface of the disc (machined, polished or cement coated) was positioned facing down (tensile tension side) on the top of the three steel spheres (2.5 mm in diameter, 120º apart, and forming a circle of 10 mm diameter) covered with a piece of cellophane paper (2.5 µm) for better contact pressure [37]. An adhesive tape (Durex, 3M, Sumaré, São Paulo, Brazil) was glued on the top surface of the specimen to improve the piston contact and tension distribution and avoid the spreading of fragments when fracture occurs [5,39]. The cyclic loading was carried out by a cylindrical tungsten piston with a flat tip of 1.6 mm diameter, according ISO:6872/2015 [37].

The step-stress protocol was established as a cyclic load applied at a frequency of 20 Hz [40] starting with an initial tension of 60 MPa on the ceramic tensile surface to 5,000 cycles, followed by increments of 20 MPa for each 10,000 cycles until the failure of the specimen. The stress and the number of cycles corresponding to the specimen fracture were recorded.

To determine the load (N) correspondent to initial step (60 MPa) and subsequent increments (20 MPa), all specimens were previously measured using digital caliper (Mitutoyo absolute 500-196-20 Digital Caliper), before and after resin cement coating, and these values were applied to Equations (1), (2), (3) and (4) [41].

$$R = \sqrt{1{,}6B^2 + d^2} - 0{,}675d \quad (1)$$

$$M = \frac{W}{4\pi}\left[(1 + v)log\frac{A}{R} + 1\right] \quad (2)$$

$$K_{2p} = \frac{E_b t_b{}^3(1 - v_a{}^2)}{E_b t_b{}^3(1 - v_a{}^2)} + \frac{3(1 - v_a{}^2)\left(1 + \frac{t_b}{t_a}\right)^2\left(1 + \frac{E_a t_a}{E_b t_b}\right)}{\left(1 + \frac{E_a t_a}{E_b t_b}\right)^2 - \left(v_a + \frac{v_b E_a t_a}{E_b t_b}\right)^2} \quad (3)$$

$$\sigma = \frac{6M}{t_a{}^2 K_{2p}}\left[\frac{E_b t_{b(1 - v_a{}^2)}}{E_b t_{b(1 - v_a{}^2)}} + \frac{t_a(1 - v_a{}^2)\left(1 + \frac{t_b}{t_a}\right)\left(1 + \frac{E_a t_a}{E_b t_b}\right)}{t_b\left(1 + \frac{E_a t_a}{E_b t_b}\right)^2 - \left(v_a \frac{v_b E_a t_a}{E_b t_b}\right)^2}\right] \quad (4)$$

where R is the radius of the disc-shaped specimen (6.75 mm), B is the radius of the piston (0.8 mm), d is the sample total thickness, M is the biaxial bending moment per unit length, W is the load (N), A is the radius of the three-balls support (5 mm), t_a is the cement thickness, t_b is the ceramic thickness, E_a is the elastic modulus of the cement (8.3 GPa) [42], E_b is the elastic modulus of lithium disilicate (102.5 GPa) [43], and ν_a and ν_b the Poisson's ratios of the ceramic (0.21) [43] and resin-based luting agents (0.27) [44].

2.5. Fractographic and bonded interfaces analysis

Representative fracture fragments generated from fatigue test were selected based on their flatter face, next to the center of the specimen, and analyzed under a stereomicroscope (Stereo Discovery V20, Carl Zeiss, Gottingen, Germany). The regions to be examined in the scanning electron microscope were pointed on suspected markings to the fracture beginning (hackles, wallner lines and compression curl). The specimens were ultrasonically cleaned with 78% isopropyl alcohol (5 min) (1440 D, 50/60 Hz, Odontobras), gold-sputtered and analyzed analyzed in the scanning electron microscope (SEM - Vega3, Tescan, Brno, Czech Republic) under a high vacuum with 20.00 kV at a working distance of approximately 13.5 mm at 200 and 1500× magnification to detect the crack origin characteristics.

Additional ceramic-resin cement-ceramic sets were prepared to observe the morphology at the bonded interfaces [36]. For that purpose, two ceramic discs of the same surface characteristic were adhesively cemented, according to item 2.3. The three-layer sets were transversely sectioned in a cutting machine (Isomet 1000, Buehler) using a diamond saw under water cooling. Then, the cross-sectional was mirror-polished (EcoMet/AutoMet 250, Buehler) using 600, 1200 and 2000 grit silica carbide papers (Norton Saint-Gobain, Guarulhos, Sao Paulo, Brazil), cleaned in an ultrasonic bath with 78% isopropyl alcohol (5 min), gold-sputtered and analyzed in the scanning electron microscope (SEM - Vega3, Tescan, Brno, Czech Republic) at 10000× magnification.

2.6. Data analysis

Flexural fatigue strength and cycles for fatigue failure data were submitted to Weibull analysis using the Super SMITH Weibull 4.0k-32 software (Wes Fulton, Torrance, USA) under the maximum-likelihood method to obtain the Weibull modulus (m — a measure of the distribution of strengths or cycles, expressing the reliability of each condition) and characteristic fatigue strength (σ0)/characteristic cycles for fatigue failure (expressing the strength value or cycles at a failure probability of approximately 63.3%). Statistical differences were considered when groups had non-overlapping confidence intervals. Additionally, the survival probability was tabulated for each step of the test.

Contact angle data assumed a normal and homoscedastic distribution (Shapiro-Wilk/Levene tests) and a t-test was conducted for evaluate the equality of means. A statistical software program (IBM SPSS Software; IBM, Armonk, NY, USA) was used for data analysis.

3. Results

M group had the worst fatigue performance (characteristic flexural fatigue strength, characteristic cycle for fatigue failure and survival probabilities) of the experimental groups (Tables 3 and 4). The step-stress profiles of the machining groups showed that the ML and MH groups failed under higher stress ranges than the M group, as indicated in Fig. 2a. Regarding the polishing condition, no differences were found between characteristic flexural fatigue strength, characteristic cycles for fatigue failure, survival probability or step-stress profile among the groups P, PL and PH (Tables 3 and 4; Fig. 2b).

Considering cement coating, experimental groups showed a similar fatigue behavior for high and low viscosity cement (MH ~ ML; PH ~ PL). Also, comparing the two ceramic surface conditions for the same resin-cement viscosity, machining and polishing had the same flexural fatigue behavior (MH ~ PH; ML ~ PL) (Tables 3 and 4).

The machining group (M) presented higher Weibull modulus for fatigue flexural strength compared with the polishing group (P), and coated specimens were statistically similar. There was no statistical difference in Weibull modulus for cycles for fatigue failure, with respect to the surface characteristics or cement viscosities (Table 3).

After surface treatment (with self-etching primer), polishing showed higher contact angles measures than machining (Fig. 1). Machining resulted in a rougher surface (Fig. 3).

Representative SEM micrographs of the fractured surfaces for each experimental group are presented in Fig. 4. It can be observed that critical flaws initiated from the face submitted to tensile stress, i.e., the surface facing the support spheres, and propagated throughout the material leading to catastrophic failure. Uncoated specimens failed from surface flaws, whereas the origin of failure in specimens coated by resin started from the cement layer and propagated through the ceramic. Ceramics coated with high or low resin-cement viscosities had no major differences as regards failure modes or critical flaw origin. High viscosity resin cement generated a layer approximately 40 μm thicker than the low viscosity resin cement.

SEM images of the ceramic-resin bonded interfaces (Fig. 5) showed that irrespective of surface condition or cement viscosity used, the resin-based luting agents were able to maintain close intimacy to the ceramic surface. Some voids were observed in the resin cement layers, probably caused by the cutting/polishing procedures required for preparation the samples for SEM analysis.

3. Discussion

This in vitro study aimed to evaluate if the resin-cement coating could revert the damage caused by machining to the strength of lithium disilicate glass-ceramic. As brittle materials, the fracture strength of ceramics is sensitive to the presence of defects, especially when located in tensile stresses concentration areas, such as the cementation surface of a restoration [8,44]. Contact between the grinding tool and the ceramic during CAD-CAM machining generates chipping defects and subsurface damage, which are associated with plastic deformation, fracture, and heat generation inside the material [9,11]. These events develop residual stresses, creating a residual compressive zone. The balance between crack population and their interaction with the compressive residual stress generated around the crack groove determine the material strengths [11]. As a result of the machining process, impairment to the mechanical behavior of glass-ceramics has been reported [10,12–15]. The results of the present study are in agreement with these findings, since it was observed that CAD-CAM machining (M group) introduced defects at ceramic surface (Fig. 3), increasing surface roughness (reported at session 2.1.1) and significantly reducing the characteristic flexural fatigue strength, number of cycles for fatigue failure and survival probabilities when compared to the polishing condition (P group).

The strengthening effect of resin-cement adhesion to the ceramic surface has been extensively investigated [17–20,28,46–49]. In the present study, this effect was observed when the machining groups received a cement coating. However, no difference in fatigue behavior were found between coated and uncoated polishing groups (Table 3; Fig. 2). Thus, the first hypothesis, that the resin cement coating increases the fatigue strength of ceramic in comparison with the non-coated specimens, was partially accepted.

These findings could be explained by the virtually defect free surface of polishing groups. Although polishing makes this ceramic more resistant, adhesion to the polished ceramic is not as effective as to the machined one. Since the reinforcement is dependent on the ceramic surface texture [46], polishing surfaces only receive a little modification due to self-etching approach, maintaining a similar roughness and etching depth to the original material [50]. However, machining reshapes the surface and introduces surface flaws, producing a rougher surface that may be accompanied by a strength limitation [51]. Nevertheless, at the same time that machining decreases ceramic strength, the adhesion could be improved by cement interlocking at the irregularities, which could promote a compensation for this strength limitation. Contact angles values depicted that machining present higher surface energy and wettability than polishing (Fig. 1).

Considering the same cement viscosity, polished and machined groups behaved statistically similar with respect to characteristic fatigue strength and characteristic cycle to failure (MH ~ PH; ML ~ PL) (Table 3). Thus, the second hypothesis was accepted. These findings confirm that the adhesion of the resin cement to machined ceramic surface was able to revert the negative effect caused by machining on its fatigue strength. This strengthening effect is an interaction between the cement and the defects present on inner ceramic surfaces, determined by the ability of the resin-cement to wet the ceramic surface [52] which is a function of the ceramic surface microstructure and roughness, the viscosity and composition of the resin-cement [18,52].

According to Fleming, Hooi and Addison (2012) [28], in an attempt to maximize the magnitude of resin-strengthening, the choice of resin-based material is a crucial factor. The effect of very low viscosity unfilled resins on the mechanical behavior of vitreous ceramics was investigated [46,53]. The authors found a significant strengthening effect following the application of unfilled resin after acid etching or alumina abrading, addressing these results to the ceramic-cement "hybrid layer". This theory is explained by a combination of Poisson constraint effects [25] and an interaction of the resin with the entire surface defect population [26]. In the course of the flexure loading, the resin within a crack deforms inside perpendicular to the crack face generating a Poisson contraction parallel to the crack surface. The compensating contraction is restricted near the crack tip, increasing the stiffness of the resin, which therefore behaves more closely to the bulk ceramic [25,26]. However, under loading conditions, the system becomes sensitive to the characteristics of the hybrid layer, depending on the complex interactions of various elements, as the elastic modulus of resin cement [20,26,28]. In this way, Barbon et al (2019) [54] investigated the influence of experimental resin-based luting agents with different inorganic filler contents on the strength of a feldspathic ceramic. The authors found that the greater is the inorganic content, the greater is the elastic modulus, viscosity, film thickness and the strengthened effect of the ceramic.

The resin cement used in this study, Variolink N, was developed based on Variolink II and the manufacturer describes its material data documentation coinciding with its predecessor, without distinguishing the physical properties between different viscosities [42]. Thus, the use of this brand of resin cement allowed analyzing the behavior of its rheological properties on the ceramic fatigue flexural strength, although high and low viscosities have a small variation of filler content (Table 1) and small differences in elastic modulus are expected [28].

In this study the two viscosities behaved similarly (MH ~ ML and PH ~ PL) (table 3) regarding the characteristic flexural fatigue strength and characteristic cycle for fatigue failure of lithium disilicate. Therefore, the third hypothesis was accepted. Since the magnitude of ceramic strengthening after resin coating has been shown to be a function of the cement capability to interpenetrate surface defects creating a "hybrid layer" [26], determined by the ceramic surface texture, mechanical properties of the resin-based material and the final resin-coating thickness [28,46], it is expected that the lower viscosity resin cement to be more capable of penetrate and fill the ceramic surface irregularities, as well as producing a thinner layer than the cement of high viscosity [28]. In fact, the film thickness generated by the low-viscosity cement was approximately 40 µm thinner than that generated by the high-viscosity cement (Fig. 4). However, despite these differences, they did not reflect in differences on the flexural fatigue strength of lithium disilicate specimens, nor in the intimacy of the cement-ceramic interface (Fig. 5). It's important to remark that the cement thickness was considered in the mathematical formulas, when calculating the stresses applied to the ceramic in a bilayer system.

Coelho et al (2019) [36] evaluated the influence of preheating different composite resins on their viscosity and strengthening yielded to a feldspar ceramic. They found a similar strengthened effect of the ceramic between the preheatings composites evaluated and the resin cement used as control, regardless of elastic modulus. However, the preheating composite thickness and viscosity was higher than resin-cement, and material dependent.

It is important to state that the interfacial relationship between resin-cement and the defects present on the ceramic surfaces is imperative for the restoration performance [18], since this region exhibits the highest concentration of tensile stresses [55] and from which some fractures have been fractographically demonstrated to propagate [8]. In the current study (Fig. 4), fractographic images indicated that the cracks propagate from the surface exposed to the tensile stress, from flaws presented in this region (uncoated specimens) or from cement trough the ceramic (coated specimens), since tensile stresses are produced by flexure and there is no support substrate like in a restoration, which could change the stress distribution behavior.

Previous studies indicated a strength degradation (difference between monotonic and fatigue strength) of approximately 44,28% ~ 53.4% for a CAD/CAM lithium disilicate [12,56], indicating a great influence of the slow crack growth of these dental ceramics [10]. In such a way, a cyclic fatigue test under water was conducted in this study. The step-stress approach consists of a time-varying tension test, in which successively higher stress levels in a predetermined number of cycles are applied to the samples [57]. Considering that the strength of brittle materials is properly described by Weibull probability distribution function [58] the step-stress data was submitted to a Weibull analysis. A previous study highlighted that the analytical solution influenced the reported failure stress and emphasized that caution is required when reported stress data among similar testing methodologies subjected to a distinct analysis were compared [59]. For that reason, the means and standard deviations of the fatigue flexural strength data was also described in the Table 3.

Weibull modulus (*m*) describes the reliability of a distribution. In the present study, the related *m* for flexural strength of the machining group (M) was statistically higher than polishing group (P). This finding suggests that the standardized machining executed by an automated system was more predictable than the manually polishing process.

This in vitro study evaluated the isolated effect of high and low viscosity resin-cement coating on the machined and polished surfaces of lithium disilicate glass-ceramic. Since it does not reproduce entirely the clinical condition, these results should be considered with caution. Further studies on these questions should be conducted evaluating the fatigue behavior of specimens cemented to a substrate, for better characterization of stress distribution state of a restoration in the clinical environment. Additionally, other properties of resin-based cements should be investigated for strengthening of lithium disilicate and other glass-ceramics.

4. Conclusion

1. Machining damages the surface of lithium disilicate ceramic, decreasing its flexural fatigue behavior. However, when a resin cement layer is bonded to their surface, this negative effect is reverted.
2. High and low viscosity resin cements behave similarly with regard to strengthening of lithium disilicate CAD-CAM ceramics.

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TABLES

Table 1. Materials used in the study: commercial name, manufacturer, batch number and composition based on the manufacturer's information.

Material	Commercial name, manufacturer (batch number)	Composition
Lithium disilicate	IPS e.Max CAD, Ivoclar Vivadent (Y42922)	SiO_2 57-80 wt%, Li_2O 11-19 wt%, K_2O 0-13 wt%, P_2O_5 0-11 wt%, ZrO_2 0-8 wt%, ZnO 0-8 wt%, other and coloring oxide 0-12 wt%
Self-etching ceramic primer	Monobond Etch & Prime, Ivoclar Vivadent (Y27772)	Ammonium polyfluoride, silane system based on trimethoxypropyl methacrylate, alcohols, water and colorant
Dual cure resin cement	Variolink N Base: White A1 (YZ1257) Catalyst High Viscosity: Yellow A3 (Y15071) Catalyst Low Viscosity: Transparent (YZ1263)	Monomer Matrix: Bis-GMA, urethane dimethacrylate and triethylene glycol dimethacrylate (Base 26.3 % wt.; Catalyst high viscosity 22 % wt.; Catalyst low viscosity 27.9 % wt.). Inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide. (Base 73.4 % wt.; Catalyst high viscosity 77.2 % wt.; Catalyst low viscosity 71.2 % wt.) Additional contents: initiators, stabilizers and pigments. Particle size is 0.04-3.0 μm, mean particle size is 0.7 μm.

Table 2. Study factors and their distribution in the experimental groups.

Surface characteristic	Cementation Protocol		
	Without Cement	Cement Coating	
		High Viscosity	Low Viscosity
Machining	M	MH	ML
Polishing	P	PH	PL

Table 3. Mean and standard deviation for the values of the flexural fatigue strength test and number of cycles for fatigue failure; Weibull analysis of the fatigue data (characteristic flexural fatigue strength – σ_0; Weibull modulus – *m*; characteristic cycle for fatigue failure; 95% confidence intervals [CI]) for the experimental groups.

Groups	Fatigue flexural strength (Mean (SD))* (MPa)	Characteristic flexural fatigue strength [95% CI] (MPa)	Weibull modulus for flexural strength [95% CI]	Cycles for fatigue failure (Mean (SD))*	Characteristic cycle for fatigue failure [95% CI]	Weibull modulus for cycles for fatigue failure [95% CI]
M	246.67 (6.67)	247.2 [246.9 – 268.3]^{A}	12.78 [8.43 – 19.36]^{A}	85263.13 (6285.58)	84173 [82372 – 102948]^{A}	4.63 [2.92 – 7.33]^{A}
P	306.67 (21.08)	337.4 [297.8 – 382.4]^{BC}	4.29 [2.89 – 6.37]^{B}	122052.33 (10735.57)	122427 [116271 – 159907]^{B}	3.36 [2.26 – 5.01]^{A}
MH	278.67 (12.26)	297.9 [276.0 – 321.5]^{B}	7.00 [4.72 – 10.37]^{AB}	109344.20 (6112.34)	118531 [107321 – 130913]^{B}	5.38 [3.64 – 7.94]^{A}
PH	314.67 (17.62)	342.0 [308.9 – 378.5]^{BC}	5.28 [3.55 – 7.86]^{B}	127837.07 (8950.11)	140906 [124027 – 160082]^{B}	4.21 [2.83 – 6.25]^{A}
ML	281.33 (12.26)	301.2 [277.1 – 327.4]^{BC}	6.44 [4.42 – 9.38]^{AB}	108339.60 (5789.89)	117388 [105999 – 130000]^{B}	5.26 [3.61 – 7.67]^{A}
PL	330.67 (17.11)	357.3 [324.7 – 393.1]^{C}	5.61 [3.82 – 8.22] ^{B}	131764.73 (8859.11)	144658 [128032 – 163444]^{B}	4.38 [2.97 – 6.49]^{A}

Different letters in each column indicate statistically significant differences for fatigue data (95% CI fail in overlapping for σ_0 and *m*).
*Descriptive data without statistical analysis

Table 4. Survival probabilities per step with respective standard errors.

Group	Fatigue flexural strength/Cycles for fatigue failure															
	160 MPa/ 10 x 10^3	**180 MPa/ 10 x 10^3**	**200 MPa/ 10 x 10^3**	**220 MPa/ 10 x 10^3**	**240 MPa/ 10 x 10^3**	**260 MPa/ 10 x 10^3**	**280 MPa/ 10 x 10^3**	**300 MPa/ 10 x 10^3**	**320 MPa/ 10 x 10^3**	**340 MPa/ 10 x 10^3**	**360 MPa/ 10 x 10^3**	**380 MPa/ 10 x 10^3**	**400 MPa/ 10 x 10^3**	**420 MPa/ 10 x 10^3**	**440 MPa/ 10 x 10^3**	**460 MPa/ 10 x 10^3**
M	1	1	0.87 (0.09)	0.73 (0.11)	0.60 (0.13)	0.13 (0.09)	0.0	-	-	-	-	-	-	-	-	-
P	1	0.93 (0.06)	0.93 (0.06)	0.87 (0.09)	0.67 (0.12)	0.60 (0.13)	0.53 (0.13)	0.33 (0.12)	0.33 (0.12)	0.33 (0.12)	0.33 (0.12)	0.27 (0.11)	0.13 (0.09)	0.07 (0.06)	0.0	-
MH	1	0.93 (0.07)	0.93 (0.07)	0.87 (0.09)	0.73 (0.11)	0.60 (0.13)	0.40 (0.13)	0.27 (0.14)	0.13 (0.09)	0.07 (0.06)	0.0	-	-	-	-	-
PH	1	1	1	1	0.73 (0.11)	0.67 (0.12)	0.53 (0.13)	0.40 (0.13)	0.40 (0.13)	0.40 (0.13)	0.27 (0.11)	0.27 (0.11)	0.07 (0.06)	0.0	-	-
ML	1	1	1	0.80 (0.10)	0.80 (0.10)	0.47 (0.13)	0.40 (0.13)	0.33 (0.12)	0.13 (0.09)	0.07 (0.06)	0.07 (0.06)	0.0	-	-	-	-
PL	1	1	1	0.93 (0.06)	0.87 (0.09)	0.87 (0.09)	0.67 (0.12)	0.67 (0.12)	0.67 (0.12)	0.27 (0.11)	0.27 (0.11)	0.20 (0.10)	0.13 (0.09)	0.07 (0.06)	0.07 (0.06)	0.0

* The symbol '-' indicates absence of specimens being tested on the respective step.

FIGURES

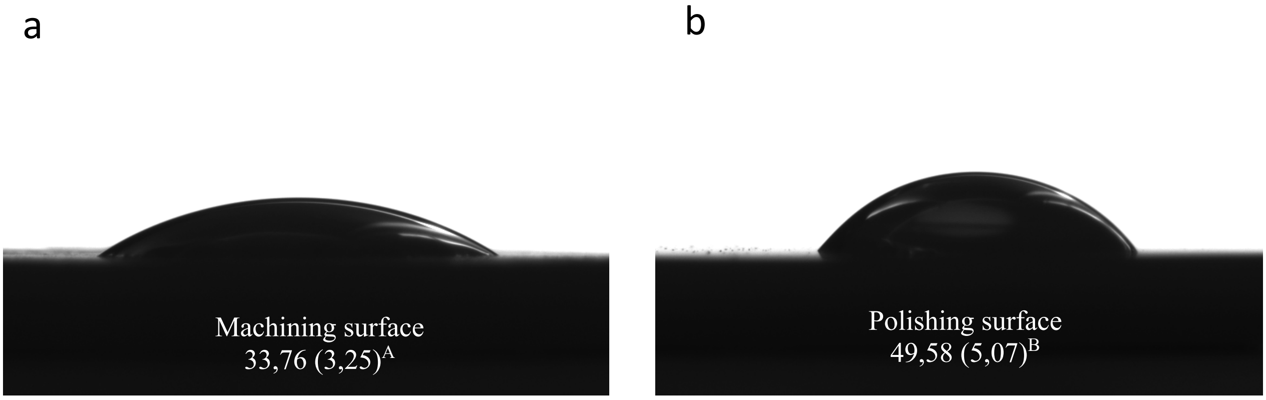
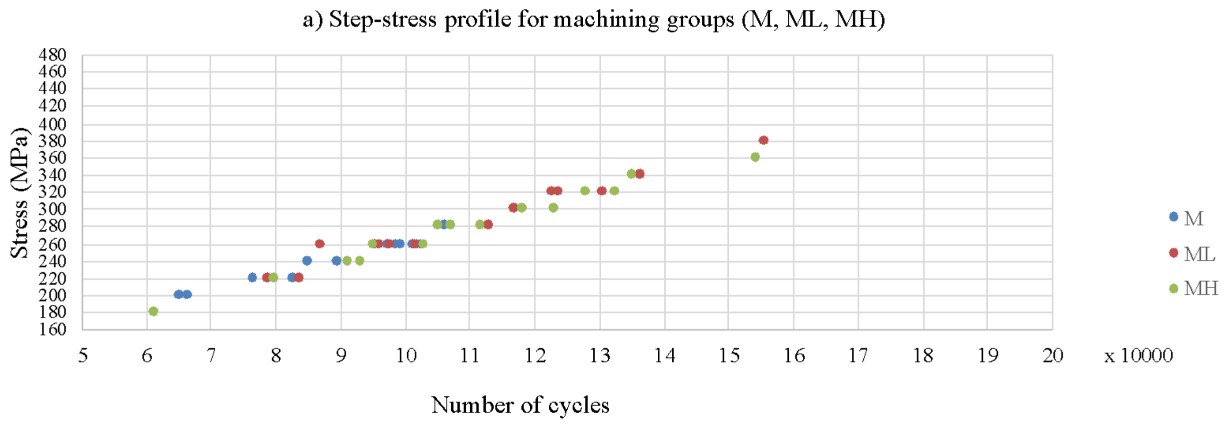


Figure 1. Representative images and means ± SD (in degrees) of contact angle measurements of machining (a) and polishing (b) surfaces after treatment with self-etching ceramic primer. Different superscript letters indicate statistically significant differences (t-test; $p < 0.05$).



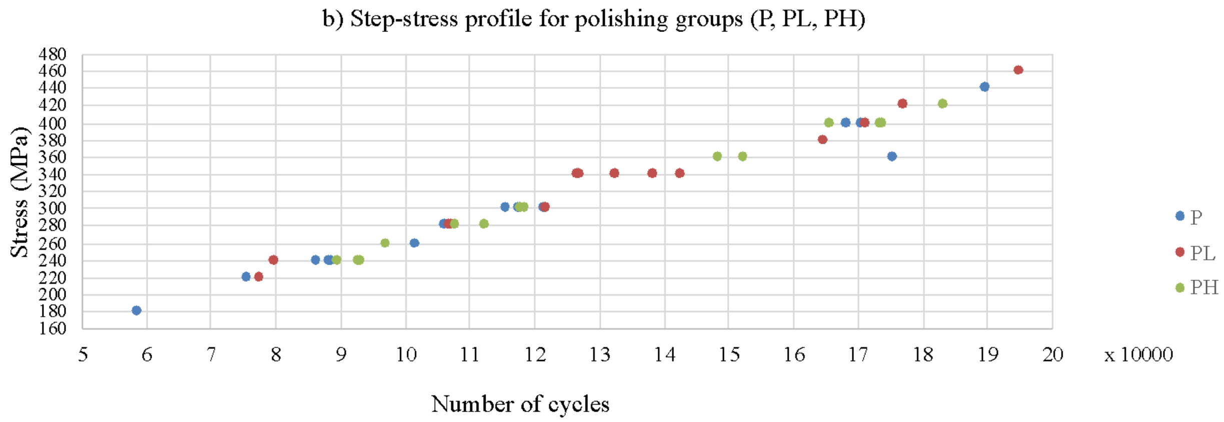


Figure 2. Step-stress profiles of machining (a) and polishing (b) groups.

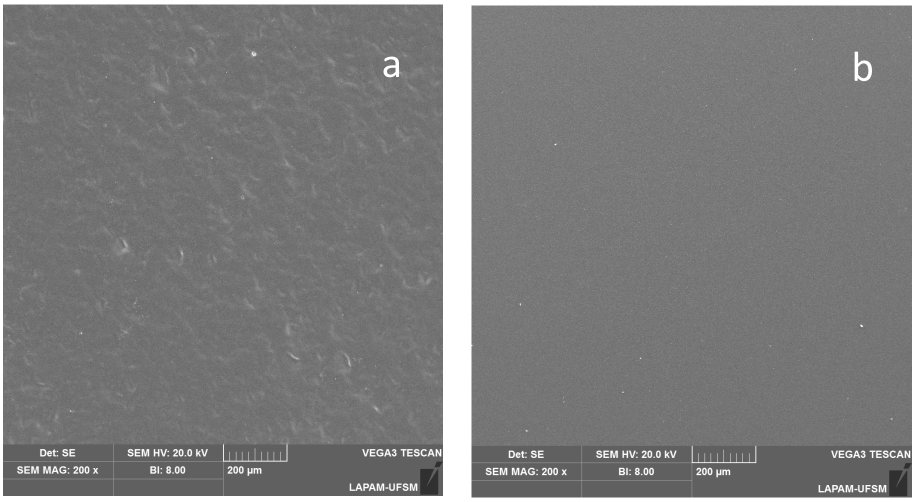
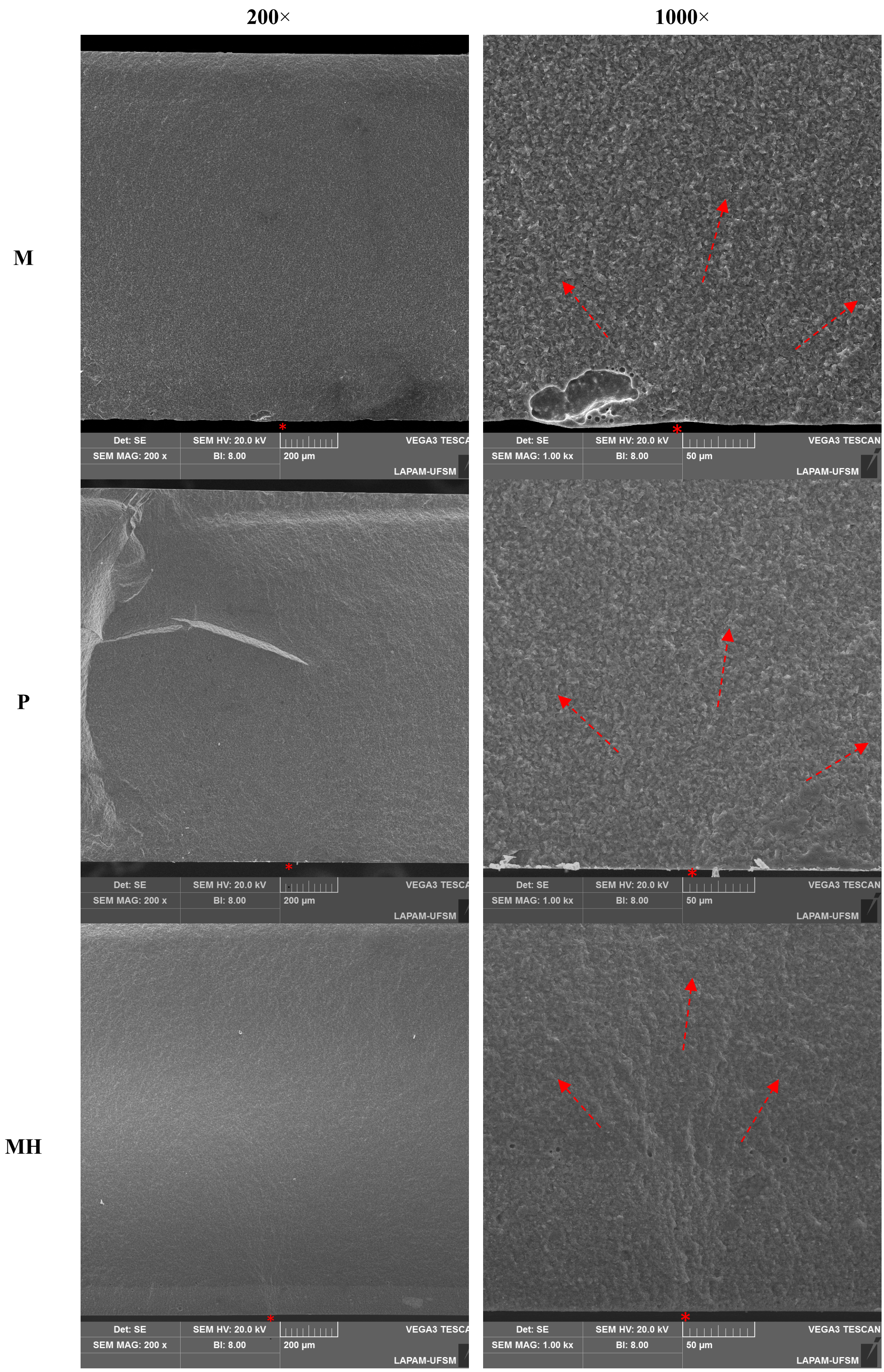


Figure 3. Representative SEM images (200× magnification) of machining (a) and polishing (b) groups. The micrographs depicted a rougher surface for machining.


200×
1000×
M
P
MH
Det: SE
SEM HV: 20.0 kV
SEM MAG: 200 x
BI: 8.00
200 μm
SEM MAG: 1.00 kx
50 μm
VEGA3 TESCAN
LAPAM-UFSM

PH

ML

PL

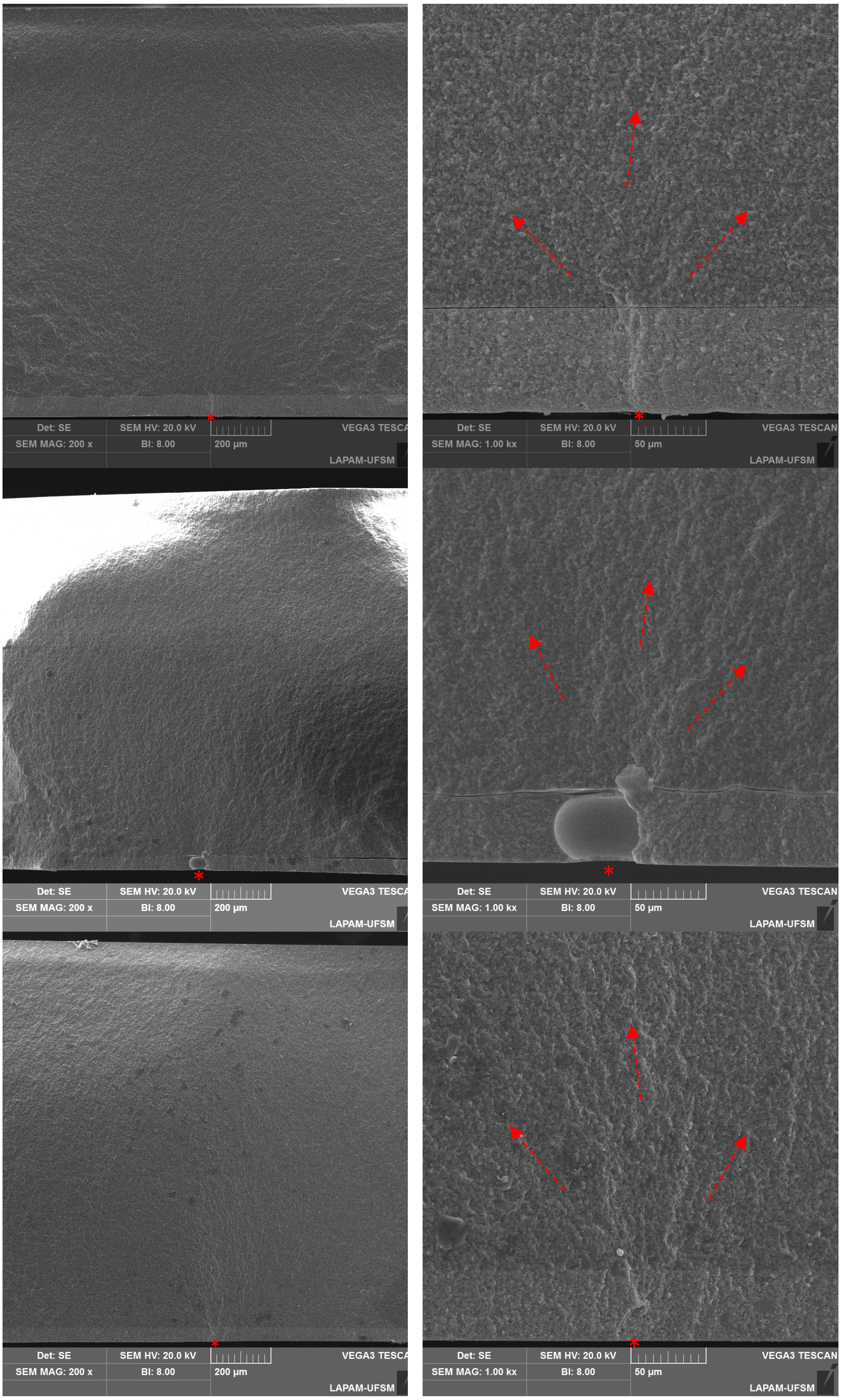


Figure 4. Representative SEM micrographics of fractographical examination at 200× and 1000× magnification. The asterisk (*) indicate that all fracture origins are located at the tensile side. M depicts a failure origin at a surface defect nearby a material intrinsic pore; P presents a failure origin not related to any specific surface characteristic, suggesting high energy accumulated before failure; MH, ML, PH and PL shows failures originating in the cement layer and

propagating through the ceramic; ML present a large bubble in cement, associated to the failure origin, and a line indicating that cement is detaching from ceramic, which is also depicted in the PH specimen. Red arrows indicate fracture propagation direction (hackles).

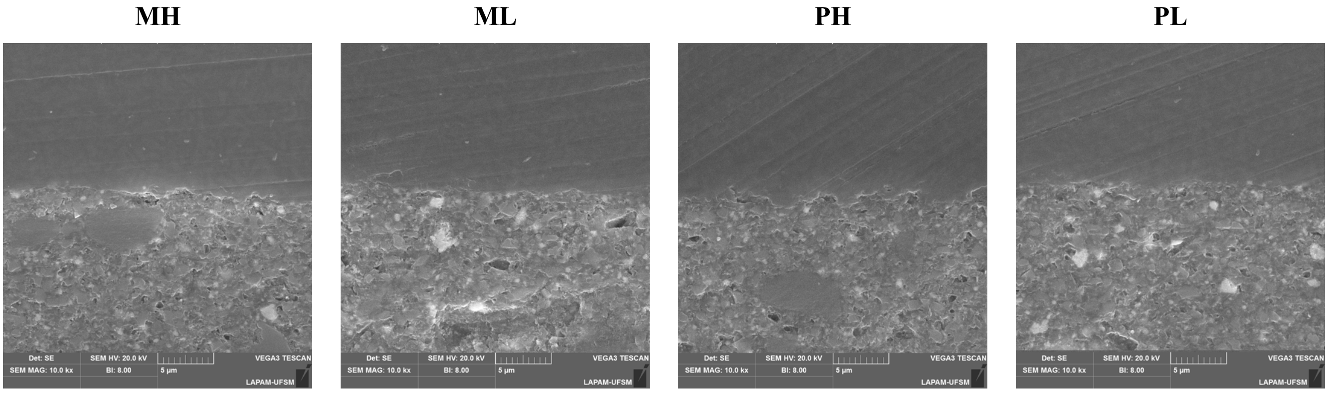


Figure 5. SEM micrographs (10000×) illustrating the intaglio surface between the ceramic and the cement, where it notices the intimacy achieved in all conditions explored.

5 ARTIGO 3 – IS THE ADHESIVE CEMENTATION ABLE TO REVERT THE NEGATIVE IMPACT OF MACHINING ON THE FATIGUE BEHAVIOR OF LITHIUM DISILICATE GLASS-CERAMIC?

Este artigo científico será submetido ao periódico Dental Materials, Elsevier, ISSN: 0109-5641, Fator de impacto = 4.070; Qualis A1. As normas para publicação estão descritas no Anexo B.

Is the adhesive cementation able to revert the negative impact of machining on the fatigue behavior of lithium disilicate glass-ceramic?

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Abstract

Objectives. To compare characteristic fatigue failure load (FFL_0), characteristic cycle for fatigue failure and survival probability of machined and polished lithium disilicate discs bonded to dentin analog with high and low viscosity resin luting agents.

Methods. Disc-shaped specimens (n = 15, Ø = 12 mm; thickness = 1.3 mm) of CAD-CAM lithium disilicate (IPS e.max CAD) were prepared and divided according to surface processing (machining (M) – CEREC inLab; and polishing (P) – laboratory procedures) and cement viscosity (high (H) and low (L)). The specimens were treated with self-etching primer (Monobond Etch and Prime) and adhesively cemented (Variolink N Base + High or Low viscosity catalyst) to dentin analogue discs (G10 - NEMA grade G10) (Ø = 12 mm; thickness = 2.2 mm). The cemented assemblies were subjected to fatigue testing using a step-stress approach (500−1500 N; step-size of 100 N; 10,000 cycles per step; 20 Hz). Weibull statistics were conducted on fatigue data (95% CI). Fractographic and topographic analyzes were also performed on scanning electron microscope (SEM).

Results. Machined groups presented statistically lower fatigue performance (FFL_0 MH 1192 [1111 – 1278]; ML 1105 [1077 – 1134]) than their polished controls (FFL_0 PH 1350 [1279 – 1424]; PL 1333 [1276 – 1392]). No differences were found between cement viscosity, regardless of surface characteristic.

Significance. It is well known that CAD-CAM machining impair mechanical performance of lithium disilicate glass-ceramics. Adhesive cementation was not able to revert this negative impact, regardless of cement viscosity. Cement viscosity did not affect fatigue results.

Key words: Computer-aided design, computer-aided manufacturing, dental ceramics, milling, load-to-failure, mechanical behavior, adhesion

1. Introduction

Lithium disilicate is a synthetic glass-ceramic that has become popular in restorative dentistry due to its good optical and mechanical properties. It has higher flexural strength compared to earlier glass-ceramics, like feldspathic and leucite reinforced, and better optical properties compared to polycrystalline ceramics, like zirconia [1]. This ceramic is commercially available in ingots, for heat-pressing processing technique, and compacted blocks, for processing via Computer aided design - computer aided manufacturing (CAD-CAM) systems [2]. Although in modest numbers, clinical failures, such as fractures, have been reported [3,4] and understanding the mechanical performance of this ceramic, as a consequence of laboratorial and clinical procedures, is necessary to predict their behavior [5,6].

Ceramics have a brittle nature, supporting little or no plastic deformation before fracturing [7]. Their strength relies on microstructure and flaw population characteristics around the failure origin [5], which becomes especially critical in areas of tensile stresses concentration, as the ceramic cementation surface [8–11]. Failures commonly derives from radial cracks formation on this surface [8,10,12], especially when subjected to cyclic stresses from mastication or fatigue testing [12,13]. Another factor that plays a critical role in brittle all-ceramic failures is subcritical crack growth (SCG), aggravated by a humid environment, such as the oral cavity. This mechanism typically leads to failure at stress levels below the critical value [5,14].

CAD-CAM systems were developed in an attempt to improve the mechanical properties of ceramics using standardized ceramic blocks processed under industrial conditions, resulting in more homogeneous and reliable materials [15,16]. However, CAD-CAM machining can induce damage at the ceramic surface and subsurface, affecting roughness and strength of the restoration [17–20].

The fabrication of all-ceramic restorations using CAD-CAM systems involves a subtractive machining process in which the prefabricated ceramic block is shaped in the desired geometry by way of the cutting edges of hard irregular diamond particles present on the grinding tool [17,21,22]. This process induces strain at the ceramic surface, resulting in micro-fractures and material loss, generating a new surface texture [21,23]. In spite of these micro-fracture events are necessary for the restoration design, the contact between the machining tool and the ceramic surface also results in formation of cracks and plastic contact damage culminating in compressive residual stresses localized on and beneath the newly formed surface [5,23,24], limiting the strength of the machined object [20,25–27].

Machining damage can act as starting points for fracture at the inner surface of ceramic restorations [28], since stress accumulation around critical flaws are responsible for crack initiation [29,30]. This was demonstrated in a study showing a decrease of about 33% in the characteristic strength of lithium disilicate specimens after machining in comparison with polishing [18].

The clinical success and longevity of ceramic restorations also depends on a satisfactory bond between the ceramic and dental substrate [31]. A successful bonding mechanism combines micro-mechanical interlocking and chemical bonding [32,33], which can be satisfactorily achieved through adhesive cementation with resin-based luting agents [32,34–36]. Adhesive cementation of all-ceramic restorations has been related to their better mechanical performance [37–41]. The strengthening effect caused by resin luting agents can be explained by a formation of a resin-ceramic hybrid layer resulting from the resin inter-penetrating the ceramic surface [39], sensitive to ceramic surface texture [40], but independent of defect severity [38]. In this way, the strength of ceramic restorations increases when the adhesion mechanism occurs [9], improving the transmission of the stress through the bonded interface [42].

The strength of ceramic restorations can be also influenced by the support properties, as elastic modulus, and cement type and thickness [9,12,43–45]. The authors have recently studied the impact of a resin-cement coating on the fatigue flexural strength of lithium disilicate glass-ceramic submitted to machining or polishing conditions (personal communication) and the damage outcome of machining was reverted. However, a cement coating does not reproduce the clinical situation, where specimens are bonded to a substrate. In this way, ceramic bonding to a dentin analog material is important to mimic the clinical environment [12].

Additionally, the ceramic strengthening magnitude following cementation procedures depends on the resin's ability to interpenetrate surface defects [46], which is determined by the wetting efficiency of the resin cement [47]. Luting agents with distinct physical properties may not behave the same way [48], once that intrinsic and mechanical properties of the resin-based material and the resin-coating thickness can influence the ceramic reinforcement [46]. In order to expand the range of clinical applications, resin-based materials with changes in viscoelastic properties were formulated, altering the proportion between resin matrix and filler particle content [49]. Low viscosity version promises minor pellicle thickness, smaller polymerization shrinkage [49] and better interaction with the ceramic surface [46]. Thus, the behavior of luting agents with different viscosities should be investigated, as they can influence the surface defects filling and the final thickness of the cement layer.

The aim of this study was to compare the fatigue behavior of machined and polished lithium disilicate discs cemented to dentin analog with high and low viscosity resin cements. The first hypothesis was that there is no difference in fatigue failure load between polished and machined ceramic specimens and the second hypothesis was that there is no difference in the fatigue behavior of ceramic cemented with high and low viscosity cement.

2. Materials e methods

In the present study a simplified assembly designed to represent a posterior tooth restoration cemented to a dentin analog substrate was submitted to fatigue testing. The occlusal restoration is represented by a lithium disilicate disc with 12 mm diameter and 1.3 mm thickness, adhesively cemented onto fiberglass epoxy resin discs (NEMA grade G10, International Paper, Hampton, SC, USA - dentin analogue material) with the same diameter and a thickness of 2.2 mm, resulting in an assembly with final thickness of 3.5 mm, which are equivalent to the average thickness from pulp wall to occlusal surface [50]. Before testing, the G10 side was glued to a steel ring with an outer diameter of 12 mm and an inner diameter of 6.5 mm, simulating the pulp chamber [50].

Compositions, trade names and manufacturers of the materials used in this study are presented in Table 1.

2.1 Specimen preparation

The fabrication of ceramic discs (Ø= 12 mm; thickness= 1.3 mm; IPS e.max CAD, Ivoclar Vivadent) was carried out by two distinct ways: machining in a Computer Aided Design-Computer Aided Manufacturing (CAD–CAM) machine, to generate restorations with machined surfaces, and cutting processed CAD-CAM blocks in laboratory, to produce specimens with polished surfaces [25].

Machining was executed in a CEREC inLab MC XL unit (Sirona Dental Systems Gmbh, Bensheim, Germany). Firstly, the design of the discs (Ø = 12 mm; thickness = 1.6 mm; chamfer = 0.1 mm) was made by a CAD software, using the Non-uniform rational basis spline (NURBS) language (Rhinoceros 6.0SR8, McNell North America, Seattle, WA) (Fig. 1a), and then exported in Standard Tessellation Language (STL) format to CAM software (inLab 18.1, Dentsply Sirona, Charlotte, NC, USA) (Fig 1b). The type of restoration was selected as “crown” mode. One set of diamond burs, one stepped and one cylindrical (Step bur 12S, reference 6240167, batch E98995; Cylinder pointed bur 12S, reference 6240159, batch M75905; Dentsply Sirona, Bensheim, Germany), were used for milling. After machining, the upper surface of the discs was adjusted using 240, 400, 600, and 1200 grit silicon carbide paper (Norton Saint-Gobain, Guarulhos, São Paulo, Brazil) in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, IL, USA) with constant water irrigation [18] to achieve the final measure (Ø = 12 mm; thickness = 1.3 ± 0.01 mm).

Polished discs specimens were fabricated reducing the CAD-CAM ceramic blocks into cylinders (12 mm diameter) by grinding in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, IL, USA) with a coarse diamond grinding disc (Apex CGD, PSA, Green 240 µm, 8in, Buehler, Lake Bluff, IL, USA) followed by 240, 400 and 600-grit silica carbide papers (Norton Saint-Gobain, Guarulhos, Sao Paulo, Brazil) under water. After this procedure, slices (1.5 ± 0.10 mm thickness) were obtained of cylinders using a diamond saw under water lubrication in a precision cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA), with a blade speed of 300 rpm. Then, the surfaces of the discs were polished with 240, 400, 600 and 1200-grit silica carbide papers (Norton Saint-Gobain, Guarulhos, São Paulo, Brazil) in the same polishing machine under water [18] to achieve the final measure (Ø = 12 mm; thickness = 1.3 ± 0.01 mm).

After finishing, the lithium disilicate discs were crystallized in a Vita Vacumat 6000 MP oven (Vita Zahnfabrik, Germany), following the manufacturer's recommendation (pre-drying temperature 403 °C for 6 min; temperature increase (under vacuum from 550–820 ºC) at the rate of 90 °C/min until reaching the firing temperature 820 °C, staying for 0.1 min; new increase of temperature (under vacuum from 820–840 ºC) at the heating rate 30 °C/min until reaching the firing temperature 840 °C, staying for 7 min; long-term cooling temperature 700 °C) [51]. After crystallization, the specimens were measured with a digital micrometer (Mitutoyo absolute 500-196-20 Digital Caliper; Takatsu- Ku, Kawasaki, Kanagawa, Japan), reaching 12 mm in diameter and 1.3 ± 0.01 mm in thickness.

Dentin analog discs were cutting from cylindrical bars of fiberglass epoxy resin with diameter of 12 mm (NEMA grade G10, International Paper, Hampton, SC, USA) in a precision cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA) using a diamond saw (blade speed 150 rpm), resulting in slices with 2.3 ± 0.10 mm thickness. The G10 discs were polished with 1200-grit silicon papers (Norton Saint-Gobain, Guarulhos, São Paulo, Brazil) in the polishing machine under water until reach the final thickness of 2.2 mm.

A single trained operator performed the polishing and adjustment procedures of all specimens.

2.1.1 Surface roughness and topographic analysis

The roughness of the bottom surface of each ceramic specimen was recorded prior to cementation using a contact profilometer (SJ-410, Mitutoyo, Japan). The parameters Ra (average surface roughness) and Rz (arithmetic mean peak-to-valley height) were obtained by three measurements, transversal to the machining path, according to the ISO 4287:1997 (cut-off 5; λC of 0.8 mm; λS of 2.5 μm) [52]. After randomization (section 2.2), a statistical software program (IBM SPSS Software; IBM, Armonk, NY, USA) was used to evaluate the roughness data (Ra and Rz parameters) of the groups, which assumed a non-parametric distribution (Shapiro-Wilk test). Thus, Kruskal-Wallis and Dunn's post-hoc tests were performed, with a significance level of 0.05. Machining groups - MH, ML - showed statistically higher roughness values (Ra 1.19 (0.12) and 1.20 (0.10); Rz 7.59 (0.61) and 7.45 (0.69)) than polishing groups - PH, PL - (Ra 0.04 (0.01) and 0.05 (0.01); Rz 0.31 (0.04) and 0.31 (0.04)). Roughness values were similar, when comparing groups with the same surface characteristic ($P>0.05$).

Representative specimens of machining and polishing groups were ultrasonically cleaned with 78% isopropyl alcohol bath for 5 min (1440 D, 50/60 Hz, Odontobras), alloy sputtered and examined by scanning electron microscope (SEM - Vega3, Tescan, Brno, Czech Republic) at 200× magnification, under a high vacuum with 20.00 kV at a working distance of approximately 13.5 mm for topographic analysis.

2.2. Experimental groups

Using the software Random Allocator (www.random.org), the specimens machined and polished (control) were randomly assigned into two groups each, resulting in four experimental groups: (1) machining and cementing with high viscosity resin cement (MH); (2) machining and cementing with low viscosity resin cement (ML); (3) polishing and cementing with high viscosity resin cement (PH); (4) polishing and cementing with low viscosity resin cement (PL).

2.3 Cementation procedures

The lithium disilicate and G10 discs were cleaned in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ind. And Com. Equip. Odonto. LTDA, Ribeirao Preto, São Paulo, Brazil) with 78% isopropyl alcohol for 5 min prior to the cementation procedure. After drying, the bonding surface of G10 discs was etched with 10% hydrofluoric acid (Condac Porcelana, FGM, Joinville, Brazil) for 60 s, rinsed with air-water spray for 30 s and ultrasonically cleaned in distilled water for 5 min. Two clicks of dental adhesive (Tetric N-Bond Universal, Ivoclar Vivadent, Schaan, Liechtenstein) was dispensed with an applicator packaging (VivaPen – Click

& Bond, Ivoclar Vivadent, Schaan, Liechtenstein) attached to a disposable brush cannula, that was scrubbed onto the G10 surfaces for 20 s and gently air-dried until a glossy, immobile film layer resulted. Then, the adhesive was light cured (Radii-cal LED curing light, SDI, Bayswater, Australia) for 10 s, according manufacturer recommendation [53].

The bonding surface of ceramic discs was conditioned with a self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent), actively rubbed with a microbrush for 20 s, kept reacting for 40 s, rinsed with air-water spray for 30 s and air dried for 30 s, following the manufacturer recommendation [54].

The dual-cure resin cements (base + catalyst High Viscosity or base + catalyst Low Viscosity) (Variolink N, Ivoclar Vivadent; Schaan, Liechtenstein) were manipulated following the manufacturer's instructions [55] and applied onto the ceramic intaglio surface, which was immediately seated on top of the corresponding pre-treated G10 disc, centrally positioned on a loading platform. Then, the upper ceramic surface was loaded with a 2.5 N load for 6 ± 1 min. Resin cement excesses were removed using a microbrush and the assembly was light-cured (Radii-cal LED curing light, SDI, Bayswater, Australia) for five exposures of 20 s each (one in each direction of 5 positions: 0º, 90º, 180º, 270º, and on top). The cemented specimens were stored at 37 ºC in distilled water from 24 h to 7 days until the fatigue testing.

Each step of the cementation procedures was conducted by the same trained operator.

2.4 Fatigue test

The fatigue failure load data were obtained using the step-stress approach. The assemblies (n=15) were submitted to cyclical intermittent loads at a frequency of 20 Hz [56] in an electrodynamic machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA) under water, by a hemispherical stainless-steel piston (Ø = 40 mm) [12], centered positioned on the ceramic occlusal surface. An adhesive tape (110 µm) was placed on the upper surface of the cemented assembly before testing to reduce contact stress concentration and avoid surface contact damage [8].

An initial load of 500 N were applied per 5,000 cycles to adjust the sample/piston contact, followed by incremental steps of 100 N per 10,000 cycles until failure. The specimens were inspected by oblique light transillumination for crack detection after each step [57]. When failure was found, testing was concluded and the fatigue failure load and number of cycles for fatigue failure were recorded for data analysis.

2.5 Fractographic and bonded interfaces analyzes

After transillumination inspection, a representative specimen from each group were selected for fractographic analysis. Ceramic fragments were detached with a scalpel from the G10 substrate to access the defect origin. The fragments were ultrasonically cleaned 78% isopropyl alcohol (5 min), gold-sputtered and analyzed under scanning electron microscopy (SEM; Vega3, Tescan, Brun, Czech Republic) at 200× and 1000× magnification to determine the crack propagation direction.

Samples of each group were selected and transversely sectioned in a cutting machine (Isomet 1000, Buehler) with a diamond saw under water cooling on a region away from the crack area to allow analysis of the adhesive interface. Then, the samples were polished (EcoMet/AutoMet 250, Buehler) using 600, 1200 and 2000 grit silica carbide papers (Norton Saint-Gobain, Guarulhos, São Paulo, Brazil), cleaned in an ultrasonic bath with 78% isopropyl alcohol (5 min), gold-sputtered and analyzed in the scanning electron microscope (SEM - Vega3, Tescan, Brno, Czech Republic) at 10000× magnification.

2.6 Data analysis

Fatigue failure load and number of cycles for fatigue failure data was submitted to Weibull analysis using the Super SMITH Weibull 4.0k-32 software (Wes Fulton, Torrance, USA) under the maximum-likelihood method to obtain the characteristic fatigue failure load and characteristic cycle for fatigue failure (expressing a failure probability of approximately 63.3%), as well as Weibull modulus (m), as a way to statistically access the reliability of a condition/parameter.

Also, the survival probability was tabulated for each load step of the test.

3. Results

Representative topographic images depicted a rougher surface for machining when compared to polishing condition (Fig. 3).

The machining groups had lower fatigue performance (characteristic failure load, characteristic cycle for failure and survival probabilities) compared to their respective polished control groups (MH < PH; ML < PL). However, no statistically significant difference could be observed among the resin cement viscosities, regardless of surface characteristic (MH ~ ML; PH ~ PL) (Table 2, Table 3 and Fig. 2).

ML presented the higher Weibull modulus (m_c) among the tested groups for cycles for fatigue failure and no statistically significant difference were found between the other groups.

However, with respect to fatigue failure load, Weibull modulus (m_f) for ML was superior only to the MH group (Table 2).

Representative SEM micrographs of the fracture occurrences are presented in Fig. 4. It can be observed that the origin of the cracks occurred from the face submitted to tensile stress. SEM images of the ceramic-resin bonded interfaces (Fig. 5) showed that the resin-cements were able to maintain close intimacy to the ceramic surface. However, the major continuity of the ceramic-cement bonding interface could be seen in the low viscosity version.

3. Discussion

Mechanical behavior of dental ceramics is highly affected by their flaw population, notably critical when placed in tensile stress concentration sites [12,58]. CAD-CAM machining process comprises surface removal with grinding diamond tools, generating chip fragments by fracture dominant mechanisms, inducing stress concentration, crack propagation and developing a new surface texture [17,21]. However, this approach creates chipping defects and subsurface damage, which from a crack can propagate and lead to catastrophic failure [24]. Thus, the flexural strength of CAD-CAM glass-ceramic is significantly reduced by machining procedures [18,20,25–27]. Also, the surface characteristics introduced by machining and pre-bonding proceedings might influence the wettability and interlocking of the resin cement and affect the mechanical behavior of the ceramic restoration [1,20,59,60]. This study showed that adhesive cementation of lithium disilicate were not able to revert the negative effect caused by machining on their fatigue behavior, regardless of cement viscosity used. The first hypothesis, that there is no difference in fatigue failure load between polished and machined ceramic specimens was rejected.

These findings are not in agreement with the results of a previous study conducted by the authors (personal communication), that found a positive effect of a resin-cement coating on the fatigue flexural strength of machined lithium disilicate, equivalent to that of polished groups. These differences can be explained because in the previous study the authors used lithium disilicate discs in a bilayer methodology, isolating the influence of the cement on the ceramic surface. In such configuration, the stresses generated due to the polymerization shrinkage of resin-based cements could maintain the ceramic with residual compressive stresses over the surface flaws, requiring more energy to reach the critical tensile stress which initiate the crack propagation and leads to a catastrophic failure [60]. Despite the strengthening effect of resin cements over ceramics are sensitive to the characteristics of a cement-ceramic hybrid layer, mediated by complex interactions [39], some deflection studies confirm a link between resin shrinkage stresses and the reinforcement of vitreous ceramics [61–64]. Additionally, 3D-finite element analysis (FEA) simulating the stresses generated during biaxial flexural tests of bilayer specimens showed that tensile stresses propagate in a conical shape from the cement layer through the center of the ceramic disc, increasing stress concentration at the luting agent layer and reducing stresses reaching the ceramic disc [65]. In contrast, at the test configuration of the current study, presenting a ceramic layer consistently adhered to and supported by a material with lower elastic modulus, the highest tensile stress concentration under occlusal loading occurs at the bottom surface of the ceramic layer, at the interface with the cement [8,43], increasing the role of surface and subsurface damages on the crack propagation, which may evolve into fractures.

Machining resulted in a roughness expressively higher than polishing (Fig. 3). De Kok et al. (2017) [1] investigated the effect of internal surface roughness at bonded and non-bonded condition on the load to failure of a lithium disilicate-based glass ceramic. The authors found that the roughness of inner surface impair the ceramic mechanical properties when it was not properly bonded to a substrate, but that is reverted when adhesion occurs. The lithium disilicate average roughness investigated by the authors was similar to achieved with machining procedures in the current study, however, a direct comparison cannot be made as the authors used air-abrasion instead of machining, which induces a cascade of events on the lithium disilicate surface and subsurface, that results in radial and lateral cracks, chipping, damage, and residual stress concentration [23,24]. In this way, even exhibiting topographic similarity, small differences in the flaw population and subsurface damage changed the stress concentration around the defects, and different fatigue behavior are achieved [66]. On the other hand, corroborating the findings of this study, Pilecco et al. (2021) [66], investigating in-lab simulations of CAD-CAM, found an even greater difference between the fatigue behavior of machined and polished specimens (29% of reduction in fatigue failure load of machining group).

Luting agents film thickness may influence the ceramic strength [9,43,44]. The actual thickness of the cement is determined by handling, ratio, temperature and material properties, with the viscosity playing a crucial role [43,59]. Theoretically, the use of cement with low viscosities allows a formation for a thinner cement layer [49], as well as, promote a better infiltration of the luting agent between the surface irregularities [46,67] and generates smaller polymerization shrinkage, reducing the possibility of gaps formation and premature marginal leakage [9,49].

Resin composites are filled suspensions of inorganic particles in an organic matrix, which gives these materials viscoelasticity [68]. The base of a wide range of cements, as the investigated in this study, contain 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropyl)phenyl]-propane (Bis-GMA), considered a very viscous monomer. For improving its rheological properties and allow a better incorporation of inorganic fillers, less viscous monomers (e.g., triethylene glycol dimethacrylate; urethane dimethacrylate) are added as a diluent for uncured pastes [49,69]. Altering the proportion of the base monomers is one way to change the viscoelasticity of resinous materials [49]. Another way is altering the filler content [70], that directly impact de elastic modulus of the material [48].

Low and high viscosity versions investigated (Table 1) caused no difference in the fatigue behavior of the ceramic, accepting the second hypothesis (Table 2, Fig. 2), in spite of the fraction variation of constituents of resin matrix and filler particles. These findings are in agreement with one previous study, in which no differences were found between high and low cement viscosities with regard to lithium disilicate fatigue strength (personal communication). However, a better continuity in the bonding interfaces seem to be seen in specimens cemented with low viscosity resin-cement (Fig. 5).

Concerning to reliability of the experimental parameters, ML group showed the higher Weibull modulus (m_c) for cycles for fatigue failure (Table 2). Actually, all the specimens of ML group failed between steps 1000 N and 1100 N (Table 3), conferring the lowest variation on its confidence interval (4751 cycles). Regarding the Weibull modulus (m_f) of fatigue failure load, ML group present better reliability than MH group (Table 2). These findings could suggest that the matrix/filler content of low viscosity cement, when interpenetrated with the rougher surface of machined specimens, present a very foreseeable behavior, while the high viscosity luting agent, which has a little more filler and lesser matrix content (Table 1), seems to have no predictability of complete filling of defects (Fig. 5), generating greater scattering of results.

The humidity present in the oral environment and cyclic loads can reduce the lifetime of glass-ceramics, since this material is susceptible to slow crack growth phenomena [5,71]. Resin-cement fill the surface defect population and may inhibit this phenomenon by limiting moisture exposure to flaw tip [47]. However, hydrophilic composite, such as those based on BisGMA/TEGDMA or UDMA, can undergo hygroscopic expansion and matrix plasticization under water exposure, leading to localized loss of adhesion, that can result in areas of stress concentrations upon loading at the resin-ceramic interface [47]. Simulate the stress/load as close as possible to the clinical situation is imperative in mechanical tests [59]. Thus, fatigue testing at a wet environment was conducted in this work [12]. According to Velho et al. (2020) [56], a frequency of 20 Hz during fatigue testing proved to be a viable alternative to accelerate the test, without interfering with the fatigue behavior and the lithium disilicate failure pattern. Hence, for a better use of time, an accelerated fatigue approach was conducted in this study. Additionally, caution was taken with respect to the load applicator. The hemispheric stainless-steel piston generated tensile stress in the cementation surface of the discs, where the failures started from, as observed in the fractographic analysis (Fig. 4), corresponding to failures clinically detected [12]. Further, a substrate with similar properties to hydrated dentin (G10) was used in an attempt to mimic the complexity of natural teeth [12].

Despite all efforts, the complexity of masticatory loads during function and the challenges of the oral environment limit direct extrapolation of the results in clinical situations. Moreover, the limitations may include the use of simplified restorations (disc to disc set-up) and the absence of thermocycling or longer periods of sample storage. Nevertheless, the results of this study hopefully guide manufacturing advances. Machining reduced the fatigue failure load of lithium disilicate ceramic by approximately 15% in comparison to polishing condition after bonding. These findings highlight the need for developing less harmful tools, which enable the incorporation of a smaller number and size of defects, with better finishing, especially on the internal surfaces of restorations, where the most deleterious stresses to their structural integrity are concentrated. The impact of machining in the fatigue behavior on cemented ceramics with different microstructures should also be investigated.

4. Conclusion

CAD-CAM machining negatively affected the fatigue behavior of lithium disilicate ceramic and resulted in higher roughness pattern. This adverse impact cannot be reverted by adhesive cementation, regardless of cement viscosity utilized.

Similar behavior was found between high and low viscosities resin cements on the lithium disilicate fatigue failure load.

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TABLES

Table 1. List of materials used: commercial name, manufacturer, batch number and composition based on the manufacturer's information.

Material	Commercial name, manufacturer (batch number)	Composition
Lithium disilicate	IPS e.Max CAD, Ivoclar Vivadent (Y42922)	SiO_2 57-80 wt%, Li_2O 11-19 wt%, K_2O 0-13 wt%, P_2O_5 0-11 wt%, ZrO_2 0-8 wt%, ZnO 0-8 wt%, other and coloring oxide 0-12 wt%
Dentin analogue material	NEMA grade G10, International Paper	Woven glass-filled epoxy
10% Hydrofluoric acid	Condac Porcelana, FGM (051020)	10% hydrofluoric acid, water, thickener, surfactant and coloring
Dental adhesive	Tetric N-Bond Universal, Ivoclar Vivadent (Y25047)	Methacrylates, etanol, water, highly dispersed silicon dioxide, initiators and stabilizers
Self-etching ceramic primer	Monobond Etch & Prime, Ivoclar Vivadent (Y27772)	Ammonium polyfluoride, silane system based on trimethoxypropyl methacrylate, alcohols, water and colorant
Dual cure resin cement	Variolink N Base: White A1 (YZ1257) Catalyst High Viscosity: Yellow A3 (Y15071) Catalyst Low Viscosity: Transparent (YZ1263)	Monomer Matrix: Bis-GMA, urethane dimethacrylate and triethylene glycol dimethacrylate (Base 26.3 % wt.; Catalyst high viscosity 22 % wt.; Catalyst low viscosity 27.9 % wt.). Inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide. (Base 73.4 % wt.; Catalyst high viscosity 77.2 % wt.; Catalyst low viscosity 71.2 % wt.) Additional contents: initiators, stabilizers and pigments. Particle size is 0.04-3.0 µm, mean particle size is 0.7 µm.

Table 2. Mean and standard deviation for fatigue failure load and number of cycles for fatigue failure and Weibull analysis of fatigue data (characteristic load and characteristic cycle for fatigue failure; Weibull modulus - *m*) depicting 95% confidence intervals [CI].

Groups	Fatigue Failure Load (Mean (SD))* (N)	Characteristic fatigue failure load [95% CI] (N)	Weibull modulus for fatigue failure load m_f [95% CI]	Cycles for fatigue failure (Mean (SD))*	Characteristic cycle for fatigue failure [95% CI]	Weibull modulus for cycles for fatigue m_c failure [95% CI]
MH	1120.00 (43.86)	1192 [1111 – 1278]^{BC}	7.65 [5.14 – 11.38]^{B}	65915.87 (3880.16)	71793 [64771 – 79577]^{BC}	5.20 [3.46 – 7.80]^{B}
PH	1286.67 (36.34)	1350 [1279 – 1424]^{A}	10.02 [6.81 – 14.76]^{A,B}	82047.67 (3879.52)	88337 [80579 – 96841]^{A}	5.85 [4.01 – 8.55]^{B}
ML	1080.00 (14.48)	1105 [1077 – 1134]^{C}	20.74 [14.45 – 29.75]^{A}	60871.20 (1280.21)	63095 [60764 – 65515]^{C}	14.23 [9.61 – 21.07]^{A}
PL	1280.00 (31.17)	1333 [1276 – 1392]^{AB}	12.28 [8.35 – 18.06]^{A,B}	76251.87 (5375.85)	82329 [74040 – 91546]^{AB}	4.88 [3.14 – 7.60]^{B}

Different letters in each column indicate statistically significant differences for fatigue data (95% CI fail in overlapping).
*Descriptive data without statistical analysis

Table 3. Survival probabilities per step with respective standard errors.

Group	Fatigue failure load/Number of cycles for fatigue failure										
	500 N/ 5 x 10^3	**600 N/ 10 x 10^3**	**700 N/ 10 x 10^3**	**800 N/ 10 x 10^3**	**900 N/ 10 x 10^3**	**1000 N/ 10 x 10^3**	**1100 N/ 10 x 10^3**	**1200 N/ 10 x 10^3**	**1300 N/ 10 x 10^3**	**1400 N/ 10 x 10^3**	**1500 N/ 10 x 10^3**
MH	1	1	1	1	0.80 (0.10)	0.60 (0.13)	0.40 (0.13)	0.33 (0.12)	0.07 (0.06)	0.0	-
PH	1	1	1	1	1	1	0.87 (0.09)	0.47 (0.13)	0.33 (0.12)	0.20 (0.10)	0.0
ML	1	1	1	1	1	0.73 (0.11)	0.67 (0.64)	0.0	-	-	-
PL	1	1	1	1	1	1	0.80 (0.10)	0.67 (0.12)	0.27 (0,11)	0.07 (0.06)	0.0

* The symbol '-' indicates absence of specimens being tested on the respective step.

FIGURES

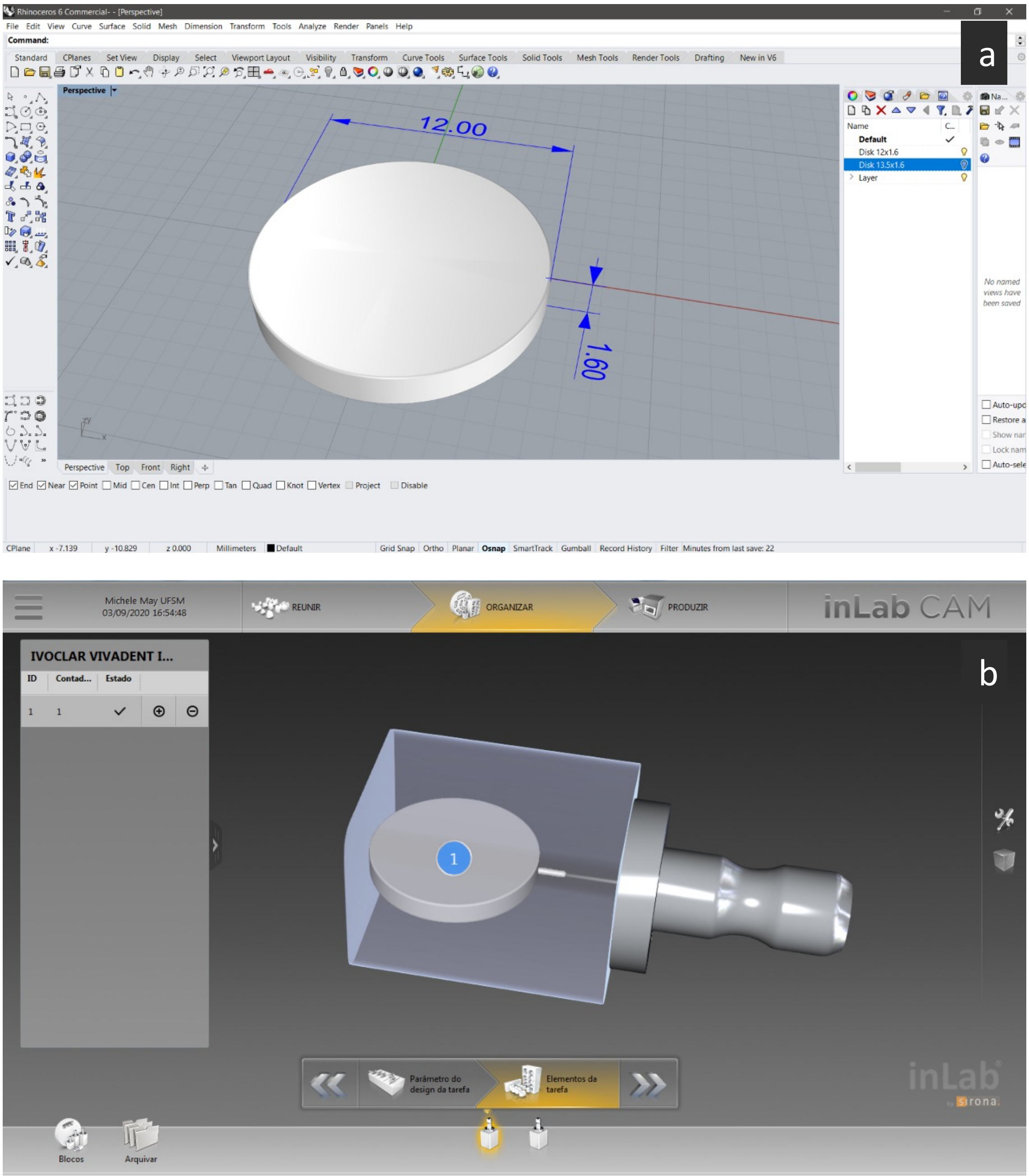

Figure 1. Rhinoceros interface of disc-shaped project (NURBS language) (a) and design imported to CAM software depicting the disc inside the ceramic block (b).

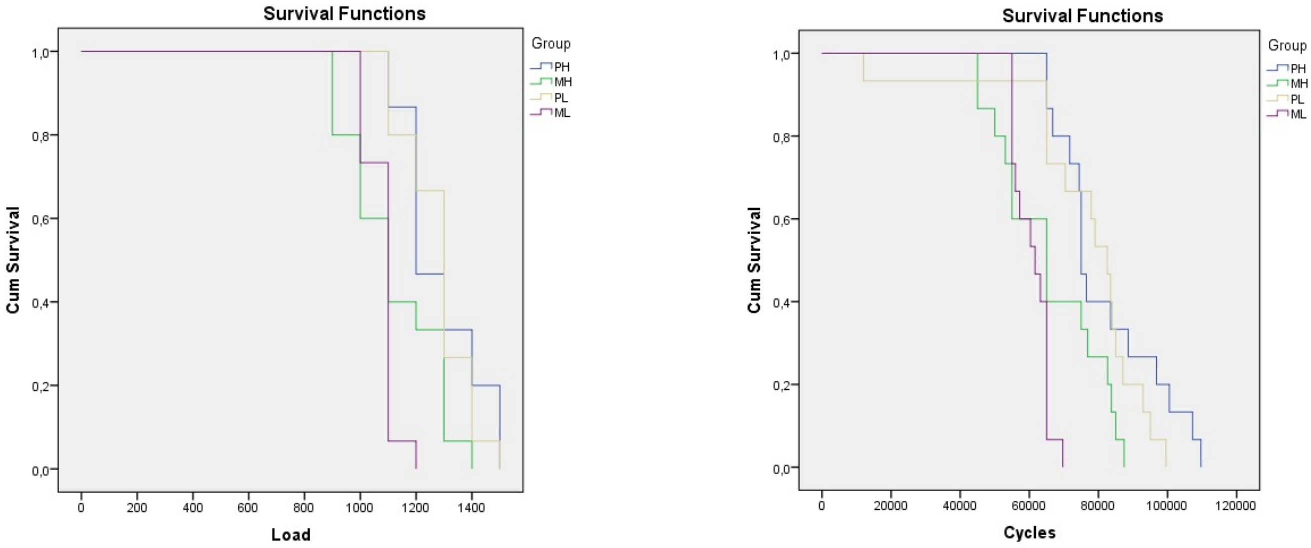

Figure 2. Survival curves according to the steps of fatigue failure load (left) and number of cycles (right) in which each disc failed.

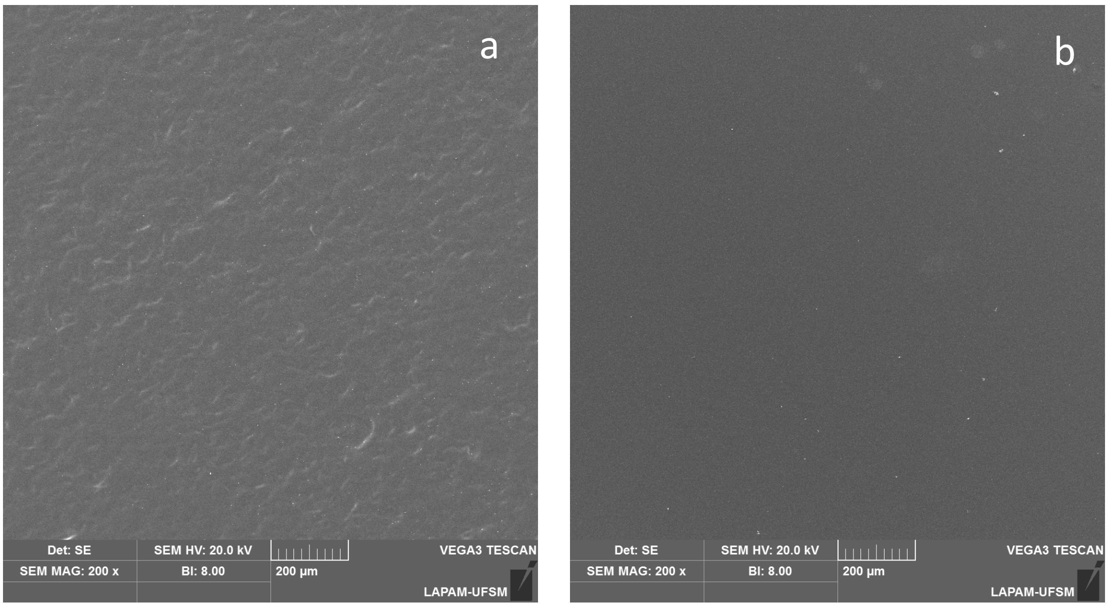

Figure 3. Representative SEM images (200× magnification) of machined (a) and polished (b) surfaces. The micrographs depicted a rougher surface for machining.

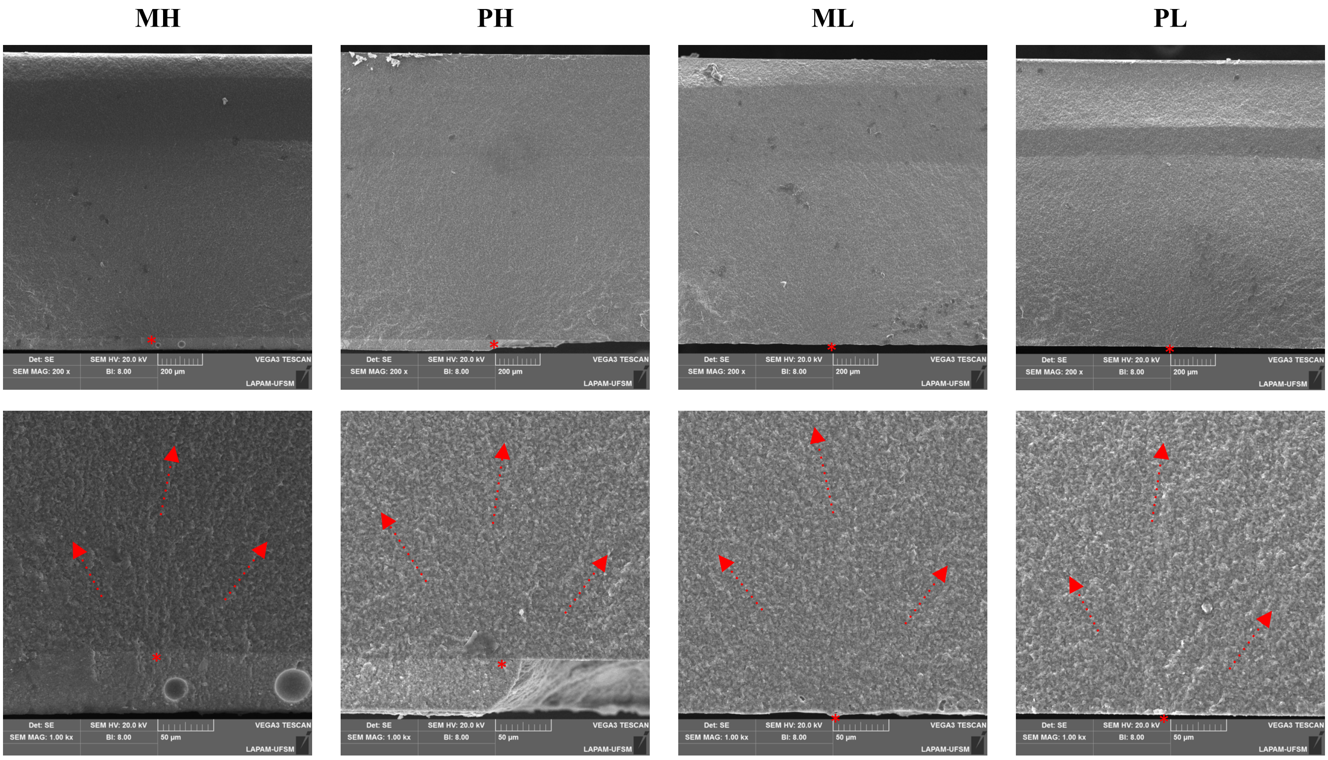

Figure 4. Representative SEM micrographics of fractographical examination at 200× (top images) and 1000× (bottom images) magnification. Asterisks (*) indicate that all fracture origins are located at the tensile side. Red arrows indicate the direction of fracture propagation (hackles).

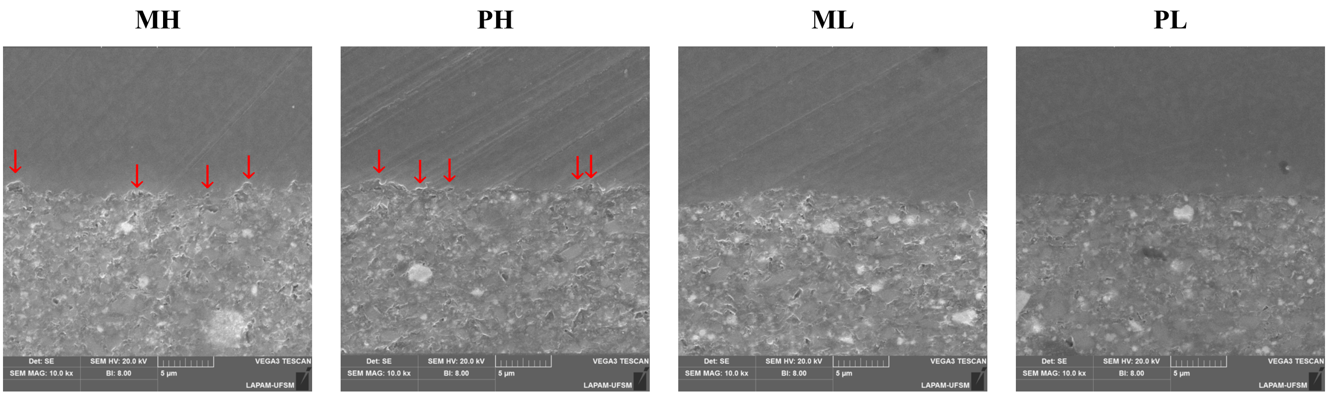

Figure 5. Micrographs (10,000×) obtained by SEM illustrating the intaglio surface between ceramic (top) and cement (bottom). It can be noticed the presence of unfilled areas (pointed by red arrows) between high viscosity resin-cement and ceramic surface. Low viscosity resin-cement seems to lead to a better filling of ceramic surface topography.

6 DISCUSSÃO

A presente tese contextualiza diversos fatores que podem interferir na resistência final de uma restauração de cerâmica vítrea reforçada por partículas para usinagem em sistema CAD-CAM, especialmente da cerâmica de dissilicato de lítio, desde seu processamento até o assentamento final da peça, com foco nos agentes cimentantes de alta e baixa viscosidade empregados.

O primeiro estudo (Capítulo 3) buscou confirmar as evidências de que a usinagem em sistema CAD-CAM teria um efeito deletério à restauração usinada, que sustentariam a condução dos demais estudos. A investigação foi ampliada para outros fatores que pudessem resultar do processamento (tratamento) da peça antes que ela fosse cimentada e incluiu outras cerâmicas vítreas na análise, de modo a contribuir com a literatura científica acerca dessa gama específica de materiais.

Segundo esta revisão sistemática, a usinagem e ajustes para adaptação da restauração com brocas diamantadas tiveram um impacto significativo no aumento da rugosidade e no enfraquecimento da peça para todas as cerâmicas investigadas. A unidade de usinagem CAD-CAM (Cerec) usa brocas com partículas de diamante de 64 µm (uma broca apresentando degraus e outra cilíndrica) operando a 42.000 rpm em cerâmicas totalmente ou parcialmente sinterizadas (CURRAN et al., 2017). Mesmo utilizando líquido lubrificante para resfriamento, essa abordagem está associada com deformações plásticas, fraturas e geração de calor dentro do material (REKOW; THOMPSON, 2005). Danos de superfície residuais de aproximadamente 9-15 µm de profundidade foram reportados para as vitrocerâmicas após usinagem com o sistema CEREC precursor, sendo responsáveis pelo desenvolvimento das fraturas envolvendo a superfície interna das restaurações (KELLY et al., 1991). A sequência de usinagem também teve efeito na redução da resistência. Para diminuir a influência dessa e de outras variáveis desconhecidas, os espécimes foram randomizados nos estudos conduzidos posteriormente (Capítulos 4 e 5).

O condicionamento com ácido fluorídrico teve resultados diferentes, dependendo da microestrutura do material, do tempo de condicionamento e da concentração do ácido fluorídrico utilizado. Para a cerâmica de dissilicato de lítio, um impacto significativo na resistência pode ser observado quando concentrações de 4,9% de ácido fluorídrico foi utilizado por 20 s ou mais. Nos estudos subsequentes (Capítulos 4 e 5) foi utilizada uma abordagem diferente, utilizando um primer autocondicionante (Monobond Etch & Prime). O tratamento de superfície da cerâmica com primer autocondicionante tem demonstrado resistência de união

(EL-DAMANHOURY; GAINTANTZOPOULOU, 2018; ROMÁN-RODRÍGUEZ et al., 2017; TRIBST et al., 2018), resistência flexural (LIMA et al., 2021) e desempenho de fadiga (DAPIEVE et al., 2020; SCHESTATSKY et al., 2019) semelhantes aos do ácido fluorídrico seguido da aplicação de silano, mas demonstrou tendência a uma maior confiabilidade mecânica (SCHESTATSKY et al., 2019) e a representar uma opção menos prejudicial em relação à alteração de superfície (MURILLO-GÓMEZ; PALMA- DIBB; DE GOES, 2018).

Para que uma adequada união ocorra entre cimento e cerâmica são necessárias interações químicas e físicas. A união física pode ser aprimorada tornando a superfície cerâmica mais rugosa, resultando em um aumento da área de superfície disponível para adesão e um maior potencial para embricamento micromecânico (WOLF et al., 1993; LACY et al., 1995). Como a usinagem da peça torna a superfície mais rugosa em decorrência do processamento, é esperado que a união à cerâmica seja potencializada por esta característica. Além disso, o fortalecimento da cerâmica pela ação do cimento resinoso foi reportado na literatura por diversos estudos (MALAMENT; SOCRANSKY, 1999; MARQUIS, 1992, PAGNIANO et al., 2005, FLEMING et al., 2006, ADDISON; MARQUIS; FLEMING, 2007; FLEMING et al., 2017, BARBON et al., 2018). Assim, a dúvida sobre se a maior área de superfície das cerâmicas usinadas para embricamento com o cimento resinoso, uma provável superioridade na capacidade adesiva proporcionada por essa característica, além da ação de fortalecimento do cimento resinoso, seria capaz de compensar o efeito danoso causado pela usinagem nas propriedades mecânicas do material ficou latente.

O segundo estudo (Capítulo 4) foi delineado para avaliar o efeito do cimento sobre a superfície cerâmica usinada, através de uma configuração bicamada. Dessa forma, cimentos com propriedades reológicas distintas foram investigados (alta e baixa viscosidades). Esse tipo de investigação vem sendo utilizado em muitos estudos para avaliação de cerâmicas menos resistentes, como feldspáticas obtidas pela técnica convencional (pó e líquido) (ADDISON; MARQUIS; FLEMING, 2007; 2008; SPAZZIN et al., 2016) e obtidas pela secção dos blocos para CAD-CAM em máquina de cortes (SPAZZIN et al., 2017; BARBON et al., 2018; 2019; COELHO et al., 2019). Não foram encontrados estudos que utilizassem essa metodologia para avaliar cerâmicas relativamente mais resistentes, reforçadas por partículas, como a cerâmica de dissilicato de lítio, tampouco processadas por usinagem pelo sistema CAD-CAM, bem como o seu comportamento à fadiga flexural. Pode-se observar um efeito protetor do cimento, compensando o dano causado pela usinagem. Entretanto, não se observou diferenças entre o comportamento das duas viscosidades de cimento investigadas.

Nessa configuração de teste, observou-se uma íntima relação do cimento com a superfície cerâmica. Pode-se atribuir os resultados às teorias de reforço previamente descritas na literatura, como a interação da camada híbrida cimento-cerâmica, onde o cimento faz uma constrição dentro dos defeitos, e passa a se comportar de maneira semelhante à cerâmica ao seu redor (WANG, 1995; ADDISON; MARQUIS; FLEMING, 2007). O mesmo efeito de reforço não foi observado na cerâmica polida, que se comportou de maneira similar à cerâmica sem cobertura, provavelmente pela menor presença de defeitos superficiais. Além disso, para essa configuração de corpos de prova, o efeito de contração de polimerização proposto por Nathanson (1993) deixa a cerâmica sob tensões compressivas residuais, efeito comprovado por estudos de deflexão (FLEMING et al., 2017) e não está sujeita ao fator de configuração de cavidade (Fator C) (FEILZER; DE GEE; DAVIDSON, 1987).

Assim, tornou-se imperativo avaliar se o efeito de reforço visualizado nesse segundo estudo seria mantido quando a cerâmica estivesse aderida a um substrato, com módulo de elasticidade semelhante à dentina, que melhor representasse a situação clínica. O terceiro estudo (Capítulo 5) utilizou uma configuração de restauração simplificada (disco-cimento-disco), comparando discos usinados *vs*. polidos e o efeito das diferentes viscosidades do cimento (alta e baixa) na carga para falha em fadiga, número de ciclos para falha e probabilidade de sobrevivência de espécimes de dissilicato de lítio cimentados em análogo de dentina (G10). Nesta configuração de teste, observou-se que o reforço causado na cerâmica pela ação do cimento foi proporcionalmente menor, com relação ao segundo estudo, não conseguindo compensar os danos introduzidos pela usinagem em sua resistência. Novamente, o comportamento das duas viscosidades do cimento foi semelhante.

Apesar de bons resultados de sobrevivência nos poucos estudos clínicos envolvendo as restaurações de dissilicato de lítio usinadas (AKIN; TOKSAVUL; TOMAN, 2014; SEYDLER; SCHMITTER, 2015; RAUCH et al., 2018; AZIZ et al., 2019), fraturas da cerâmica ainda são uma realidade (RAUCH et al., 2018). Essa realidade pode ser ainda maior na prática clínica, onde diversas variáveis envolvidas podem não ter sido consideradas em ensaios controlados devido aos critérios de elegibilidade dos pacientes. Assim, fatores que podem limitar a sobrevida das restaurações devem ser estudados e minimizados. Observou-se nos estudos que os defeitos introduzidos pela usinagem ainda são importantes, apesar de todos os avanços nos sistemas durante os últimos anos e do efeito de reforço conferido pelos cimentos resinosos. Entretanto, é importante salientar que outros sistemas de usinagem e cerâmicas com diferentes microestruturas ou diferentes espessuras podem se comportar de maneira distinta. Além disso, a complexidade das cargas mastigatórias durante a função e os desafios do meio bucal limitam a extrapolação direta dos resultados em situações clínicas. Outro fator limitante foi não termos conduzido envelhecimento. No entanto, os resultados desta tese orientam a necessidade de avanços nos sistemas de usinagem, através do desenvolvimento de ferramentas menos agressivas, que possibilitem a incorporação de menor número e tamanho de defeitos, com melhor acabamento, especialmente nas superfícies internas das restaurações, onde se concentram as tensões mais nocivas à sua integridade estrutural. Trabalhos futuros devem se concentrar em técnicas de mitigação de danos no desempenho mecânico das cerâmicas submetidas à usinagem em sistema CAD-CAM.

7 CONCLUSÃO

A usinagem das restaurações vitrocerâmicas (incluindo a cerâmica à base de dissilicato de lítio) e os ajustes realizados com brocas diamantadas impactam negativamente sua resistência estrutural e aumentam a sua rugosidade superficial.

O efeito do condicionamento com ácido fluorídrico é dependente da microestrutura da vitrocerâmica, do tempo de aplicação e da concentração do mesmo. Para a cerâmica de dissilicato de lítio, condicionamento com ácido fluorídrico com concentração superior a 4,9% e tempo de condicionamento de 20 s ou mais diminuem a sua resistência flexural sem afetar a sua rugosidade superficial.

A adesão ao cimento resinoso proporciona um reforço na cerâmica de dissilicato de lítio usinada. Entretanto, quando a cerâmica é cimentada a um substrato análogo à dentina, esse reforço não é suficiente para reverter os danos oriundos da usinagem.

Cimentos de alta e baixa viscosidades se comportam de maneira semelhante no que diz respeito ao reforço da cerâmica de dissilicato de lítio.

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ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *THE JOURNAL OF PROSTHETIC DENTISTRY*

Article Types

Articles are classified as one of the following: research/clinical science article, clinical report, technique article, systematic review, or tip from our readers. Required sections for each type of article are listed in the order in which they should be presented.

Research and Education/Clinical Research

The research report should be no longer than 10-12 double-spaced, typed pages and be accompanied by no more than 12 high-quality illustrations. Avoid the use of outline form (numbered and/or bulleted sentences or paragraphs). The text should be written in complete sentences and paragraph form.

Abstract (approximately 400 words): Create a structured abstract with the following subsections: Statement of Problem, Purpose, Material and Methods, Results, and Conclusions. The abstract should contain enough detail to describe the experimental design and variables. Sample size, controls, method of measurement, standardization, examiner reliability, and statistical method used with associated level of significance should be described in the Material and Methods section. Actual values should be provided in the Results section.

Clinical Implications: In 2-4 sentences, describe the impact of the study results on clinical practice.

Introduction: Explain the problem completely and accurately. Summarize relevant literature, and identify any bias in previous studies. Clearly state the objective of the study and the research hypothesis at the end of the Introduction. Please note that, for a thorough review of the literature, most (if not all references) should first be cited in the Introduction and/or Material and Methods section.

Material and Methods: In the initial paragraph, provide an overview of the experiment. Provide complete manufacturing information for all products and instruments used, either in parentheses or in a table. Describe what was measured, how it was measured, and the units of measure. List criteria for quantitative judgment. Describe the experimental design and variables, including defined criteria to control variables, standardization of testing, allocation of specimens/subjects to groups (specify method of randomization), total sample size, controls, calibration of examiners, and reliability of instruments and examiners. State how sample sizes were determined (such as with power analysis). Avoid the use of group numbers to indicate groups. Instead, use codes or abbreviations that will more clearly indicate the characteristics of the groups and will therefore be more meaningful for the reader. Statistical tests and associated significance levels should be described at the end of this section.

Results: Report the results accurately and briefly, in the same order as the testing was described in the Material and Methods section. For extensive listings, present data in tabular or graphic form to help the reader. For a 1-way ANOVA report of, F and P values in the appropriate location in the text. For all other ANOVAs, per guidelines, provide the ANOVA table(s). Describe the most significant findings and trends. Text, tables, and figures should not repeat each other. Results noted as significant must be validated by actual data and P values.

Discussion: Discuss the results of the study in relation to the hypothesis and to relevant literature. The Discussion section should begin by stating whether or not the data support rejecting the stated null hypothesis. If the results do not agree with other studies and/or with accepted opinions, state how and why the results differ. Agreement with other studies should also be stated. Identify the limitations of the present study and suggest areas for future research.

Conclusions: Concisely list conclusions that may be drawn from the research; do not simply restate the results. The conclusions must be pertinent to the objectives and justified by the data. In most situations, the conclusions are true for only the population of the experiment. All statements reported as conclusions should be accompanied by statistical analyses.

References: See Reference Guidelines and Sample References page.

Tables: See Table Guidelines.

Illustrations: See Figure Submission and Sample Figures page.

Clinical Report

The clinical report describes the author's methods for meeting a patient treatment challenge. It should be no longer than 4 to 5 double-spaced, pages and be accompanied by no more than 8 high-quality illustrations. In some situations, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Abstract: Provide a short, nonstructured, 1-paragraph abstract that briefly summarizes the problem encountered and treatment administered.

Introduction: Summarize literature relevant to the problem encountered. Include references to standard treatments and protocols. Please note that most, if not all, references should first be cited in the Introduction and/or Clinical Report section.

Clinical Report: Describe the patient, the problem with which he/she presented, and any relevant medical or dental background. Describe the various treatment options and the reasons for selection of the chosen treatment. Fully describe the treatment rendered, the length of the follow-up period, and any improvements noted as a result of treatment. This section should be written in past tense and in paragraph form.

Discussion: Comment on the advantages and disadvantages of the chosen treatment and describe any contraindications for it. If the text will only be repetitive of previous sections, omit the Discussion.

Summary: Briefly summarize the patient treatment.

References: See Reference Guidelines and Sample References page.

Illustrations: See Figure Submission and Sample Figures page.

Dental Technique

The dental technique article presents, in a step-by-step format, a unique procedure helpful to dental professionals. It should be no longer than 4 to 5 double-spaced, typed pages and be accompanied by no more than 8 high-quality illustrations. In some situations, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Abstract: Provide a short, nonstructured, 1-paragraph abstract that briefly summarizes the technique.

Introduction: Summarize relevant literature. Include references to standard methods and protocols. Please note that most, if not all, references should first be cited in the Introduction and/or Technique section.

Technique: In a numbered, step-by-step format, describe each step of the technique. The text should be written in command rather than descriptive form ("Survey the diagnostic cast" rather than "The diagnostic cast is surveyed.") Include citations for the accompanying illustrations.

Discussion: Comment on the advantages and disadvantages of the technique, indicate the situations to which it may be applied, and describe any contraindications for its use. Avoid excessive claims of effectiveness. If the text will only be repetitive of previous sections, omit the Discussion.

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Systematic Review

The author is advised to develop a systematic review in the Cochrane style and format.
The Journal has transitioned away from literature reviews to systematic reviews. For more information on systematic reviews, please see www.cochrane.org. An example of a Journal systematic review: Torabinejad M, Anderson P, Bader J, Brown LJ, Chen LH, Goodacre CJ, Kattadiyil MT, Kutsenko D, Lozada J, Patel R, Petersen F, Puterman I, White SN. Outcomes of root canal treatment and restoration, implant-supported single crowns, fixed partial dentures, and extraction without replacement: a systematic review. J Prosthet Dent 2007;98:285-311.

The systematic review consists of:

An Abstract using a structured format (Statement of Problem, Purpose, Material and Methods, Results, Conclusions).

Text of the review consisting of an introduction (background and objective), methods (selection criteria, search methods, data collection and data analysis), results (description of studies, methodological quality, and results of analyses), discussion, authors' conclusions, acknowledgments, and conflicts of interest. References should be peer reviewed and follow JPD format.

Tables and figures, if necessary, showing characteristics of the included studies, specification of the interventions that were compared, the results of the included studies, a log of the studies that were excluded, and additional tables and figures relevant to the review.

Tips From Our Readers

Tips are brief reports on helpful or timesaving procedures. They should be limited to 2 authors, no longer than 250 words, and include no more than 2 high quality illustrations. Describe the procedure in a numbered, step-by-step format; write the text in command rather than descriptive or passive form ("Survey the diagnostic cast" rather than "The diagnostic cast is surveyed").

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The number of authors is limited to 4; the inclusion of more than 4 must be justified in the letter of submission. (Each author's contribution must be listed.) Otherwise, contributing authors in excess of 4 will be listed in the Acknowledgments. There can only be one corresponding author.

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- Abbreviate units of measurement without a period in the text and tables (9 mm). Insert a nonbreaking space between all numbers and their units (100 mm, 25 MPa) except before % and °C. There should never be a hyphen between the number and the abbreviation or symbol except when in adjectival form (100-mm span).

- Spell out “degrees” for angles. Use the degree symbol only for temperature, include a space between the number and degree symbol (e.g., 37 C).
- Contractions such as don’t, it’s, wouldn’t, etc are not used in scientific writing.
- Avoid using the words ""respectively"" or ""former/latter."" Both force the reader to stop and backtrack.
- For the common statistical outcomes P, a, ß omit the zero before the decimal point as these cannot be greater than 1.
- Proprietary names function as adjectives. Nouns must be supplied after their use, as in Vaseline petroleum jelly. Wherever possible, use only the generic term.
- Do not use trademark symbols as they are not consistent with Journal style.

Some Elements of Effective Style

- Short words. Short words are preferable to long ones if shorter word is equally precise.
- Familiar words. Readers want information that they can grasp easily and quickly. Simple, familiar words provide clarity and impact.
- Specific rather than general words. Specific terms pinpoint meaning and create word pictures; general terms may be fuzzy and open to varied interpretations.
- Brisk opening. Plunge into your subject in the first paragraph of the article.
- Limited use of modifying words and phrases. Check your adjectives, adverbs, and prepositional phrases. If they are not needed, strike them out.
- No unnecessary repetition. An idea may be repeated for emphasis—so long as that repetition is effective.
- Short sentence length. Twenty words or less is recommended. Rambling sentences cluttered with subordinate clauses and other modifiers are hard to read and may cause readers to lose their train of thought. Short sentences should, however, be balanced with somewhat longer ones to avoid monotony.
- Paragraphs. Break up long sections into paragraphs but avoid the use of single sentence paragraphs.
- Restraint. Writers who use flamboyant words or overstate their proposition or conclusions discredit themselves. Facts speak for themselves.
- Clearly stated conclusions. Don’t hedge. If you don’t know something, say so.

Objectionable Terms

The following are selected objectionable terms and their proper substitutes. For a complete list of approved prosthodontic terminology, consult the eighth edition of the Glossary of Prosthodontic Terms (J Prosthet Dent 2005;94:10-92).

Or visit JPD http://www.prosdent.org and click on Collections/Glossary of Prosthodontic Terms.

- Alginate use Irreversible hydrocolloid
- Bite use Occlusion
- Bridge use Partial fixed dental prosthesis
- Case use Patient, situation, or treatment as appropriate

- Cure use Polymerize
- Final use Definitive
- Freeway space use Interocclusal distance
- Full denture use Complete denture
- Lower (teeth, arch) use Mandibular
- Model use Cast
- Modeling compound use Modeling plastic impression compound
- Muscle trimming use Border molding
- Overbite, overjet use Vertical overlap, horizontal overlap
- Periphery use Border
- Post dam, postpalatal seal use Posterior palatal seal
- Prematurity use Interceptive occlusal contact
- Saddle use Denture base
- Study model use Diagnostic cast
- Take impressions, photographs, radiographs use Make
- Upper (teeth, arch) use Maxillary
- X-ray, roentgenogram use Radiograph

In addition, specimen should be used rather than sample when referring to an example regarded as typical of its class.

Additional Terminology Guidelines

Acrylic
An adjective form that requires a noun, as in acrylic resin.

Affect, effect
Affect is a verb; effect is a noun.

African American
Spelled thus and preferred over Negro and black in both adjective (African American patients) and noun (… of whom 20% were African Americans) forms.

Average, mean, median
Mean and average are synonyms. Median refers to the midpoint in a range of items; the midpoint has many items above as below it.

Basic
Like fundamental, this word is often unnecessary. An example of unnecessary use: Dental implants consist of two basic types: subperiosteal and endosteal.

Between, among
Use between when 2 things are involved and among when there are more than 2.

Biopsy
This noun should NOT be used as a verb. A biopsy was performed on the Tissue, rather than: The tissue was biopsied.

Centric
An adjective that requires a noun, as in centric relation.

Currently, now, at present, etc.
These expressions are often unnecessary, as in: This technique is currently being used…

Data
Use as a plural, as in: The data were…

Employ
Should not become an elegant variation of use, as in This method is employed …

Ensure
Preferred over insure in the sense of to make certain.

Fewer, less
Use fewer with nouns that can be counted (fewer patients were seen) and less with nouns that cannot be counted (less material was used).

Following
After is preferred.

Imply, infer
The speaker implies; the listener infers.

Incidence
The rate at which a disease occurs in a given time; sometimes confused with prevalence (the total number of cases of a disease in a given region).

Majority
Means more than half; use most when you mean almost all. **Male, female**
For adult humans, use men and women. For children, use boys and girls.

Must, should
Must means that the course of action is essential. Should is less strong and means that the course of action is recommended.

Numbers
Spell out numbers used in titles or headings and numbers at the beginning of a sentence. The spelled version may also be preferable in a series of consecutive numbers that may confuse the reader (eg, 2 3.5-inch disks should be written two 3.5-inch disks). In all other cases, use Arabic numerals.

Orient
Proper form; avoid orientate.

Pathologic
Use instead of pathological. Other words in which the suffix -al has been dropped include biologic, histologic, and physiologic.

Pathology
The study of disease; often mistaken for pathosis (the condition of disease)

Percent
Use the percent sign in the text, as in The distribution of scores was as follows: adequate, 8%; oversized, 23%; and undersized, 69%. But spell out when the percent opens a sentence, as in Twenty percent of the castings …

Prior to
Before is preferred.

Rare, infrequent, often not, etc.
Whenever possible, these vague terms should be backed up with a specific number.

Rather
Like very, this word should be avoided.

Regimen
A planned program for taking medication, dieting, exercising, etc. Not to be confused with regime, meaning a system of government or management.

Sex
Use "sex" rather than "gender" unless you are referring to the socially constructed roles, behaviors, activities, and attributes that a given society considers appropriate for men and women.

Symptomatology
The science or study of symptoms; this word is not a synonym for the word symptoms.

Technique
Preferred over technic.

Using
Avoid the dangling modifier in sentences such as "The impression was made using vinyl polysiloxane impression material." Write "with" or "by using" instead.

Utilize
Use is preferred.

Vertical
An adjective that needs a noun, as in vertical relation.

Via
Use through, with, or by means of.

White
Preferred over Caucasian. This is true only if the patient is from the Caucasus region of Eastern Europe. If not, use the term, white to describe the patient.

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ANEXO B – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *DENTAL MATERIALS*

GUIDE FOR AUTHORS

INTRODUCTION

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The Editor and Publisher reserve the right to make minimal literary corrections for the sake of clarity. Authors for whom English is not the first language should have their manuscripts read by colleagues fluent in English. If extensive English corrections are needed, authors may be charged for the cost of editing. For additional reference, consult issues of *Dental Materials* published after January 1999 or the Council of Biology Editors Style Manual (1995 ed.).

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"... an Au-Pd alloy (Estheticor Opal, Cendres et Metaux, Switzerland)."
• specify statistical significance test methods.

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• state implications of the results, relate to composition. • indicate limitations of findings.
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