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INFLUÊNCIA DO SUBSTRATO NO COMPORTAMENTO MECÂNICO EM FADIGA DE CERÂMICAS ODONTOLÓGICAS CIMENTADAS ADESIVAMENTE

Santa Maria, RS
2022

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

Orientador: Prof° Dr°. Luiz Felipe Valandro
Coorientadora: Profª Drª. Andressa Borin Venturini
Coorientador estrangeiro: Prof° Dr°. Cornelis Johannes Kleverlaan

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Luiz Felipe Valandro, Dr. (UFSM)
(Presidente/Orientador)

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João Paulo Mendes Tribst, Dr. (ACTA)

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Sara Fraga, Dra. (UFRGS)

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Dedico este trabalho

A minha família que me apoia em todos os momentos da minha caminhada e nas decisões que tomo. Nada seria possível sem uma base forte de amor e união. Tudo que conquisto é partilhado entre todos e reflexo da educação, da dedicação e do carinho que recebi. Todos são importantes para mim e fazem parte da conclusão de mais essa etapa.

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RESUMO

INFLUÊNCIA DO SUBSTRATO NO COMPORTAMENTO MECÂNICO EM FADIGA DE CERÂMICAS ODONTOLÓGICAS CIMENTADAS ADESIVAMENTE

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Três artigos científicos compõem essa tese com objetivo de avaliar o comportamento em fadiga de cerâmicas odontológicas frente a diferentes condições do substrato aos quais foram adesivamente cimentadas. No primeiro estudo, discos (n= 15; Ø= 10 mm; espessura= 0,7mm ou 1,0mm) de duas cerâmicas policristalinas a base de zircônia (ZR) foram cimentados sobre substratos de resina epóxi (RE), resina composta (RC) ou liga metálica (LM) para avaliar a influência de substratos com diferentes módulos elásticos (*E*) no comportamento mecânico em fadiga de duas gerações (segunda e terceira) de ZR em diferentes espessuras. Os espécimes foram ensaiados sob teste de fadiga acelerada (frequência= 20 Hz) com patamares de cargas crescentes até a presença de trincas ou carga máxima de 2800N. Os resultados de ZR cimentada sobre LM (maior *E*) foram superiores em ambas as gerações e espessuras avaliadas. A ZR de segunda geração foi superior à terceira geração em todas variáveis. Para espessuras, os espécimes de 1,0 mm apresentaram melhores resultados que 0,7 mm apenas para a ZR de segunda geração. No segundo estudo, discos (n= 15; Ø= 10 mm, espessura= 1,0 mm) de ZR, cerâmica feldspática (FEL), cerâmica vítrea reforçada por dissilicato de lítio (DL) e de uma rede cerâmica infiltrada por polímero (PICN) foram adesivamente cimentados a substratos de RE ou LM, também com objetivo de avaliar influência do *E* do substrato no comportamento em fadiga das restaurações cerâmicas. Foi realizado ensaio de fadiga acelerada com metodologia semelhante ao estudo anterior. Todas as cerâmicas avaliadas apresentaram resultados superiores quando cimentadas sobre o substrato de maior módulo elástico (LM). Nessa condição, DL apresentou resultados semelhantes à ZR, sendo ambos superiores à FEL e PICN. Sobre substrato de menor *E* (RC), PICN foi similar à ZR e DL, enquanto FEL apresentou resultados inferiores aos demais materiais avaliados. Análise de Elementos Finitos (FEA) foi executada para os dois primeiros estudos e corroborou os resultados encontrados no ensaio de fadiga. No terceiro artigo, objetivou-se avaliar a influência do tratamento de superfície do substrato no comportamento em fadiga de DL cimentada sobre eles. Discos de RC foram confeccionados simulando núcleos protéticos resinosos e submetidos à diferentes tratamentos da superfície após a remoção de cimento provisório. Discos de DL foram adesivamente cimentados sobre os discos de substrato tratados, e as amostras foram submetidas ao ensaio de fadiga (n= 15) com metodologia similar aos dois primeiros estudos, sendo a carga aplicada sobre o disco cerâmico. Amostras dos substratos foram também submetidos à análises topográficas, onde foram encontradas diferentes características na superfície do substrato de RC. Entretanto, nenhum protocolo influenciou no comportamento mecânico dos discos cerâmicos. Assim, conclui-se a partir da presente tese que o módulo de elasticidade tem efeito positivo no comportamento em fadiga quando as restaurações cerâmicas são cimentadas adesivamente sobre substratos de maior módulo de elasticidade, enquanto que a espessura dos discos cerâmicos das diferentes gerações de ZR teve influência apenas no comportamento mecânico da segunda geração. O tratamento de superfície do substrato não influenciou o comportamento mecânico da cerâmica de DL.

Palavras-chave: Carregamento cíclico. Cerâmicas Dentárias. Distribuição de Tensões. Módulo de elasticidade. Restaurações monolíticas.

ABSTRACT

INFLUENCE OF SUBSTRATE MATERIAL ON THE FATIGUE MECHANICAL BEHAVIOR OF ADHESIVELY LUTED DENTAL CERAMICS

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Three studies compose this thesis that aims to evaluate the fatigue mechanical behavior of dental ceramics in relation to the different conditions of the substrate to which they were adhesively luted. In the first study, discs (n= 15; Ø= 10 mm; thickness= 0.7 mm or 1.0 mm) of two zirconia-based ceramics (ZR) were cemented on substrates of epoxy resin (ER), resin composite (RC) or metallic alloy (MA) to evaluate the influence of the elastic modulus (*E*) of the substrate on the fatigue behavior of two generations (2^{nd}g and 3^{rd}g) of ZR with different thicknesses. The specimens were submitted to an accelerated fatigue test (frequency= 20 Hz) with increasing load levels until the presence of cracks or a maximum load of 2800N. ZR cemented onto MA (higher *E*) presented improved results in both generations and thicknesses. 2^{nd}g ZR had superior fatigue behavior than 3^{rd}g ZR. Regarding the thickness, the 1.0mm specimens had improved results than the 0.7mm only for the 2^{nd}g ZR group. In the second study, discs (n= 15; Ø= 10 mm, thickness= 1.0 mm) of ZR, feldspathic ceramic (FEL), lithium disilicate reinforced glass ceramic (LD) and polymer-infiltrated ceramic network (PICN) were adhesively cemented onto ER or MA substrates, also with the objective of evaluating the influence of the *E* of the substrate on the fatigue behavior of ceramic restorations. The accelerated fatigue test was performed similarly to the previous study. All the ceramics had better mechanical behavior when cemented onto MA (stiffer material), with LD presenting similar results to ZR, both superior to FEL and PICN. PICN was similar to ZR and DL when they were bonded onto a softer substrate (RC). In this condition, FEL showed lower results than the other materials. Finite Element Analysis (FEA) was performed for the first two studies, corroborating the results found in the fatigue test. The objective of the third study was to evaluate the influence of the surface treatment of the substrate on the fatigue behavior of lithium disilicate ceramics cemented onto the treated RC substrate. RC discs were made to simulate resin composite prosthetic cores and subjected to different surface treatments after the removal of temporary cement. LD discs were adhesively cemented onto the treated substrate discs, and the samples were subjected to a fatigue test (n= 15) with similar methodology to the first two studies, with the load application on the ceramic disc surface. Substrate samples were also submitted to topographic analysis, where different characteristics were found on the surface of the RC substrate, but no protocol influenced the mechanical behavior of LD. Thus, the modulus of elasticity had a positive effect on the fatigue behavior when the ceramic restorations were adhesively cemented onto stiffer substrates, while the thickness of the ceramic discs of the different generations of ZR influence only the mechanical behavior of the second generation of zirconia. The surface treatment of the substrate did not influence the mechanical behavior of the DL ceramic.

Keywords: Cyclic loading. Dental ceramics. Elastic modulus. Monolithic restorations. Stress distribution.

SUMÁRIO

1 INTRODUÇÃO

O desenvolvimento de materiais restauradores capazes de aliar propriedades mecânicas superiores e características ópticas que mimetizam os tecidos dentários têm permitido a evolução de cerâmicas odontológicas no sentido de aprimorar as características estéticas e restauradoras em situações clínicas de intensa solicitação mecânica (DENRY; KELLY, 2008; KWON et al., 2018). As diferentes opções disponíveis apresentam características e comportamentos distintos de acordo com a composição química e microestrutura do material (DA SILVA et al., 2017). Enquanto as cerâmicas vítreas apresentam estética superior devido sua porção vítrea que permite maior translucidez, as cerâmicas policristalinas reconhecidamente oferecem resistência mecânica elevada em virtude da característica típica de densificação de cristais como óxido de zircônio, que impedem a propagação de trincas no seu interior (ZHANG; KELLY, 2017).

A difusão desses materiais está também atrelada ao desenvolvimento dos sistemas *Computer assisted design/computer assisted machining* (CAD/CAM). Devido a possibilidade de as restaurações serem planejadas e executadas a partir de *softwares* para desenvolvimento anatômico da restauração monolítica (compostas por um único material) esse sistema oferece agilidade e previsibilidade aos tratamentos protéticos (BELLI, et al., 2017; DA SILVA et al., 2017). Além disso, cerâmicas usinadas por essas ferramentas oferecem vantagens consideráveis quando comparadas aos métodos de fabricação convencionais através de injeção e estratificação, devido a facilidade de processamento e menor presença de defeitos intrínsecos na sua estrutura (SEYDLER; SCHMITTER, 2015). Ainda, os materiais cerâmicos estão disponíveis em blocos homogêneos fabricados industrialmente com diferentes microestruturas, com número reduzido de falhas decorrentes do processamento (TINSCHERT et al., 2000) e, consequentemente, promovendo comportamento mecânico mais confiável frente às cargas mastigatórias (ZHANG; KELLY, 2017).

Originalmente, as cerâmicas feldspáticas tornaram-se amplamente utilizadas em restaurações indiretas devido sua similaridade estética aos elementos dentários. No entanto, devido à baixa resistência desse material, torna-se contraindicado para situações de alta solicitação estrutural, necessitando que uma infraestrutura metálica seja utilizada a fim de promover a resistência necessária para tornar a utilização desse material possível dentro de um conjunto complexo de forças e vetores diversos (ZHANG; KELLY, 2017). Com o intuito de eliminar a interferência dos metais no resultado estético, optou-se como alternativa acrescentar partículas de reforço às cerâmicas convencionais, aumentando-se a quantidade de matriz cristalina na composição do material cerâmico e, consequentemente, promovendo maior resistência mecânica do material (LAWN et al., 2004).

As partículas de reforço adicionadas às cerâmicas vítreas proporcionam o desvio da trajetória de propagação de trincas e, desta forma, impedem que a falha completa da restauração ocorra (DENRY; HOLLOWAY; ROSENSTIEL, 1998; GONZAGA et al., 2011). Dentre as cerâmicas vítreas reforçadas por partículas cristalinas destaca-se a cerâmica de dissilicato de lítio que devido a microestrutura de pequenos cristais em forma de agulha aleatoriamente interligados promove excelente resistência mecânica (ABOUSHELIB; SLEEM, 2014; ZHANG; SAILER; LAWN, 2013). Além disso, as propriedades ópticas desse material são muito satisfatórias, tornando-o amplamente utilizado na odontologia atual sobretudo associado à técnica digital por meio de sistemas CAD/CAM (GUARDA et al., 2013; KWON et al., 2018).

Em relação às cerâmicas policristalinas, destaca-se a zircônia tetragonal policristalina estabilizada por ítrio (Y-TZP), que têm como propriedades principais uma adequada estabilidade química e dimensional, resistência mecânica e tenacidade à fratura elevadas (PICONI; MACCAURO, 1999). Esse material possui três formas cristalinas de acordo com a temperatura em que se encontra: monolítica à temperatura ambiente, tetragonal entre 1170ºC e 2370ºC; e cúbica acima de 2370ªC (PICONI; MACCAURO, 1999). A partir da adição de óxido de ítrio (Y_2O_3) é possível manter a fase tetragonal estável mesmo em temperatura ambiente, possibilitando uma resistência elevada (PICONI; MACCAURO, 1999; STAWARCZYK et al., 2017). No entanto, a estabilização deve ser classificada como parcial, uma vez que pode ser desfeita quando o material é submetido à estímulos externos induzindo um processo chamado de tenacificação, onde os cristais em forma tetragonal retornam para forma monoclínica que, por terem maior volume, comprimem em uma dimensão microestrutural os defeitos gerados durante a solicitação da estrutura, prevenindo a propagação de trincas e melhorando o comportamento mecânico da cerâmica frente a tensões (KELLY; DENRY, 2008).

A primeira geração de zircônia odontológica apresentava como desvantagem a baixa translucidez, sendo considerada uma cerâmica de menor aceitabilidade estética (STAWARCZYK et al., 2017). Por esse motivo, o material foi indicado para confecção de infraestruturas, substituindo ao metal em coroas bicamadas, ou seja, infraestrutura confeccionada por Y-TZP recoberta com uma cerâmica vítrea de maior translucidez (KELLY; DENRY, 2008). No entanto, estudos clínicos apresentam o lascamento da cerâmica de cobertura como falha recorrente nessas restaurações (BELLI et al., 2016; SAILER et al., 2015). Desta forma, alterações estruturais foram realizadas a fim de promover maior translucidez para tornar possível a confecção de coroas monolíticas de Y-TZP (STAWARCZYK et al. 2017).

A primeira alteração se deu pela redução no número e tamanho dos óxidos de alumínio (Al_2O_3), o que permitiu melhor transmissão de luz nas então chamadas zircônias de segunda geração. Entretanto, essa alteração não foi suficiente para gerar melhorias estéticas a ponto de eliminar a necessidade de recobrimento cerâmico vítreo (STAWARCZYK et al. 2017). Na sequência evolutiva, a terceira geração surgiu a partir da modificação na estrutura cristalina, onde o aumento na proporção do óxido de ítrio (Y_2O_3) incorporou à estrutura tetragonal cristais em fase cúbica, que apresentam maior volume e permitem translucidez satisfatória para uso em coroas monolíticas (ZHANG et al., 2016). Por outro lado, a incorporação destes cristais cúbicos afeta negativamente na transformação de fase característica desse material (mecanismo de tenacificação) e, consequentemente, tornam a zircônia totalmente estabilizada por ítrio menos resistente que sua geração antecessora (STAWARCZYK et al., 2017).

Além da estética favorável, a possibilidade de confecção de coroas a partir de um único material (restaurações monolíticas) permite preparos protéticos menos invasivos (NAKAMURA et al., 2015). Em condições clínicas que exijam menor espessura oclusal, cerâmicas vítreas apresentam resistência limítrofe para resistir às cargas mastigatórias a que são submetidas (ZIMMERMANN et al., 2017). Em contrapartida, ainda que as alterações estruturais da terceira geração de zircônia tenham reduzido a resistência mecânica característica comparada à zircônia de segunda geração, coroas monolíticas com espessura menor que 1,0 milímetro (indicada para as cerâmicas vítreas reforçadas) são possíveis devido ao excelente comportamento mecânico das cerâmicas policristalinas (NORDAHL; VULT VON STEYERN; LARSSON, 2015; ZIMMERMANN et al., 2017). Nesse sentindo, compreende-se atualmente que restaurações indiretas em zircônia apresentam resistência mecânica suficiente para suportar cargas mastigatórias estáticas habituais e parafuncionais mesmo com espessura entre 0,5 e 1,5 mm (NORDAHL; VULT VON STEYERN; LARSSON, 2015; SORRENTINO et al., 2016).

Entretanto, a longevidade clínica destas restaurações totalmente cerâmicas no ambiente bucal está associada às cargas cíclicas a que são submetidas quando em função (ZHANG et al., 2006). Nesse sentido, os ensaios de fadiga cíclica reproduzem uma condição próxima do que ocorre clinicamente (WISKOTT; NICHOLLS; BELSER, 1995). A falha por fadiga ocorre de maneira súbita sob tensões inferiores a resistência nominal do material (BONFANTE; COELHO, 2016). Defeitos pré-existentes na superfície submetida a tração atuam como zona de concentrações de tensões e, sob aplicação de carga intermitente, sofrem crescimento lento e subcrítico até a falha completa da restauração (ZHANG et al., 2006). Através da interpretação dos dados de métodos de fadiga acelerados, são obtidas informações sobre número de ciclos/carga média para falha em fadiga, taxas de sobrevivência e confiabilidade de um tratamento restaurador (BASSO et al., 2016; SHEMBISH et al., 2016).

Dentre as propriedades mecânicas que caracterizam as cerâmicas odontológicas frente aos ensaios de fadiga, o módulo de elasticidade deve ser avaliado quando consideramos diferentes combinações de materiais presentes nas reabilitações protéticas (DAL PIVA et al., 2018; FACENDA et al., 2019; PEREIRA et al., 2019). Também chamado de módulo elástico ou módulo de Young, este refere-se à rigidez relativa de um material, sendo obtido através de uma constante de proporcionalidade entre as taxas de tensão necessária para causar determinada deformação (ANUSAVICE; SHEN; RAWLS, 2013). Em outras palavras, pode-se concluir que um alto módulo elástico indica uma grande capacidade de um material resistir a cargas elevadas sem se deformar, o que reduz o concentração de tensões nos defeitos presentes na região sob tensão e, consequentemente, melhora o comportamento em fadiga das restaurações.

Para restaurações monolíticas, isso pode predizer bons resultados para um material como a zircônia, que apresenta elevado módulo elástico associado à resistência flexural superior às demais cerâmicas odontológicas (FRAGA et al., 2017; NISHIOKA et al., 2018). No entanto, a diferença entre módulo de Young da restauração e do substrato tem papel importante na distribuição de tensões ao longo do conjunto restauração-dente (DAL PIVA et al., 2018; YAN; KAIZER; ZHANG, 2018). Nesse caso, materiais com módulo de elasticidade similar à dentina sugerem um bom comportamento mecânico devido a melhor dissipação de tensões ao longo das estruturas dentárias (TRIBST et al., 2018). Por essa razão, uma nova classe de materiais restauradores indiretos tem ganhado notoriedade em relação às opções atualmente disponíveis (COLDEA; SWAIN; THIEL, 2013). Esses novos materiais (comercialmente chamados de cerâmicas híbridas) são constituídos de uma rede cerâmica reforçada por uma rede polimérica, estão disponíveis para uso em consultório através de sistemas CAD/CAM e dispensam a necessidade de queima e cristalização, apresentando a facilidade de processamento e de ajuste final, sendo pontos importantes para tornar o material uma opção clinicamente relevante (BOTTINO et al., 2015). Além disso, o que o torna ainda mais atrativo é a forma como se comporta frente às cargas mastigatórias, absorvendo-as homogeneamente devido à similaridade do seu módulo elástico ($E \cong 30$ GPa) com a dentina ($E \cong 18.6$ GPa) (FACENDA; BORBA; CORAZZA, 2018; TRIBST et al., 2018).

Sendo assim, percebe-se que é necessário considerar não apenas o módulo de elasticidade dos materiais restauradores, como também dos substratos aos quais as restaurações são cimentadas (FACENDA et al., 2019; ZIMMERMANN et al., 2017). Os valores diferem significativamente quando comparamos substratos de dentina e resina composta ($E \cong 18{,}6$GPa e E≅11GPa) e as ligas odontológicas (E≅220GPa) utilizadas para confecção de núcleos metálicos fundidos (DAL PIVA et al., 2018). Quando forças mastigatórias intermitentes são exercidas, a superfície de cimentação da peça cerâmica concentra grande parte das tensões de tração responsáveis pelo início da falha em coroas unitárias (KELLY, J R et al., 1990; QUINN et al., 2005; THOMPSON; ANUSAVICE, 1994). Com isso, é possível questionar se restaurações monolíticas cimentadas sobre substratos de maior módulo elástico (núcleo metálico) desempenham resultados mais longevos por conta de uma menor solicitação flexural. Até o presente, estudos laboratoriais têm avaliado o efeito de diferentes condições de tratamento e cimentação das cerâmicas odontológicas na sua resistência mecânica (MAY et al., 2012; ROJPAIBOOL; LEEVAILOJ, 2017; VENTURINI et al., 2019a; YAN; KAIZER; ZHANG, 2018). Entretanto, utilizam como substrato materiais de módulos elástico semelhantes à dentina e, portanto, desconsideram as diferentes possibilidades clínicas as quais as restaurações indiretas são submetidas (ZIMMERMANN et al., 2017).

Por outro lado, sabe-se que uma boa adesão promove melhor distribuição de tensões através da restauração e, portanto, diversos estudos tem sido conduzidos no sentido de avaliar o efeito de diferentes tratamentos da superfície de cerâmicas no comportamento mecânico das restaurações (AMARAL et al., 2016; CAMPOS et al., 2017; FRAGA et al., 2018; GUILARDI et al., 2019; PROCHNOW et al., 2018). Nesse sentido, não apenas o módulo de elasticidade demarcam a diferença entre os diferentes substratos aos quais as restaurações cerâmicas são habitualmente cimentadas, como também a resistência de união aos cimentos resinosos, muito embora até o presente momento, pouco se saiba sobre a influência da adesão ao substrato no desfecho mecânico em fadiga da restauração (CADORE-RODRIGUES et al., 2021a). Deve-se ainda levar em consideração que, mesmo em substratos de mesmo material, diferentes protocolos e etapas prévias à cimentação adesiva modificam a resistência de união aos agentes de cimentação (COTES et al., 2015; KLOSA et al., 2020). Por exemplo, a superfície do preparo para coroas protéticas geralmente passam por um período de cimentação provisória até que a etapa laboratorial de confecção da restauração definitiva seja concluída e, nessas condições, estudos de adesão relataram que diferentes tratamentos de superfície do núcleo de resina composta podem modificar a adesão entre a resina composta do preparo e o cimento resinoso (COTES et al., 2015; KLOSA et al., 2020).

Frente às questões apresentadas, a presente tese foi composta por artigos decorrentes de três investigações científicas com objetivo de avaliar a influência do substrato e de suas diferentes condições de superfície no comportamento mecânico de cerâmicas odontológicas submetidas a um ensaio de fadiga acelerada. O primeiro artigo, intitulado “***Fatigue resistance***

of simplified CAD-CAM restorations: Foundation material and ceramic thickness effects on the fatigue behavior of partially- and fully-stabilized zirconia", visou avaliar o efeito de três substratos com módulos de elasticidade diferentes (resina epóxi, resina composta e liga metálica) e duas espessuras da restauração (0,7 ou 1,0mm) no comportamento mecânico em fadiga de duas gerações de cerâmicas policristalinas cimentadas adesivamente. O segundo artigo, intitulado "***Influence of the foundation substrate on the fatigue behavior of bonded glass, zirconia polycrystals, and polymer infiltrated ceramic simplified CAD-CAM restorations***", investigou o comportamento mecânico em fadiga de cerâmicas com diferentes composições e microestruturas (feldspática, dissilicato de lítio, infiltrada por polímero e zirconia) cimentadas adesivamente sobre dois substratos com módulos de elasticidade diferentes (resina epóxi e liga metálica). Por fim, o terceiro artigo, intitulado "***Influence of surface treatment of resin composite substrate on the load-bearing capacity under fatigue of lithium disilicate monolithic simplified restorations***", se propôs avaliar a influência de diferentes condições topográficas de um substrato de resina composta no comportamento mecânico em fadiga de restaurações de dissilicato de lítio. Foram propostos diferentes protocolos de remoção do cimento provisório, simulando uma condição clínica de cimentação definitiva da restauração cerâmica sobre um núcleo de reconstrução de resina composta após um período com restauração provisória.

2. ARTIGO 1 – FATIGUE RESISTANCE OF SIMPLIFIED CAD–CAM RESTORATIONS: FOUNDATION MATERIAL AND CERAMIC THICKNESS EFFECTS ON THE FATIGUE BEHAVIOR OF PARTIALLY- AND FULLY-STABILIZED ZIRCONIA

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Fatigue resistance of simplified CAD-CAM restorations: Foundation material and ceramic thickness effects on the fatigue behavior of partially- and fully-stabilized zirconia

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Running tittle: Foundation and ceramic thickness on the fatigue behavior of zirconia restorations

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Fatigue resistance of simplified CAD-CAM restorations: Foundation material and ceramic thickness effects on the fatigue behavior of partially- and fully-stabilized zirconia

ABSTRACT

Objective. To evaluate the fatigue failure load, number of cycles until failure and survival probability of partially (PSZ) and fully-stabilized (FSZ) polycrystalline zirconia disc shaped specimens with different thicknesses adhesively cemented onto foundations with distinct elastic moduli.

Methods. Disc-shaped specimens (n = 15, Ø = 10 mm; thickness = 1.0 and 0.7 mm) of CAD/CAM PSZ and FSZ blocks were adhesively cemented onto discs with different foundations (Ø = 10 mm; thickness = 2.0 mm) made from epoxy resin, composite resin or Ni-Cr metallic alloy. The cemented assemblies were subjected to fatigue testing using a step-stress approach (600-2800 N; step-size of 100 N; 10,000 cycles per step; 20 Hz) and the data was submitted to specific statistical tests ($\alpha = 0.05$). Fractography and finite element (FEA) analyzes were also performed.

Results. PSZ and FSZ presented higher fatigue failure load, number of cycles until failure and survival probabilities when cemented onto metallic alloy. All PSZ specimens survived the fatigue test when cemented onto Ni-Cr alloy (100% probability of survival at 2800 N; 230,000 cycles). Regardless of the foundation type, PSZ had better fatigue behavior than FSZ. For thickness, thinner PSZ restorations underperformed when bonded to softer foundations, while FSZ groups and groups bonded to metallic foundations had no statistical difference.

Significance. The foundation material strongly influences the fatigue performance of PSZ and FSZ restorations, which presented mechanical behavior improvements when bonded to a metallic foundation. PSZ restorations showed better fatigue behavior than FSZ, while the ceramic thickness only influenced PSZ restorations bonded to softer foundations.

Keywords: Dental ceramics, All-ceramics, Monolithic restorations, Full-contour restoration, Fatigue failure, Zirconia, Substrate, Elastic Modulus.

Highlights:

- PSZ restorations showed higher fatigue performance than FSZ.
- A stiffer foundation allowed better fatigue performance of FSZ and PSZ restorations.
- The ceramic thickness only influenced PSZ restorations bonded to softer foundations.

1. Introduction

Monolithic ceramic crowns enable minimally invasive prosthetic preparations since they eliminate metallic infrastructures and become an option in clinical situations lacking occlusal space among antagonist teeth [1]. Glass ceramics have limitations in terms of mechanical strength to withstand the cyclic loads in clinical conditions where ultra-thin occlusal veneers are required [2]. In this sense, polycrystalline ceramics such as zirconia provide satisfactory mechanical strength to endure chewing loads, even with reduced thicknesses, being recommended for this use [3,4].

Although the first generation of zirconia presents excellent mechanical properties, it is esthetically unsatisfactory due to its low translucency, which led to the need for changes in the composition of this material to promote favorable optical results and make their application possible for monolithic crowns [5]. The second generation shows better light transmission because of the reduced number and particle size of aluminum oxides (Al_2O_3) in its composition. However, although this change can make it an available option for posterior monolithic restoration, its translucency could not eliminate the necessity for vitreous ceramic veneer for aesthetic rehabilitations [5,6]. Thus, the third generation subsequently emerged from modifying the crystal structure, where the increase in the proportion of yttrium oxide (Y_2O_3) incorporated cubic phase crystals into the tetragonal structure, which present a larger volume and enables satisfactory translucency for use in monolithic crowns [5–8]. This generation can be classified as fully-stabilized zirconia because it has no phase transformation, meaning no toughening mechanism [5]. Thus, even if it is considered as a material with better optical capabilities, it has the drawback of lower mechanical behavior [5,7].

As ceramic materials are extremely brittle, their mechanical performance under intermittent loading may be affected by restorative foundations upon which they are cemented [9]. The foundation materials (or core build-up material) significantly differ in their modulus of elasticity (E) when comparing dentin and composite resin ($E \sim$18,6GPa and $E \sim$11GPa) to metallic alloy ($E \sim$ 220GPa) [9,10]. These different materials for restorative foundations generate greater or lesser bending in the ceramic as a function of its elastic modulus which modifies the ceramic cracking initiation generated on the inner surface [9,11,12]. Thus, foundations with high modulus of elasticity tend to induce lower flexion of the restorative material and consequently lower tensile stress concentration [10,13]. Thus, it can be considered that smaller occlusal ceramic thicknesses will have less influence on the load until failure [2]. From this standpoint, better understanding of fatigue behavior of zirconia monolithic restorations with different thicknesses cemented onto distinct foundations appears to be clinically relevant, being an important guide for clinical plausibility.

Thus, this study aims to compare the mechanical behavior under intermittent cyclical loading (fatigue failure load, number of cycles until failure and survival probability) of simplified restorations made of partially (PSZ) and fully-stabilized (FSZ) polycrystalline zirconia material with two thicknesses cemented onto foundation materials with distinct elastic moduli (softer or stiffer). The null hypotheses are: (1) distinct foundations will not affect the fatigue mechanical behavior of adhesively cemented zirconia simplified restorations; (2) different ceramic thicknesses will present similar fatigue mechanical capacities when cemented onto the same foundation; and (3) PSZ and FSZ simplified restorations will have similar fatigue behavior.

2. Materials and Methods

This study design included 12 experimental groups (n = 15) considering 3 factors (Figure 1):

i) **Two zirconia material levels**: PSZ – Partially-stabilized zirconia material; and FSZ – Fully-stabilized zirconia material.

ii) **Two ceramic thickness levels**: 0.7 mm and 1.0 mm.

iii) **Three foundation levels**: ER – Epoxy resin; CR – composite resin; and MA – metallic alloy.

Simplified geometry was used for testing in the present study. The test assembly consisted of a simplified occlusal restoration for a posterior tooth represented by a zirconia disc with a final diameter of 10 mm, which was adhesively cemented onto different foundations (epoxy resin, composite resin and metallic alloy) with a thickness of 2 mm. The simplified restoration was made with different zirconia materials: partially-stabilized zirconia (PSZ) and fully-stabilized zirconia (FSZ) and two different thicknesses (0.7mm and 1.0mm). The description of the materials used in this study are presented in Table 1.

2.1. Specimen Preparation

2.1.1 Ceramic discs

CAD/CAM discs of PSZ (Ø = 98.5 mm/thickness = 16 mm; Zenostar T, Wieland Dental, Pforzheim, Germany) and FSZ (Ø = 98.5 mm/thickness = 20 mm; IPS ZirCAD MT Multi, Ivoclar Vivadent, Schaan, Liechtenstein) were sectioned to obtain smaller blocks (PSZ = ±14 × 14 × 16 mm; FSZ = ±14 × 14 × 20 mm) which were then shaped into cylinders (Ø =

12 mm) by grinding using a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) with silica carbide (SiC) papers (#600 and #1200-grit; 3M, Sumare, Brazil) under water-cooling [14]. Next, the cylinders were sliced into discs of ± 1.4 mm and ± 1.0 mm in thickness by a diamond disc (Buehler Isomet Wafering blade series 15LC no. 11-4276 with 6 inches' diameter [152 mm] and 0.020 in [0.5 mm] in thickness, being a medium coarse diamond grinding tool) coupled to a cutting machine (Isomet 1000, Buehler). The discs were then manually polished with silica carbide (SiC) papers (#400, #600 and #1200-grit) to remove any irregularities introduced by cutting until ± 1.2 mm and 0.9 mm in thickness [14]. Next, they were sintered according to the manufacturer's instructions for each respective material in a specific furnace (Zyrcomat 6000 MS, Vita Zahnfabrik, Bad Sackingen, Germany). After sintering, all discs were inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper, Takatsu-ku, Kawasaki, Kanagawa, Japan) to guarantee that the final dimensions were in accordance with the study design (10 mm x 1.0 mm and 10 mm x 0.7 mm, in diameter and thickness, respectively). Any specimens presenting discrepancies in dimensions above the recommended deviation (± 0.1 mm) were replaced [14].

The PSZ and FSZ zirconia discs with two thicknesses (0.7 and 1.0 mm) were randomly allocated (www.randomizer.org) according to the foundation material (n=15; Figure 1).

2.1.2 Foundation discs (epoxy resin, composite resin and metallic alloy)

Discs of three different materials were obtained (epoxy resin, composite resin and metallic allow) with final dimensions of 10 mm diameter and 2 mm thickness, as described below. After production, all surfaces of the foundation discs were polished with silica carbide (SiC) papers (#1200-grit; 3M, Sumare, Brazil).

2.1.2.1 Epoxy resin

Epoxy resin plates (Epoxy Plate 150 Plate 150 × 350 x 2.0 mm; Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany) were shaped into 60 discs (thickness= 2.0 mm) using a cylindrical diamond drill (internal diameter = 10 mm; Diamant Boart, Brussels, Belgium) coupled to a bench drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) under refrigeration.

2.1.2.2 Composite resin

A total of 60 composite resin discs (Tetric N-Ceram, Ivoclar Vivadent) were prepared using a metallic template (Ø = 10 mm; thickness = 2.0 mm). Each increment (± 2 mm) was inserted using a #1 spatula (Golgran, São Caetano do Sul, Brazil) and light-activated for 20 s

(1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia). The last layer was covered with a polyester strip and compressed using a glass slide to obtain a flat surface. The sample was light-activated for 20 s through the glass plate with the polyester strip in contact with the composite resin surface.

2.1.2.3 Metallic alloy (Ni-Cr)

A chemically cured acrylic resin (VIPI, Pirassununga, Brazil) was used to prepare 60 discs using a metallic matrix (Ø = 10 mm; thickness = 2.0 mm). These acrylic discs were then used as standards for casting the Ni-Cr alloy discs (4all, Ivoclar Vivadent). After the casting procedure, all metallic alloy discs were inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper, Takatsu-ku, Kawasaki, Kanagawa, Japan) to guarantee the final dimensions (Ø = 10 mm; thickness = 2.0 mm). Any discs presenting discrepancies in dimensions above 0.1 mm were discarded.

2.2 Surface treatments and bonding procedure

Prior to cementation procedures, all specimens (ceramics and foundations) were cleaned in an ultrasonic bath (Vitasonic, Vita Zanhfabrik; Bad Sackingen, Germany) with distilled water (5 min). The surface of the ceramic and foundations was treated as described in Table 2.

After the treatments, Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the treated surfaces of all foundation discs for 30 s and air-dried until a thin layer was obtained. A primer containing multiple bond promoters (Monobond Plus, Ivoclar Vivadent) was concomitantly scrubbed on the bonding surfaces of all zirconia discs for 15 s and then kept reacting for 45 s (1 min for the entire procedure).

The resin cement (Multilink Automix, Ivoclar Vivadent) was then manipulated according to the manufacturer's instructions and applied on the silanized surface of the zirconia discs. These discs were placed over the foundation discs and bonded under a constant load of 2.5 N with an adapted surveyor (B2, BioArt, São Carlos, Brazil). The cement excess was subsequently removed with a microbrush and light-activation was performed for 20 s on each surface (0°, 90°, 180°, 270°, and occlusal surface) (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia).

2.3 Fatigue tests (Step-stress approach)

The cemented assemblies (n = 15) were submitted to a fatigue test in an electric testing machine (Instron ElectroPuls E3000; Instron Corporation, Norwood, USA) using the step-stress methodology [15]. Cyclic loads were applied by a hemispherical stainless-steel piston 40 mm in diameter with the specimen immersed in water (Figure 2). An adhesive tape (110 µm) was placed on the occlusal ceramic disc before testing between the piston and the ceramic surface to reduce contact stress concentration by improving the contact between the piston and specimen [13, 16].

Cyclic loading was applied at a frequency of 20 Hz for 10,000 cycles at each load step, starting with an initial load of 600 N and followed by progressive load levels of 100 N (700 N, 800 N, 900 N, up to 2800 N) until failure detection. At the end of each load step, the specimen was visually inspected for the presence of failure (cracks and/or fracture) by light oblique transillumination [17]. If failure was detected, the test was ended and the collected data were recorded for statistical analysis (FFL – fatigue failure load and CFF – cycles for failure); however, if the specimen survived, the load increment was increased and the test proceeded until observing failure.

2.4 Fractographic analysis

After the cyclic fatigue tests, all specimens were analyzed for contact damage and to identify the crack direction by visual inspection under oblique light transmission. The failed specimens (i.e. radial cracks) were cut into two halves (Isomet 1000, Buehler) perpendicularly to the crack direction, and analyzed under stereomicroscope (Discovery V20, CarlZeiss, Gottingen, Germany). Random specimens were also analyzed under scanning electron microscope (Vega3, Tescan, Czech Republic) to determine fractographic characteristics.

2.5 Finite Element Analysis (FEA)

FEA was used to evaluate the stress distribution in the test set-up. To do so, models consisting of two generations of zirconia discs, the cut steel ball layer and the foundations (epoxy resin, composite resin and metallic alloy) were made.

Two tridimensional models (0.7 and 1 mm) were created using a software program (Rhinoceros version 5.0 SR14, McNeel North America, Seattle,WA, USA), and the analysis was performed using ANSYS software CAE (ANSYS 19.3, ANSYS Inc., Houston, TX, USA). A static structural analysis was applied according to the fatigue experimental set-up. All materials were considered isotropic, linear and homogeneous. Young's modulus (GPa) and Poisson's ratios for dental zirconia (E = 200 GPa; v = 0.31), epoxy resin (E = 14.9 GPa; v = 0.31), composite resin (E = 11 GPa, v = 0.28), metallic alloy (E = 220 GPa; v = 0.3), resin cement (E = 7.5 GPa, v = 0.3) and stainless steel ring/sphere (E =190 GPa; v = 0.27) were obtained from previous studies [18,19]. The models were composed of 11,489 hexahedron solid elements with 53,775 nodes for the 0.7 mm groups and 12,062 hexahedron solid elements with 56,129 nodes for the 1.0 mm groups. The connection was considered bonded for all materials except frictional (0.12) for piston/restoration and rough to alloy/resin cement. The models were loaded (100 N) at the top of the piston and the bottom surface of the base was constrained.

After the coherence and mesh convergence test, maximum principal stress (MPS) was used as failure criteria to compare the groups.

2.6 Data analysis

A statistical software program (Statistix, Tallahasse, FL, USA) was used with a significance level of 0.05. Three-way ANOVA was performed to identify the interaction between the independent (ceramic, foundation, and thickness) and dependent (FFL – fatigue failure load and CFF – cycles for failure) variables, followed by the Tukey post-hoc test for the interaction of all variables (ceramics × foundations × thickness). A survival analysis was also performed in the SPSS Software program (IBM, Armonk, NY, USA) using the Kaplan Meier and Mantel-Cox (Log Rank) survival tests and the survival probability was tabulated for each step of the test.

Fractographic and Finite Element analyses were qualitatively analyzed.

3. Results

According to the three-way ANOVA results for FFL and CFF data, the ceramics ($P<.001$, $F= 250.124$ and $F= 212.472$), foundations ($P<.001$, $F= 493.670$ and $F= 461.670$) and thickness ($P<.001$, $F= 19.354$ and $F= 12.930$) had significant effects. In addition, a statistically significant interaction was found between ceramics × foundations ($P<.001$, $F= 25.194$ and $F= 23.175$), ceramics × thickness ($P<.001$, $F= 19.354$ and $F= 13.535$) and foundation × thickness ($P<.001$, $F= 16.86$ and $F= 12.879$). Despite this, no interaction between the three factors (ceramics × foundations × thickness) were found ($P>.05$, $F= 0.466$ and $F= 1.133$) (Table 3).

Regarding the different zirconia generations, the PSZ (partially stabilized by yttrium oxide) showed higher fatigue performance than FSZ (fully stabilized) when the same foundations and thicknesses were evaluated. When analyzing the different foundations, both PSZ and FSZ bonded to metallic alloy obtained the highest load-bearing capacities, regardless of the ceramic thickness. There was no statistical difference when comparing the composite resin vs. the dentin analogue for the same type of zirconia material.

Regarding different restoration thicknesses, only the 0.7 mm PSZ groups cemented onto a softer foundation (dentin analogue or composite resin) presented statistically lower FFL and CFF values than 1.0 mm PSZ groups. The ceramic thickness showed no statistically significant influence for FSZ restorations. Table 4 and Fig. 3 summarizes the survival rates of the restorations for loading steps and number of cycles until failure.

The failure analysis under a light microscope showed that radial cracks starting from the cemented surface took place in all failed specimens, and no Hertzian cone cracks were detected. Representative SEM micrographs of the fracture surfaces are presented in Fig. 4. No specimen failed in the FSZ_MA groups up to the maximum load of 2800 N applied in the fatigue test.

The FEA Analysis (Figure 5) demonstrated that maximum peak stress (MPS) occurred on the inner surface of the ceramic discs. Simplified restorations with less thickness (0.7 mm) concentrated higher stress values for an occlusal load of 100 N in all foundations. The groups cemented on metallic alloy under the same applied load presented the lowest MPS values for both 0.7 mm and 1.0 mm of thickness (648.74 MPa and 341.91 MPa, respectively).

4. Discussion

The present study demonstrated that partially (PSZ) and fully-stabilized (FSZ) polycrystalline zirconia simplified restorations bonded onto stiffer foundations presented enhanced mechanical behavior under intermittent cyclical loading (fatigue failure load, number of cycles until failure and survival probability) compared to the same restorations cemented onto a dentin analogue or composite resin foundation. Therefore, the first null hypothesis was rejected. It was also observed that only the PSZ restorations cemented on softer foundations (dentin analogue or composite resin) had worse fatigue behavior when comparing the 1.0- and 0.7-mm thicknesses, thus rejecting the second hypothesis. Moreover, PSZ and FSZ simplified restorations have different behavior under intermittent cyclical loading, thereby rejecting the third hypothesis.

The failure of ceramic monolithic crowns is not only dependent on the nominal resistance of the material used, but also on the elastic modulus mismatch between the ceramic and prosthetic foundation [20]. Actually, prosthetic preparations may be composed by different materials such as composite resin, metallic alloys and dentin reminiscent, presenting a significant difference in elasticity among these options (about 11 to 18 GPa for softer foundations such as composite resin and dentin; 220 GPa for stiff metallic alloys) [9,10,18,21]. The ceramic material undergoes flexion under intermittent masticatory loads, and the failure is started at the cementation interface (ceramic, resin cement and foundation) due to tensile stress concentration in this region [13,18]. However, load to failure of these materials is increased when adhesively cemented onto foundations with a higher modulus of elasticity, which prevents the ceramic from flexing [9,10]. The present study corroborates with this premise, since better fatigue performance of ceramic restorations under cyclic loads was observed when they were cemented onto a high elastic modulus metallic foundation (Table 3).

Recent studies have shown that second generation zirconia (PSZ) presented higher load-bearing capacity under cyclic loading than third generation materials (FSZ) [22,23], since fully-stabilized zirconia does not present phase transformation (t-m), and consequently no toughening mechanism occurs to prevent crack propagation. In addition, third generation zirconia has a larger grain size and yttria content in its composition and shows more intergranular spaces when compared to the second generation [24], thereby leading to a similar or less fracture load than a lithium disilicate glass ceramic when cemented onto epoxy resin foundations, depending on the ceramic thickness [25]. On the other hand, FSZ has better translucency than PSZ as the main advantage [5]. From the mechanical behavior point of view, we in fact found that the third generation zirconia had a lower fatigue performance compared to the second generation, regardless of the ceramic thickness or foundation material (even with a stiffer foundation).

Regarding the two thicknesses evaluated in our study, the absence of phase transformation, the lower flexural strength of the material and the stress distribution throughout the restoration may explain the reason why no difference was found for the FSZ groups. Due to the lower tensile strength of FSZ compared to PSZ [5,22], the tensile stress peak on the inner surface of both thicknesses may have exceeded the nominal material resistance under similar load applications [26]. On the other hand, the load needed to generate sufficient tensile stress peek to initiate crack formation in 1.0 mm FSZ specimens was significantly greater than that of 0.7 mm. Nevertheless, taking into consideration the fatigue load to failure found by us and the estimated load in clinical service (around 541N to 590N) [27,28], we might hypothesize that all of our tested combinations can perform well clinically, although with different results in relation to longevity, as demonstrated in the analysis of survival probabilities (Table 4).

The finite element analysis (Figure 5) verifies that the Maximum Principal Stress occurs on the restoration inner surface. The same tensile stress peak on the inner surface is observed in comparing the two types of zirconia evaluated for any of the thickness or foundation variables, since only Young's modulus (200 GPa) of the evaluated ceramics was considered in this analysis. However, it is known that the intrinsic characteristics of each material must be evaluated to predict mechanical behavior [26]. This means that specific characteristics of each material, such as grain size, toughening mechanism and the previously mentioned intergranular spaces, imply a difference in mechanical behavior in the fatigue test [22–24].

Representative images of scanning electron microscopy showed failures mainly occurring in the cementation area, such as the aforementioned clinical characteristic [13]. However, the cracks also proceeded to the foundation in composite resin groups, and none of the evaluated zirconia systems was able to protect the integrity of the foundation [8]. This is understandable, since the fatigue failure loads of zirconia restorations are much higher than the loads that composite resins can withstand, thus when a crack initiates in zirconia it did not stop and advances into the composite foundation [29,30]. On the other hand, none of the evaluated zirconia under the different explored thicknesses showed different results (FFL, CFF, or survival rates) between the epoxy resin and the composite resin foundations. An explanation for the same fatigue behavior of a restoration cemented on dentin analogue and composite resin may be the similar modulus of elasticity between these two materials, which further supports our findings on this aspect.

Finally, we have to state that our study has some limitations such as using the fatigue evaluation of simplified restorations (disc-disc set-up), the absence of long-term aging for bond degradation, only axial cyclic loading application without sliding movement and the load limit set by the equipment used. From the clinical stand-point, the risk of a grayish effect when using a zirconia restoration on the metallic foundation must be highlighted, therefore caution must be taken with our results to prevent misinterpretations.

As a final consideration, our findings mainly contribute to clarify an important clinical issue: the elastic modulus of the foundation material (or core material) may strongly influence the mechanical behavior of partially (second generation) or fully-stabilized (third generation) polycrystalline zirconia simplified restorations when under intermittent cyclical loading.

5. Conclusions

- The stiffer foundation material improved the fatigue performance of adhesively cemented partially and fully-stabilized zirconia restorations.
- The ceramic thickness only influenced the fatigue behavior of PSZ restorations bonded to softer foundations, while it had no effect for FSZ restorations.
- PSZ restorations presented higher fatigue performance than FSZ restorations, regardless of the ceramic thickness or foundation material.

REFERENCES

[1] Nakamura K, Harada A, Inagaki R, Kanno T, Niwano Y, Milleding P, et al. Fracture resistance of monolithic zirconia molar crowns with reduced thickness. Acta Odontol Scand 2015;73:602–8. https://doi.org/10.3109/00016357.2015.1007479.

[2] Zimmermann M, Egli G, Zaruba M, Mehl A. Influence of material thickness on fractural strength of CAD/CAM fabricated ceramic crowns. Dent Mater J 2017;36:778–83. https://doi.org/10.4012/dmj.2016-296.

[3] Nordahl N, Vult von Steyern P, Larsson C. Fracture strength of ceramic monolithic crown systems of different thickness. J Oral Sci 2015;57:255–61. https://doi.org/10.2334/josnusd.57.255.

[4] Sorrentino R, Triulzio C, Tricarico MG, Bonadeo G, Gherlone EF, Ferrari M. In vitro analysis of the fracture resistance of CAD-CAM monolithic zirconia molar crowns with different occlusal thickness. J Mech Behav Biomed Mater 2016;61:328–33. https://doi.org/10.1016/j.jmbbm.2016.04.014.

[5] Stawarczyk B, Keul C, Eichberger M, Figge D, Edelhoff D, Lümkemann N. Three generations of zirconia: From veneered to monolithic. Part I. Quintessence Int 2017;48:369–80. https://doi.org/10.3290/j.qi.a38057.

[6] Zhang Y, Lawn BR. Novel Zirconia Materials in Dentistry. J Dent Res 2018;97:140–7. https://doi.org/10.1177/0022034517737483.

[7] Inokoshi M, Shimizu H, Nozaki K, Takagaki T, Yoshihara K, Nagaoka N, et al. Crystallographic and morphological analysis of sandblasted highly translucent dental zirconia. Dent Mater 2018;34:508–18. https://doi.org/10.1016/j.dental.2017.12.008.

[8] Zhang Y, Mai Z, Barani A, Bush M, Lawn B. Fracture-resistant monolithic dental crowns. Dent Mater 2016;32:442–9. https://doi.org/10.1016/j.dental.2015.12.010.

[9] Pereira GKR, Graunke P, Maroli A, Zucuni CP, Prochnow C, Valandro LF, et al. Lithium disilicate glass-ceramic vs translucent zirconia polycrystals bonded to distinct substrates: Fatigue failure load, number of cycles for failure, survival rates, and stress distribution. J Mech Behav Biomed Mater 2019;91:122–30. https://doi.org/10.1016/j.jmbbm.2018.12.010.

[10] Facenda JC, Borba M, Benetti P, Della Bona A, Corazza PH. Effect of supporting substrate on the failure behavior of a polymer-infiltrated ceramic network material. J Prosthet Dent 2019;121:929–34. https://doi.org/10.1016/j.prosdent.2018.08.008.

[11] Campos F, Valandro LF, Feitosa SA, Kleverlaan CJ, Feilzer AJ, De Jager N, et al. Adhesive cementation promotes higher fatigue resistance to zirconia crowns. Oper Dent 2017;42:215–24. https://doi.org/10.2341/16-002-L.

[12] Monteiro JB, Oliani MG, Guilardi LF, Prochnow C, Rocha Pereira GK, Bottino MA, et al. Fatigue failure load of zirconia-reinforced lithium silicate glass ceramic cemented to a dentin analogue: Effect of etching time and hydrofluoric acid concentration. J Mech Behav Biomed Mater 2018;77:375–82. https://doi.org/10.1016/j.jmbbm.2017.09.028.

[13] Kelly JR. Clinically relevant approach to failure testing of all-ceramic restorations. J Prosthet Dent 1999;81:652–61. https://doi.org/10.1016/S0022-3913(99)70103-4.

[14] Pereira GKR, Amaral M, Simoneti R, Rocha GC, Cesar PF, Valandro LF. Effect of grinding with diamond-disc and -bur on the mechanical behavior of a Y-TZP ceramic. J Mech Behav Biomed Mater 2014;37:133–40. https://doi.org/10.1016/j.jmbbm.2014.05.010.

[15] Kelly JR, Cesar PF, Scherrer SS, Della Bona A, van Noort R, Tholey M, et al. ADM guidance-ceramics: Fatigue principles and testing. Dent Mater 2017;33:1192–204.

https://doi.org/10.1016/j.dental.2017.09.006.

[16] Gressler L, Kelly JR, Antonio M, Hill T. Influence of the resin cement thickness on the fatigue failure loads of CAD / CAM feldspathic crowns. Dent Mater 2015:2–7. https://doi.org/10.1016/j.dental.2015.04.019.

[17] Dibner AC, Kelly JR. Fatigue strength of bilayered ceramics under cyclic loading as a function of core veneer thickness ratios. J Prosthet Dent 2016;115:335–40. https://doi.org/10.1016/j.prosdent.2015.09.017.

[18] Dal Piva AM de O, Tribst JPM, Borges ALS, Souza RO de A e., Bottino MA. CAD-FEA modeling and analysis of different full crown monolithic restorations. Dent Mater 2018;34:1342–50. https://doi.org/10.1016/j.dental.2018.06.024.

[19] Miranda JS, de Carvalho RLA, de Carvalho RF, Borges ALS, Bottino MA, Özcan M, et al. Effect of different loading pistons on stress distribution of a CAD/CAM silica-based ceramic: CAD-FEA modeling and fatigue survival analysis. J Mech Behav Biomed Mater 2019;94:207–12. https://doi.org/10.1016/j.jmbbm.2019.03.011.

[20] Yan J, Kaizer MR, Zhang Y. Load-bearing capacity of lithium disilicate and ultra-translucent zirconias. J Mech Behav Biomed Mater 2018;88:170–5. https://doi.org/10.1016/j.jmbbm.2018.08.023.

[21] Creugers N, Mentink A, Fokkinga W, Kreulen C. 5-Year Follow-Up of A Prospective Clinical Study on Various Types of Core Restorations. Int J Prosthodont 2005;18:34–9. https://doi.org/10.1016/j.prosdent.2005.05.013.

[22] Pereira GKR, Guilardi LF, Dapieve KS, Kleverlaan CJ, Rippe MP, Valandro LF. Mechanical reliability, fatigue strength and survival analysis of new polycrystalline translucent zirconia ceramics for monolithic restorations. J Mech Behav Biomed Mater 2018;85:57–65. https://doi.org/10.1016/j.jmbbm.2018.05.029.

[23] Zucuni CP, Pereira GKR, Valandro LF. Grinding, polishing and glazing of the occlusal surface do not affect the load-bearing capacity under fatigue and survival rates of bonded monolithic fully-stabilized zirconia simplified restorations. J Mech Behav Biomed Mater 2020;103:103528. https://doi.org/10.1016/j.jmbbm.2019.103528.

[24] Sulaiman TA, Abdulmajeed AA, Shahramian K, Lassila L. Effect of different treatments on the flexural strength of fully versus partially stabilized monolithic zirconia. J Prosthet Dent 2017;118:216–20. https://doi.org/10.1016/j.prosdent.2016.10.031.

[25] Longhini D, Rocha COM, De Oliveira LT, Olenscki NG, Bonfante EA, Adabo GL. Mechanical behavior of ceramic monolithic systems with different thicknesses. Oper Dent 2019;44:E244–53. https://doi.org/10.2341/18-083-L.

[26] Tribst JPM, Dal Piva AM de O, Penteado MM, Borges ALS, Bottino MA. Influence of ceramic material, thickness of restoration and cement layer on stress distribution of occlusal veneers. Braz Oral Res 2018;32:e118. https://doi.org/10.1590/1807-3107bor-2018.vol32.0118.

[27] Calderon PDS, Kogawa EM, Lauris JRP, Conti PCR. The influence of gender and bruxism on the human maximum bite force. J Appl Oral Sci 2006;14:448–53. https://doi.org/10.1590/S1678-77572006000600011.

[28] Chong MX, Khoo CD, Goh KH, Rahman F, Shoji Y. Effect of age on bite force. J Oral Sci 2016;58:361–3. https://doi.org/10.2334/josnusd.15-0675.

[29] Calheiros FC, Pfeifer CSC, Brandão LL, Agra CM, Ballester RY. Flexural properties of resin composites:Influence of specimen dimensions and storage conditions. Dent Mater J 2013;32:228–32. https://doi.org/10.4012/dmj.2012-271.

[30] Jalkh EBB, Machado CM, Giannini M, Beltramini I, De Toledo Piza MM, Coelho PG, et al. Effect of thermocycling on biaxial flexural strength of CAD/CAM, bulk fill, and conventional resin composite materials. Oper Dent 2019;44:E254–62. https://doi.org/10.2341/18-146-L.

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
Partially Stabilized Zirconia	Zenostar T, Wieland Dental, Ivoclar Vivadent (U20833)	ZrO2 > 99.0 wt%; Y_2O_3 > 4.5 % – ≤ 6.0 wt%. HfO_2 ≤ wt5.0%; other oxides ≤ 1.0 wt%
Fully Stabilized Zirconia	IPS ZirCAD MT Multi, Ivoclar Vivadent (W82689)	ZrO_2 86 – 93.5 wt%, Y_2O_3 > 6.5 % – ≤ 8.0 wt%, HfO_2 ≤ 5.0 wt%, Al_2O_3 ≤ 1.0 wt%, other oxides ≤ 1.0 wt%
10% Hydrofluoric acid	Condac Porcelana, FGM (060519)	10% hydrofluoric acid, water, thickener, surfactant and coloring
Silane coupling agent	Monobond Plus, Ivoclar Vivadent (W90329)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate
Primer	Multilink Primer (A and B), Ivoclar Vivadent (W88902/W4494)	Primer A: water, initiators; primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabilizer
Dual cure resin cement	Multilink Automix, Ivoclar Vivadent (W07285)	Dimethacrylates, HEMA, barium glass filler, Ba-Al-Fluoro-Silicate glass, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments
Dentin analogue material	Epoxy Plate 150 Plate 150 × 350 x 2.0 mm; Carbotec GmbH & Co. KG (229861)	Continuous filament woven fiberglass bonded with epoxy resin
Metallic alloy	4all, Ivoclar Vivadent (T32718)	Ni 61.4%, Cr 25.7%, Mo 11.0%, Si 1.5%, Mn <1.0%, Al <1.0%, C <1.0%
Composite Resin	Tetric N-Ceram, Ivoclar Vivadent (U36522)	Dimethacrylates, fillers (barium glass, ytterbium trifluoride, mixed oxide and copolymers), additives, catalysts, stabilizers and pigments. Particle size of inorganic fillers is between 40 nm and 3000 nm.
Aluminum oxide	Metallic treatment: Bio-Art Equipamentos Odontológicos Ltda. (58221) / Zirconia treatment: Polidental Indústria e Comércio (44493)	Aluminum Oxide (120 µm/45 µm)

Table 2. Surface treatments for ceramic materials and foundations

Material	Surface treatment procedures
Zirconia	Air-abrasion with aluminum oxide particles (Al_2O_3; 45 µm particles size, Polidental) for 10 s, at 10 mm of distance and 2.8 bars of pressure, followed by ultrasonic cleaning (distilled water for 5 min).
Epoxy Resin	Etching with 10% hydrofluoric acid (Condac porcelain, FGM) for 60 s, rinsing for 30 s and ultrasonic cleaning (distilled water for 5 min).
Metallic Alloy	Air-abrasion with aluminum oxide particles (Al_2O_3; 120 µm particles size, Bio-Art) for 10 s, at 10mm distance and 5 bars of pressure, followed by ultrasonic cleaning (distilled water for 5 min).
Composite Resin	Ultrasonic cleaning (distilled water for 5 min).

Table 3. Results for fatigue failure load in Newtons (FFL) and number of cycles until failure (CFF).

Groups' Codes	Dental zirconia	Ceramic Thickness	Substrate	FFL (Mean – 95%CI)	CFF (Mean – 95%CI)
PSZ_0.7ER	**Partially-stabilized polycrystalline zirconia (PSZ)**	**0.7 mm**	**Epoxy Resin**	1567 (1373 - 1760)^{E}	106,667 (87,294 – 126,040)^{DE}
PSZ_1.0ER		**1.0 mm**		2100 (1978 - 2222)^{CD}	160,000 (147,795 – 172,205)^{C}
PSZ_0.7CR		**0.7 mm**	**Composite Resin**	1380 (1247 - 1513)^{EF}	88,000 (747,29 – 101271)^{EF}
PSZ_1.0CR		**1.0 mm**		1900 (1709 - 2091)^{D}	126,065 (100,388 – 151,741)^{D}
PSZ_0.7MA		**0.7 mm**	**Metallic Alloy**	*2800^{A}	*230,000^{A}
PSZ_1.0MA		**1.0 mm**		*2800^{A}	*230,000^{A}
FSZ_0.7ER	**Fully-stabilized polycrystalline zirconia (FSZ)**	**0.7 mm**	**Epoxy Resin**	827 (706 – 948)^{G}	32,667 (20,558 – 44,775)^{G}
FSZ_1.0ER		**1.0 mm**		900 (751 – 1049)^{G}	39,349 (24,917 – 53,781)^{G}
FSZ_0.7CR		**0.7 mm**	**Composite Resin**	880 (759 – 1001)^{G}	37,753 (25,945 – 49,562)^{G}
FSZ_1.0CR		**1.0 mm**		1107 (928 – 1285)^{FG}	60,025 (42,408 – 77,642)^{FG}
FSZ_0.7MA		**0.7 mm**	**Metallic Alloy**	2673 (2532 – 2814)^{AB}	217,333 (203,220 – 231,447)^{AB}
FSZ_1.0MA		**1.0 mm**		2373 (2141 – 2606)^{BC}	187,333 (164,076 – 210,591)^{BC}

Different letters indicate statistically significant differences ($P < .05$) depicted by three-way ANOVA and Tukey post-hoc test.
*Statistical analysis was not possible, because all samples survived until the final step (2800 N / 230,000 cycles).

Table 4. Survival rates – probability of specimens to exceed the respective load to failure (FFL) and number of cycles until failure (CFF) steps and its respective standard error values.

Groups	Steps of load to failure and steps of number of cycles until failure																						
	600N 10 x 10^3	**700N 20 x 10^3**	**800N 30 x 10^3**	**900N 40 x 10^3**	**1000 N 50 x 10^3**	**1100 N 60 x 10^3**	**1200 N 70 x 10^3**	**1300 N 80 x 10^3**	**1400 N 90 x 10^3**	**1500 N 100 x 10^3**	**1600 N 110 x 10^3**	**1700 N 120 x 10^3**	**1800 N 130 x 10^3**	**1900 N 140 x 10^3**	**2000 N 150 x 10^3**	**2100 N 160 x 10^3**	**2200 N 170 x 10^3**	**2300 N 180 x 10^3**	**2400 N 190 x 10^3**	**2500 N 200 x 10^3**	**2600 N 210 x 10^3**	**2700 N 220 x 10^3**	**2800 N 230 x 10^3**
PSZ_0.7 ER	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.87 (0.09)	0.68 (0.12)	0.68 (0.12)	0.68 (0.12)	0.40 (0.13)	0.40 (0.13)	0.33 (0.12)	0.0	-	-	-	-	-	-	-	-	-
PSZ_1.0 ER	1	1	1	1	1	1	1	1	1	1	1	1	0.87 (0.09)	0.87 (0.09)	0.87 (0.09)	0.53 (0.13)	0.20 (0.10)	0.13 (0.09)	0.07 (0.06)	0.0	-	-	-
PSZ_0.7 CR	1	1	1	1	0.87 (0.9)	0.73 (0.11)	0.73 (0.11)	0.60 (0.13)	0.47 (0.13)	0.27 (0.11)	0.13 (0.09)	0.0	-	-	-	-	-	-	-	-	-	-	-
PSZ_1.0 CR	1	1	1	1	1	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.87 (0.09)	0.73 (0.11)	0.67 (0.12)	0.60 (0.13)	0.40 (0.13)	0.40 (0.13)	0.20 (0.10)	0.20 (0.10)	0.13 (0.09)	0.0	-	-	-	-
PSZ_0.7 MA	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
PSZ_1.0 MA	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
FSZ_0.7 ER	0.73 (0.11)	0.53 (0.13)	0.47 (0.13)	0.20 (0.10)	0.13 (0.09)	0.13 (0.09)	0.07 (0.06)	0.0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
FSZ_1.0 ER	0.87 (0.09)	0.60 (0.13)	0.40 (0.13)	0.33 (0.12)	0.33 (0.12)	0.20 (0.10)	0.20 (0.10)	0.07 (0.06)	0.0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
FSZ_0.7 CR	0.80 (0.10)	0.73 (0.11)	0.53 (0.13)	0.40 (0.13)	0.13 (0.09)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
FSZ_1.0 CR	0.93 (0.06)	0.80 (0.10)	0.80 (0.10)	0.73 (0.11)	0.60 (0.13)	0.47 (0.13)	0.33 (0.12)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.0	-	-	-	-	-	-	-	-	-
FSZ_0.7 MA	1	1	1	1	1	1	1	1	1	1	1	1	1	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.80 (0.10)	0.80 (0.10)	0.80 (0.10)	0.80 (0.10)	0.73 (0.11)	0.67 (0.12)
FSZ_1.0 MA	1	1	1	1	1	1	1	1	1	1	0.93 (0.06)	0.93 (0.06)	0.80 (0.10)	0.80 (0.10)	0.73 (0.11)	0.67 (0.12)	0.67 (0.12)	0.60 (0.13)	0.53 (0.13)	0.40 (0.13)	0.33 (0.12)	0.33 (0.12)	0.27 (0.11)

* Kaplan Meier and Mantel-Cox tests.

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

FIGURES

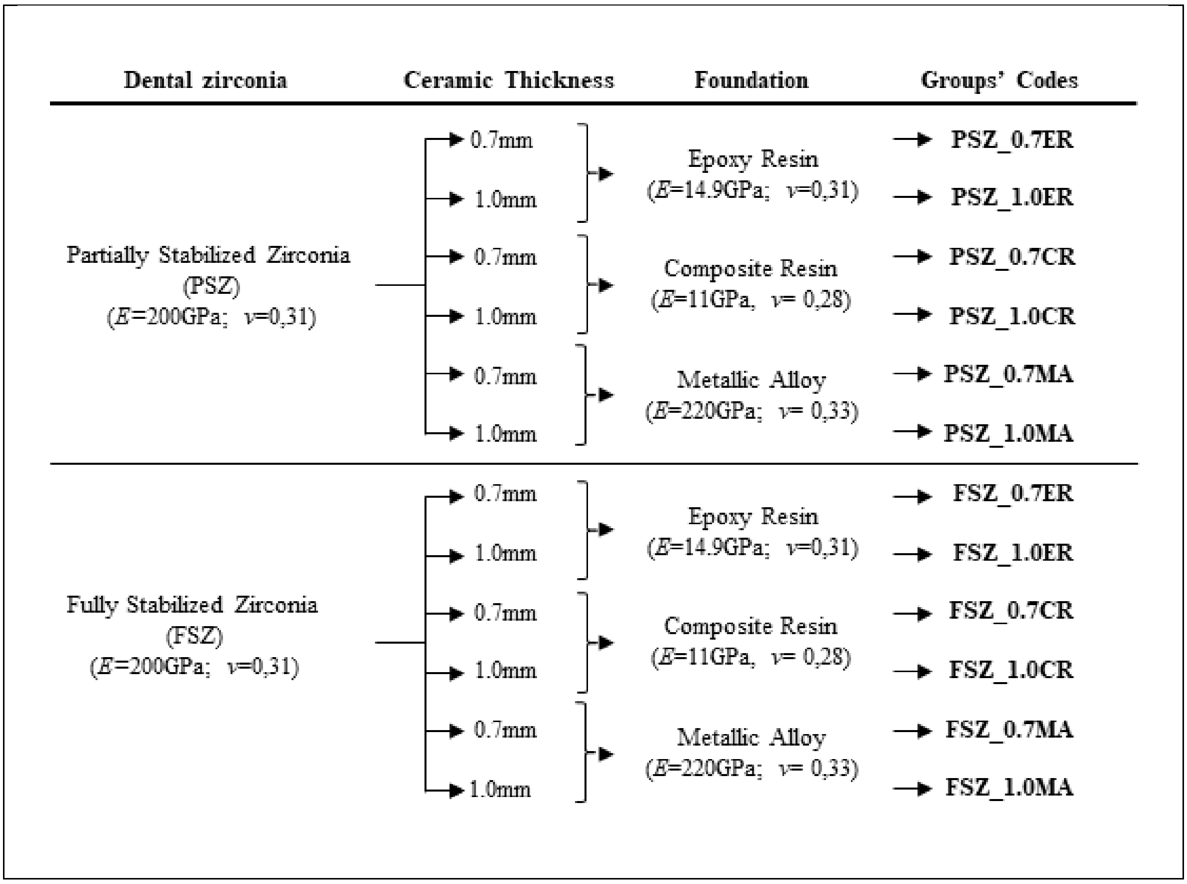


Figure 1. Study design with Young's modulus (*E* in GPa) and Poisson's ratios (*v*) of the zirconia and foundations.

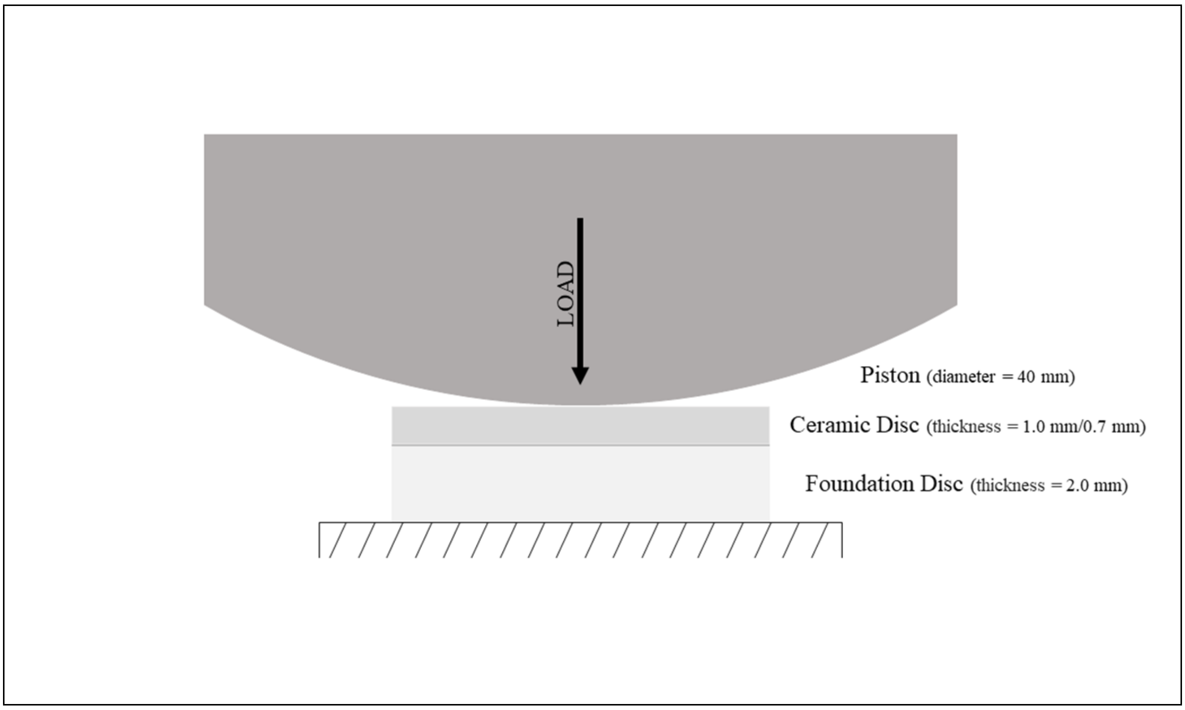


Figure 2. Fatigue test assembly.

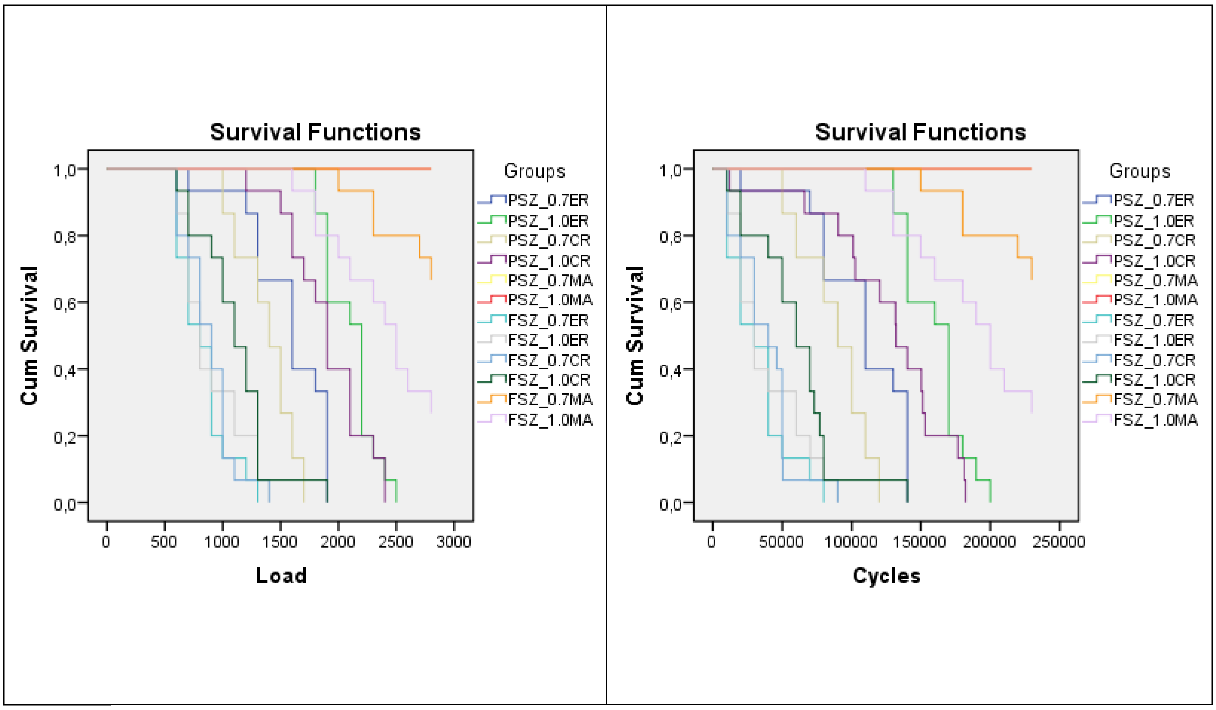


Figure 3. Survival curves according to the steps of fatigue failure load (left) and number of cycles (right) in which each disc failed, obtained by Kaplan–Meier and Log-rank tests. * Censored legends are related to the presence of specimens that survived through all steps of testing on such condition.

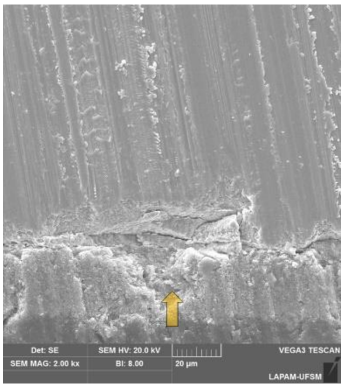
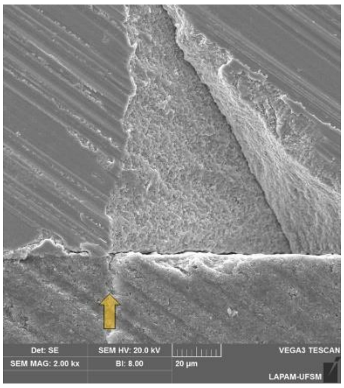
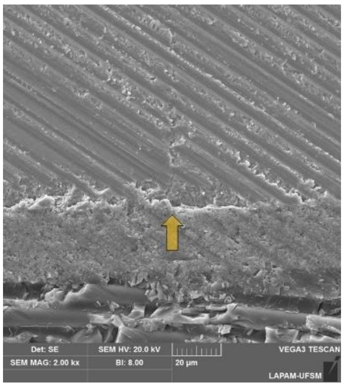
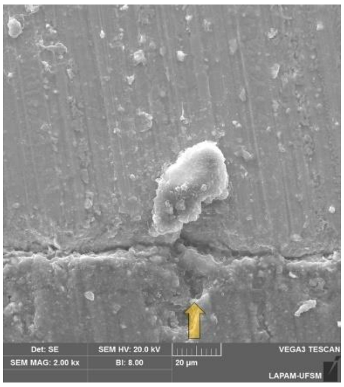
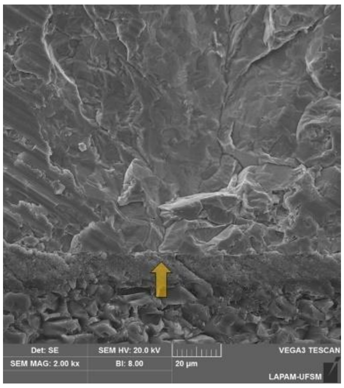
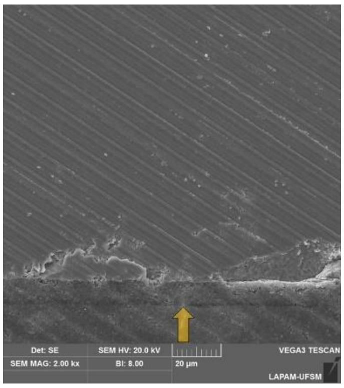
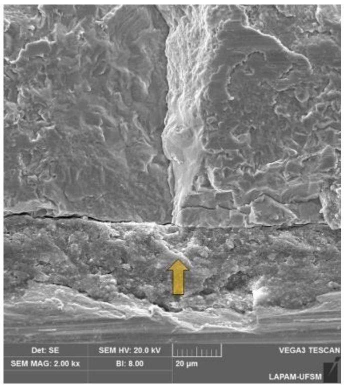
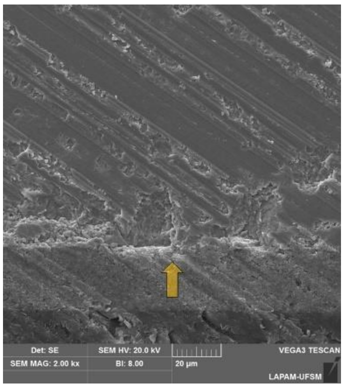
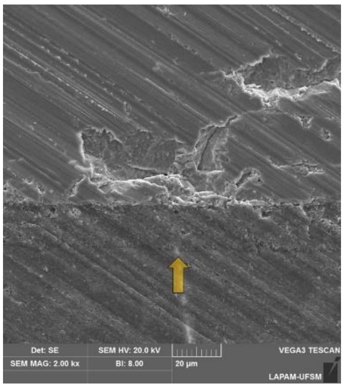
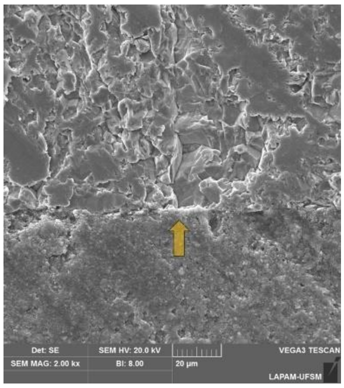
		Epoxy Resin	Composite Resin	Metallic Alloy
		2000x	2000x	2000x
Testing groups	PSZ_ 0.7			*
	PSZ_ 1.0			*
	FSZ_0.7			
	FSZ_1.0			
* No specimen failed up to the maximum load of 2800 N applied in the fatigue test.				

Figure 4. Representative SEM images of failure surfaces from tested ceramic discs, illustrating that all failures originated at cementation surface from defects present at the ceramic surface, pointed by yellow arrows.

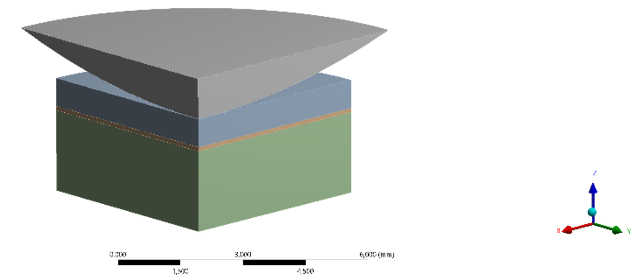
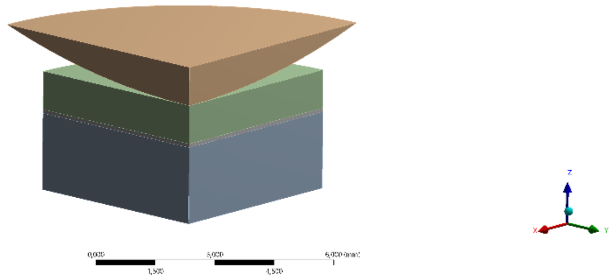
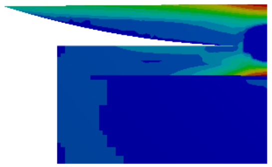
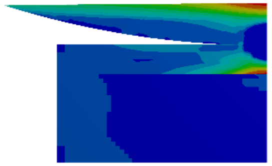
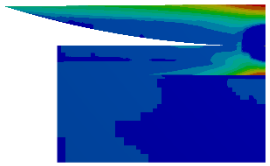
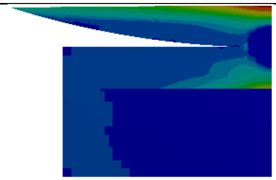
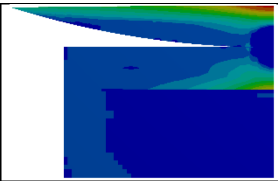
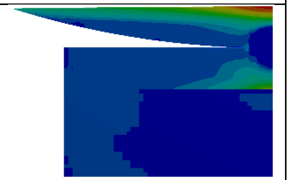
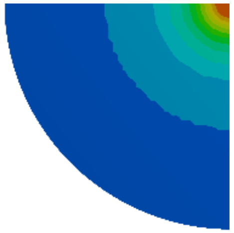
Groups*	**PSZ/FSZ_0.7ER**	**PSZ/FSZ_0.7CR**	**PSZ/FSZ_0.7MA**	**PSZ/FSZ_1.0ER**	**PSZ/FSZ_1.0CR**	**PSZ/FSZ_1.0MA**
Tridimensional models						
Side Section view						
Inner surface view						
MPS	729.7 MPa	761.29 MPa	648.74 MPa	395.88 MPa	415.40 MPa	341.91 MPa
Scale						
*PSZ and FSZ groups show the same results in the FEA analysis due to the same elastic modulus of both materials.						

Figure 5. FEA analysis demonstrates the tensile stress in the inner surface of the restorations (Maximum Principal Stress 0,7– MPS). Under the same applied load, the stress concentration on the inner surface of ceramic discs decreased in thicker ceramic samples and on a stiffer foundation.

3. ARTIGO 2 – INFLUENCE OF THE FOUNDATION SUBSTRATE ON THE FATIGUE BEHAVIOR OF BONDED GLASS, ZIRCONIA POLYCRYSTALS, AND POLYMER INFILTRATED CERAMIC SIMPLIFIED CAD-CAM RESTORATIONS

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Influence of the foundation substrate on the fatigue behavior of bonded glass, zirconia polycrystals, and polymer infiltrated ceramic simplified CAD-CAM restorations

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Short title: Foundation substrate and the fatigue behavior of bonded ceramic restorations.

Influence of the foundation substrate on the fatigue behavior of bonded glass, zirconia polycrystals, and polymer infiltrated ceramic simplified CAD-CAM restorations

Abstract

This study evaluated the influence of distinct substrates on the mechanical fatigue behavior of adhesively cemented simplified restorations made of glass, polycrystalline or polymer infiltrated-ceramics. CAD/CAM ceramic blocks (feldspathic – FEL; lithium disilicate – LD; yttria-stabilized zirconia – YZ; and polymer-infiltrated ceramic network – PICN) were shaped into discs (n= 15, Ø= 10 mm; thickness= 1.0 mm), mimicking a simplified monolithic restoration. After, they were adhesively cemented onto different foundation substrates (epoxy resin – ER; or Ni-Cr metal alloy – MA) of the same shape (Ø= 10 mm; thickness= 2.0 mm). The assemblies were subjected to fatigue testing using a step-stress approach (200N-2800 N; step-size of 200 N; 10,000 cycles per step; 20 Hz) upon the occurrence of a radial crack or fracture. The data was submitted to two-way ANOVA ($\alpha = 0.05$) to analyze differences considering 'ceramic material' and 'type of substrate' as factors. In addition, a survival analysis (Kaplan Meier with Mantel-Cox log-rank post-hoc tests; $\alpha = 0.05$) was conducted to obtain the survival probability during the steps in the fatigue test. Fractographic and finite element (FEA) analyzes were also conducted. The factors 'ceramic material', 'type of substrate' and the interaction between both were verified to be statistically significant ($p < .001$). All evaluated ceramics presented higher fatigue failure load (FFL), cycles for failure (CFF) and survival probabilities when cemented to the metallic alloy substrate. Among the restorative materials, YZ and LD restorations presented the best fatigue behavior when adhesively cemented onto the metallic alloy substrate, while FEL obtained the lowest FFL and CFF for both substrates. The LD, PICN and YZ restorations showed similar fatigue performance considering the epoxy resin substrate. A more rigid foundation substrate improves the fatigue performance of adhesively cemented glass, polycrystalline and polymer infiltrated-ceramic simplified restorations.

Keywords: Dental ceramics, Cyclic loading, Monolithic restorations, Fatigue failure, Adhesion, Elastic Modulus.

Highlights:

- A more rigid substrate promotes higher fatigue performance of ceramic restorations.
- Feldspathic glass ceramic showed the lower fatigue behavior, regardless of the substrate material.
- Lithium disilicate and zirconia showed statistically similar fatigue performance on a metallic substrate.
- All tested materials showed statistical similar fatigue performance on a substrate with low modulus of elasticity, except for feldspathic ceramic.

1. Introduction

The advancement of dental ceramic materials in the last decades has provided several alternatives for indirect restorations and oral rehabilitations, especially considering Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) materials. These materials are available in homogeneous industrially manufactured blocks with different microstructures, reducing processing flaws (Tinschert et al., 2000) to promote reliable mechanical behavior against the chewing loads in the oral environment, and consequently to resist against crack propagation (da Silva et al., 2017; Kelly et al., 1989; Stawarczyk et al., 2017; Zhang and Kelly, 2017). Glass, polycrystalline and polymer infiltrated-ceramics are currently being widely indicated for monolithic restorations due to the ability to adequately replace lost dental structures by their mechanical properties and for having optical characteristics which enable wide application in prosthetic rehabilitations (da Silva et al., 2017; Venturini et al., 2019).

Glass and polymer infiltrated-ceramics are acid-sensitive, i.e., they can be conditioned with hydrofluoric acid, which promotes topographic modifications by the removal of the vitreous matrix, micromechanical interlocking, and consequently favorable bonding conditions (Scherer et al., 2018; Straface et al., 2019; Venturini et al., 2018). The high glass content of glass ceramics enables favorable translucency, making these ceramics highly esthetic, especially for anterior segment rehabilitations (da Silva et al., 2017). At the same time, PICN has a similar elastic modulus to dentin and adhesive cements, enabling stress distribution during masticatory loading, and therefore being a good option for posterior monolithic crowns (Dal Piva et al., 2018). In contrast, yttrium-stabilized zirconia (YZ) is a polycrystalline ceramic composed of crystals without glass content (Zhang and Kelly, 2017), which makes acid etching ineffective, but grants good mechanical strength, fracture toughness, and Young's modulus in the same order of magnitude of stainless-steel alloys (Piconi and Maccauro, 1999). Recent composition modifications in YZ ceramics such as the increase in the proportion of yttrium oxide (Y_2O_3) and obtaining cubic phase crystals, in addition to the tetragonal conventional structure, enabled satisfactory translucency for use in monolithic crowns, but it undeniably reduced the mechanical properties to values close to those observed by lithium disilicate glass-ceramics (Stawarczyk et al., 2017; Zhang et al., 2016; Zhang and Lawn, 2018).

Monolithic restorations generally enable less invasive prosthetic preparations (Nakamura et al., 2015), reducing the final ceramic thickness, which are great advantages in the attempt to solve critical clinical situations when lacking occlusal space between antagonist teeth (Denry and Kelly, 2014; Zimmermann et al., 2017). It also eliminates the use of metal infrastructures and reduces the risk of chipping, which can be common when using veneering porcelain in bilayer systems (Belli et al., 2016). In this sense, YZ ceramics have been exhibiting greater strength, even in extremely thin restorations (between 0.5 to 1.0 mm), and therefore they are indicated in cases of high mechanical demand (Nordahl et al., 2015). Moreover, it is important to consider that the foundation substrate upon which these materials are cemented has important influence on the mechanical behavior of these restorations (Pereira et al., 2019). Similarly, the material for prosthetic core build-up should be considered, even in glass and polymer infiltrated ceramics which traditionally have lower resistance than YZ, since it can even lead to a similar mechanical behavior of YZ, depending on the bonded substrate (Facenda et al., 2019; Kelly, 1999), and also giving good resistance with only small thickness, i.e. of 1.0mm.

Dental ceramics in clinical practice can either be adhesively cemented onto foundation substrates with low modulus of elasticity, such as composite resin and dentin (11 to 15 GPa), or with high modulus such as cast metal cores (±200 GPa) (Facenda et al., 2019). Some studies allege that a more rigid base can reduce the bending caused by chewing, and thus considerably increase the failure load of restorations, even in thicknesses of around 1 mm (Kelly, 1999; Zimmermann et al., 2017). Despite this, such an assumption still needs to be corroborated by more studies. In this sense, laboratory tests using cyclical fatigue better reproduce a clinical condition (Wiskott et al., 1995), since cyclical fatigue failure occurs suddenly at stresses below the nominal material strength due to crack propagation in the tensile stress area (Bonfante and Coelho, 2016). From this standpoint, better knowledge on the fatigue performance of full-contour ceramic restorations cemented onto different substrates is necessary to guide clinical choices. Therefore, the following question arises: what is the mechanical behavior under intermittent cyclic loading of monolithic restorations made of ceramics with distinct microstructures cemented onto foundation substrates materials with distinct elastic moduli?

In view of the above, the aim of this study was to evaluate the fatigue behavior of different ceramic materials (glass, polycrystalline and polymer infiltrated-ceramic) cemented onto substrates with distinct elastic moduli (epoxy resin – low modulus of elasticity, or Ni-Cr metal – high modulus of elasticity). The hypotheses are that: i) the metallic alloy substrate will promote higher fatigue mechanical behavior of adhesively cemented glass, polycrystalline- and polymer infiltrated-ceramic simplified restorations; and ii) the distinct ceramics cemented onto the same substrate material will influence the fatigue mechanical behavior.

2. Materials and Methods

2.1 Materials and study design

Table 1 describes the materials used in the present study, commercial name, manufacturers, batch numbers, compositions, elastic moduli and Poisson ratio of the ceramic and substrate materials.

This study was designed in 8 groups considering 2 factors (n = 15): i) Four levels of 'Ceramic material': FEL – feldspathic ceramic; PICN – polymer infiltrated ceramic; LD- lithium disilicate-based glass ceramic; or YZ – yttria-stabilized zirconia material; and ii) Two levels for 'Type of foundation substrate': ER – Epoxy resin; or MA - metallic alloy.

The simplified test assembly consists of a simplified occlusal restoration for a posterior tooth represented by ceramic discs with a final diameter of 10 mm and 1 mm of thickness which is adhesively cemented onto different substrates (low and high modulus of elasticity) with 2 mm of thickness.

2.2. Specimens Preparation

2.2.1. Ceramic discs

CAD/CAM ceramic blocks were glued to metallic rings (Ø = 10 mm for feldspathic ceramic, lithium disilicate and polymer infiltrated ceramic blocks and Ø = 12 mm for zirconia blocks) to be ground into cylinders in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) using silica carbide (SiC) papers (#600 and #1200-grit; 3M, Sumare, Brazil) under water cooling. Next, the cylinders were cut (Isomet 1000, Buehler) with a diamond disc (Buehler Isomet Wafering blade series 15LC no. 11-4276 with diameter of 6 inches (152 mm) and 0.020 (0.5 mm) in thickness, being a medium coarse diamond grinding tool) to obtain disc-shaped ceramic specimens. A total of 30 discs of each glass and polymer infiltrated-ceramic were obtained with a thickness of 1.2 mm, and 30 zirconia discs were obtained with a thickness of 1.4 mm.

Both surfaces of all ceramic discs were polished (EcoMet/AutoMet 250, Buehler) with silica carbide (SiC) papers (#400, #600 and #1200-grit; 3M, Sumare, Brazil) until a final thickness of 1.0 mm for the glass and polymer infiltrated-ceramics and 1.2 mm for the polycrystalline-ceramic (due to the sintering contraction of this material) to remove any irregularity introduced by cutting. Then, lithium disilicate specimens were crystallized (Vacumat 6000 MP, Vita Zahnfabrik, Bad Sackingen, Germany), and zirconia specimens were sintered (Zyrcomat 6000 MS, Vita Zahnfabrik; 1450°C – 120 min) in each respective specific furnace according to the manufacturer's instructions (lithium disilicate: 840° C, vacuum – 7 min; zirconia: 1450°C – 120 min).

All discs were inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper, Takatsu-ku, Kawasaki, Kanagawa, Japan) to guarantee the final dimensions according the experimental design (10 mm x 1.0), excluding any which presented discrepancies in dimensions above the recommended deviation (±0.05 mm).

2.2.2. Substrate discs (epoxy resin and metallic alloy)

Discs of two different materials (epoxy resin and metallic alloy) were obtained with final dimensions of 10 mm diameter and 2 mm thickness, and were inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper) to guarantee the dimensions, discarding any of the discs with discrepancies above 0.05 mm. All cementation surfaces of the substrate discs were polished (#1200-grit SiC paper; 3M).

2.2.2.1. Epoxy resin

Epoxy resin plates were shaped into 60 discs (thickness= 2.0 mm) using a cylindrical diamond drill (internal diameter = 10 mm; Diamant Boart, Brussels, Belgium) coupled to a bench drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) under refrigeration. The use of epoxy resin as a dentin analogue material was validated in terms of resin cement bond strength by a previous study (Kelly et al., 2010).

2.2.2.2. Metallic alloy (Ni-Cr)

A chemically cured acrylic resin (VIPI, Pirassununga, Brazil) was used to prepare 60 discs using a metal matrix (Ø = 10 mm; thickness = 2.0 mm). Then, these acrylic discs were used as standards for casting Ni-Cr alloy discs (4all, Ivoclar Vivadent, Schaan, Liechtenstein).

2.3. Surface treatments and bonding procedures

All specimens (ceramic and substrate discs) were cleaned in an ultrasonic bath (Vitasonic, Vita Zanhfabrik) with distilled water (5 min) prior to cementation procedures. All ceramic discs were randomly allocated (www.randomizer.org) into eight study groups (n=15) according to the two study factors (described a priory at subsection 2.1).

The cementation surface of the ceramic and substrate discs was treated according to Table 2. Only two operators were responsible for each step (ceramic and substrate surface treatments) for standardization.

Afterward, the resin cement (Multilink Automix, Ivoclar Vivadent) was manipulated according to the manufacturer's instructions and applied on the silanized surface of the ceramic discs. Next, a load of 2.5 N was applied over the cemented assemblies with an adapted surveyor (B2, BioArt, São Carlos, Brazil). Then, the cement excess was removed and photo-activation was performed for 20 s on each surface (0°, 90°, 180°, 270°, and occlusal surface) (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia).

2.4. Fatigue tests (Step-stress approach)

The cemented assemblies (n = 15) were submitted to a fatigue test in an electric testing machine (Instron ElectroPuls E3000; Instron Corporation, Norwood, USA) using the step-stress methodology (Kelly et al., 2017; Venturini et al., 2019). Cyclic loads were applied by a 40mm-diameter stainless steel ball piston with the specimen immersed in water (Figure 1). An adhesive tape and a polyethylene sheet (0.1 mm thick) were placed on the occlusal ceramic surface before testing between the piston and the ceramic surface to reduce contact stress concentration by improving the contact between the piston and specimen (ISO 6872-2015, 2015).

Cyclic loading was applied at a frequency of 20 Hz for 10,000 cycles at each load step, starting with an initial load of 200 N and followed by progressive load levels of 200 N (400 N, 600 N, 800 N, up to 2800 N) until failure detection. The specimen was visually inspected at the end of each load step for the presence of failure (cracks and/or fracture) by light oblique transillumination (Dibner and Kelly, 2016). If failure was detected, the test was ended and the collected data were recorded for statistical analysis (FFL – fatigue failure load and CFF – cycles for failure), but if the specimen survived, a load increment was increased and the test proceeded until observing failure.

2.5. Fractographic Analysis

After the cyclic fatigue tests, all specimens were analyzed for contact damage and to identify the crack direction by visual inspection under oblique light transmission. The fractured specimens (i.e., radial cracks) were analyzed under stereomicroscope (Discovery V20, CarlZeiss, Gottingen, Germany) in order to determine the crack propagation. Random specimens were also analyzed under scanning electron microscope (Vega3, Tescan, Czech

Republic) to inspect the fracture considering the absence of Hertzian cone cracks and other fractographic characteristics.

2.6. Finite Element Analysis (FEA)

FEA was used to evaluate the stress distribution in the test set-up. To do so, models consisting of ceramic discs, the cut steel ball layer and the foundation substrates (epoxy resin and metallic alloy) were made.

Tridimensional models were created using a software program (Rhinoceros version 5.0 SR8, McNeel North America, Seattle,WA, USA), while the analysis was performed using the ANSYS CAE software (ANSYS 19.3, ANSYS Inc., Houston, TX, USA). A static structural analysis was applied according to the fatigue experimental set-up. All materials were considered isotropic, linear, and homogeneous. Young's moduli (GPa) and Poisson's ratios for feldspar ceramic (E = 48.7 GPa; v = 0.23), polymer infiltrated ceramic (E = 30 GPa; v = 0.28), lithium disilicate (E = 95 GPa; v = 0.25), YZ (E = 200 GPa; v = 0,31), epoxy resin (E = 14.9 GPa; v = 0,31), metallic alloy (E = 220 GPa; v = 0,3), resin cement (E = 7,5 GPa, v = 0,3) and stainless steel ring/sphere (E = 190 GPa; v = 0,27) were obtained from previous studies (Dal Piva et al., 2018; Miranda et al., 2019). The models were composed of 12,062 hexahedron solid elements with 56,129 nodes. The connections among base, restoration and resin cement were considered perfectly bonded and frictional (0.12) to the piston and restoration. The models were loaded (100 N) at the top of the piston and constrained at the bottom surface of the base. After the coherence and mesh convergence test, the maximum principal stress was used as failure criteria to compare the groups.

2.7. Data analysis

A statistical software program (IBM SPSS Software; IBM, Armonk, NY, USA) was used to conduct the data analysis. Firstly, two-way ANOVA was performed (α=0.05) to access the influence of the 'ceramic material' and 'type of foundation substrate' factors under study, as well as to also access the interaction between such factors. Secondly, the load to failure and number of cycles for failure data were subjected to a survival analysis (Kaplan Meier with Mantel-Cox (log-rank) post-hoc tests (α=0.05). The survival rates were subsequently tabulated for each step of the test (for both FFL and CFF).

Next, FFL and CFF data were submitted to Weibull analysis using the Super SMITH Weibull 4.0k-32 software (Wes Fulton, Torrance, United States) under the maximum-likelihood method to obtain the Weibull modulus of each condition. The Weibull modulus is used as a measure for the distribution of the values, which is a way to statistically access the mechanical reliability of a condition/parameter.

Fractographic and Finite Element analysis were qualitatively analyzed.

3. Results

Two-way ANOVA data corroborates the statistical influence for both the 'ceramic material' (FFL – $p < .001$, $F = 76.58$; CFF – $p < .001$, $F = 76.83$) and 'type of substrate' (FFL – $p < .001$, $F = 1233.72$; CFF – $p < .001$, $F = 1260.67$) factors, as well as for the interaction between both factors (FFL – $p < .001$, $F = 11.65$; CFF – $p < .001$, $F = 11.54$).

Polymer infiltrated ceramic network (PICN), yttria-stabilized zirconia (YZ) and lithium disilicate glass-ceramic (LD) had similar fatigue failure load (FFL) and cycles for failure (CFF) when bonded to the epoxy resin (ER) substrate, with higher FFL/CFF than feldspathic ceramic (FEL) in this substrate condition (ER: PICN=YZ=LD >FEL). However, YZ=LD>PICN>FEL when the ceramics were cemented onto metallic alloy (MA) substrate (Table 3).

The ceramic materials cemented onto metallic alloy substrate had statistically significant increase in the two evaluated parameters (FFL and CFF) (Table 3). The survival analysis corroborates the aforementioned findings: the samples cemented onto a stiffer substrate lasted longer until failure (Table 4). Fig. 2 shows the survival curves for the two evaluated parameters (fatigue failure load – FFL; cycles for failure – CFF).

For the Weibull modulus, all evaluated ceramics showed greater mechanical structural reliability when cemented onto a stiffer substrate (the metallic alloy) (stiffer > softer). In addition, no difference was detected among the tested ceramics when cemented onto stiffer substrate. In fact, there was only a statistical difference in Weibull modulus among the simplified restorations when adhesively cemented onto the softer substrate (epoxy resin), where YZ presented lower values compared to LD (Table 3).

The failure analysis under a light microscope showed that radial cracks starting from the cemented surface took place in all failed specimens, and there was no Hertzian cone cracks. Representative SEM micrographs of the fracture surfaces are presented in Fig. 3. The YZ.MA group showed catastrophic failures with fragment loss due to a high-applied load.

Fig. 4 shows that the metallic alloy substrate promoted a reduced Maximum Principal Stress (MPS) concentration in all restorative materials. In addition, the restorative materials with the highest elastic modulus (LD and YZ) presented the highest MPS values in both evaluated substrates.

4. Discussion

The present study demonstrated that simplified restorations of glass, polycrystalline and polymer infiltrated-ceramics adhesively cemented onto a metallic substrate presented improved fatigue behavior compared to the same restorations cemented onto a dentin analogue substrate (epoxy resin). In addition, the different restorative materials showed different behaviors when cemented onto the same foundation substrate material. Thus, the hypotheses were accepted.

Indirect restorative materials suffer flexural demands under usual chewing and consequently increase the tensile stress concentration at the cementation interface between restoration and substrate (Dal Piva et al., 2018; Kelly, 1999). In this sense, the majority of bulk fractures of ceramic crowns initiated from the intaglio surface (cementation surface) where tensile stresses are concentrated (Kelly et al., 1989; Thompson et al., 1994). Although the different classes of dental ceramics and their characteristics are well known in the literature (da Silva et al., 2017; Venturini et al., 2019), the fatigue behavior of these materials deserves a better evaluation considering the mechanical context required in a buccal environment, since these materials can be cemented onto different substrates, such as dental substrate or prosthetic core reconstruction materials (Creugers et al., 2005; Zimmermann et al., 2017).

Recent studies have suggested that substrates with higher elastic modulus such as Ni-Cr alloy with an elastic modulus ten times greater than dentin (Dal Piva et al., 2018; Facenda et al., 2019), prevent ceramic bending and consequently improve the material's response to loads on its occlusal surface (Facenda et al., 2019; Machry et al., 2021; Pereira et al., 2019). The present study agrees with this premise when it presents that it was possible to promote better fatigue performance of restorations under cyclic loads when they were cemented onto a metallic substrate with high elastic modulus. This was also corroborated through the Finite Element Analysis (Fig. 4), where a reduction in the maximum principal stress (MPS) can be seen in all restorative materials when cemented onto a metallic substrate. In addition, our study considered the Weibull modulus, which allows accessing the structural mechanical reliability of each cemented assembly according to the average data dispersion, where high values may reflect higher structural reliability (Venturini et al., 2019). In this sense, all ceramics showed high Weibull modulus values when cemented onto a stiffer substrate due to the low variability of the results obtained. This allows us to infer good reliability of ceramic materials in this condition.

Our representative images by scanning electron microscope can suggest that the failures originate in the cementation area (in agreement with the aforementioned clinical characteristic) (Kelly, 1999) due to the absence of Hertzian cone cracks on the occlusal surface also evaluated under transillumination inspection. Furthermore, total ceramic fractures and detachment of restoration were observed for the ceramics cemented onto metallic alloys. In this scenario, the failures occurred suddenly under extremely high load values, unlike the crack propagation which occurs at values below the nominal load of the materials which are typical for fatigue (Bonfante and Coelho, 2016; May et al., 2012). In addition, the main reason for ceramic debonding may be the low adhesion between the resin cement and the dental metallic alloys such as Ni-Cr (Freitas and Francisconi, 2004; Jamel et al., 2019), which may have caused the complete material rupture after crack initiation. Therefore, the influence of bond strength between cement and substrate on fatigue behavior should be separately evaluated in future studies.

Finite element analysis (Fig. 4) enabled observing that not only the rigidity of substrates, but also the elastic moduli of the evaluated dental ceramics (glass, polycrystalline zirconia, and polymer infiltrated-ceramic) are related to the way that stresses are distributed through restoration. Highly rigid restorative materials such as zirconia tend to be subject to higher stress peaks on the inner surface of the restoration (Tribst et al., 2018), which could be associated with a higher probability of crack propagation. Nevertheless, the microstructure and the composition of the ceramic material must also be considered for the mechanical performance evaluation, not only the Young's modulus (da Silva et al., 2017). Polycrystalline ceramics have excellent mechanical properties due to their tightly connected crystalline composition which is capable of deflecting cracks that spread or penetrate the gaps formed during the deformation caused by chewing (Stawarczyk et al., 2017; Zhang and Kelly, 2017). Furthermore, high strength values are expected from dental zirconia compared to glass ceramics such as feldspathic ceramic and lithium disilicate (Kwon et al., 2018). However, our study shows a similar fatigue behavior between the LD and YZ restorations cemented onto the two evaluated substrates. This can be explained by the fact that it is a third-generation zirconia, and is more esthetically favorable compared to previous generations, but with reduced strength and toughness due to the increased grain size and the inability to undergo toughening mechanism by the phase transformation (Machry et al., 2021; Pereira et al., 2018; Zucuni et al., 2020) which occurs in first and second zirconia generations (partially stabilized). Likewise, PICN has been shown to be a material with excellent capacity to absorb chewing loads, even being indicated for extra thin restorations in the posterior region (Abu-Izze et al., 2018; Dal Piva et al., 2018; Maeder et al., 2019; Tribst et al., 2018). PICN in the present study had similar fatigue mechanical behavior to LD and YZ when cemented onto dentin analogue material. Although the flexural strength of these materials is known to be lower than the others (Guilardi et al., 2020), its fatigue behavior is favored when adhesively cemented and may present failure load even higher than LD (Velho et al., 2020). Unlike brittle materials such as glass and polycrystalline ceramics, this material has an elastic modulus close to human dentin and resin cements. Therefore, it generates a more uniform stress distribution in the system (restoration, cement and dentin), and consequently less chance of starting crack propagation on the internal surface of the restoration (Coldea et al., 2013; Tribst et al., 2018).

It is important to point out that the thickness evaluated in our study was the minimum indicated by the lithium disilicate manufacturer (1 mm). The literature shows that reduced thicknesses can be applied to dental ceramics with satisfactory results (Abu-Izze et al., 2018; Maeder et al., 2019; Nordahl et al., 2015; Yan et al., 2018; Zimmermann et al., 2017). However, studies evaluating different ceramic thicknesses do not consider substrate variability and its elastic modulus. It could be questioned whether the behavior of these materials would be improved by reduced thickness when cemented onto a high modulus substrate, since we observed a considerable increase in the fatigue performance of lithium disilicate cemented onto the Ni-Cr substrate. Likewise, the esthetics of restorations must also be evaluated, and at this point lower thicknesses of translucent materials could be unable to mask dark substrates such as a metal alloy (Bacchi et al., 2019). Hence, from the clinical point of view, a grayish effect might occur when translucent ceramic materials are cemented onto metallic substrates, and as such we stress caution in interpreting our findings.

Finally, we have to state that our study has some limitations such as using the fatigue evaluation of simplified restorations and the load application direction, which did not consider lateral forces and sliding commonly present in the oral environment. Nevertheless, our findings contribute to clarify an important clinical issue, being that the elastic modulus of the substrate may influence the fatigue mechanical behavior of the ceramic restoration.

5. Conclusions

- A stiffer substrate improves the fatigue performance of adhesively cemented glass, polycrystalline and polymer infiltrated-ceramic restorations, while a decrease in mechanical performance was observed when these restorations were bonded onto a softer substrate.

- The lithium disilicate, polycrystalline and polymer infiltrated-ceramics presented similar fatigue performance when cemented onto a dentin analogue substrate.
- The lithium disilicate and polycrystalline ceramics presented similar fatigue performance when cemented onto a metal alloy substrate.

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REFERENCES

Abu-Izze, F.O., Ramos, G.F., Borges, A.L.S., Anami, L.C., Bottino, M.A., 2018. Fatigue behavior of ultrafine tabletop ceramic restorations. Dent. Mater. 34, 1401–1409. https://doi.org/10.1016/j.dental.2018.06.017

Bacchi, A., Boccardi, S., Alessandretti, R., Pereira, G.K.R., 2019. Substrate masking ability of bilayer and monolithic ceramics used for complete crowns and the effect of association with an opaque resin-based luting agent. J. Prosthodont. Res. 63, 321–326. https://doi.org/10.1016/j.jpor.2019.01.005

Belli, R., Petschelt, A., Hofner, B., Hajtó, J., Scherrer, S.S., Lohbauer, U., 2016. Fracture Rates and Lifetime Estimations of CAD/CAM All-ceramic Restorations. J. Dent. Res. 95, 67–73. https://doi.org/10.1177/0022034515608187

Bonfante, E.A., Coelho, P.G., 2016. A Critical Perspective on Mechanical Testing of Implants and Prostheses. https://doi.org/10.1177/0022034515624445

Coldea, A., Swain, M. V., Thiel, N., 2013. Mechanical properties of polymer-infiltrated-ceramic-network materials. Dent. Mater. 29, 419–426. https://doi.org/10.1016/j.dental.2013.01.002

Creugers, N., Mentink, A., Fokkinga, W., Kreulen, C., 2005. 5-Year Follow-Up of A Prospective Clinical Study on Various Types of Core Restorations. Int. J. Prosthodont. 18, 34–39. https://doi.org/10.1016/j.prosdent.2005.05.013

da Silva, L.H., de Lima, E., Miranda, R.B. de P., Favero, S.S., Lohbauer, U., Cesar, P.F., 2017. Dental ceramics: A review of new materials and processing methods. Braz. Oral Res. 31, 133–146. https://doi.org/10.1590/1807-3107BOR-2017.vol31.0058

Dal Piva, A.M. de O., Tribst, J.P.M., Borges, A.L.S., Souza, R.O. de A. e., Bottino, M.A., 2018. CAD-FEA modeling and analysis of different full crown monolithic restorations. Dent. Mater. 34, 1342–1350. https://doi.org/10.1016/j.dental.2018.06.024

Denry, I., Kelly, J.R., 2014. Emerging ceramic-based materials for dentistry. J. Dent. Res. 93, 1235–1242. https://doi.org/10.1177/0022034514553627

Dibner, A.C., Kelly, J.R., 2016. Fatigue strength of bilayered ceramics under cyclic loading as a function of core veneer thickness ratios. J. Prosthet. Dent. 115, 335–340. https://doi.org/10.1016/j.prosdent.2015.09.017

Facenda, J.C., Borba, M., Benetti, P., Della Bona, A., Corazza, P.H., 2019. Effect of supporting substrate on the failure behavior of a polymer-infiltrated ceramic network material. J. Prosthet. Dent. 121, 929–934. https://doi.org/10.1016/j.prosdent.2018.08.008

Freitas, A.P. de, Francisconi, P.A.S., 2004. Effect of a metal primer on the bond strength of the resin-metal interface. J. Appl. Oral Sci. 12, 113–116. https://doi.org/10.1590/s1678-77572004000200006

ISO 6872, 2015. Dentistry - Ceramic Materials. International Organization for Standardization, n.d.

J. Robert Kelly, D.D.S., M.S.,* Stephen D. Campbell, D.M.D., M.M.Sc., * and, H. Kent Bowen, P.D.**., 1989. Fracture-surface analysis of dental ceramics. J. Prosthet. Dent. 62, 536–541. https://doi.org/10.1002/9781118144152.ch21

Jamel, R.S., Nayif, M.M., Abdulla, M.A., 2019. Influence of different surface treatments of nickel chrome metal alloy and types of metal primer monomers on the tensile bond strength of a resin cement. Saudi Dent. J. 31, 343–349. https://doi.org/10.1016/j.sdentj.2019.03.006

Kelly, J.R., 1999. Clinically relevant approach to failure testing of all-ceramic restorations. J. Prosthet. Dent. 81, 652–661. https://doi.org/10.1016/S0022-3913(99)70103-4

Kelly, J.R., Cesar, P.F., Scherrer, S.S., Della Bona, A., van Noort, R., Tholey, M., Vichi, A., Lohbauer, U., 2017. ADM guidance-ceramics: Fatigue principles and testing. Dent. Mater. 33, 1192–1204. https://doi.org/10.1016/j.dental.2017.09.006

Kelly, J.R., Rungruanganunt, P., Hunter, B., Vailati, F., 2010. Development of a clinically validated bulk failure test for ceramic crowns. J. Prosthet. Dent. 104, 228–238. https://doi.org/10.1016/S0022-3913(10)60129-1

Kwon, S.J., Lawson, N.C., McLaren, E.E., Nejat, A.H., Burgess, J.O., 2018. Comparison of the mechanical properties of translucent zirconia and lithium disilicate. J. Prosthet. Dent. 120, 132–137. https://doi.org/10.1016/j.prosdent.2017.08.004

LF, G., P, S., A, W., de Jager N, GKR, P., CJ, K., MP, R., LF, V., 2020. Fatigue performance of distinct CAD/CAM dental ceramics. J. Mech. Behav. Biomed. Mater. 103, 103540. https://doi.org/10.1016/j.jmbbm.2019.103540

Machry, R. V, Cadore-Rodrigues, A.C., Luiz, A., Borges, S., Pereira, G.K.R., Kleverlaan, C.J., Venturini, A.B., Felipe, L., 2021. Fatigue resistance of simplified CAD – CAM restorations : Foundation material and ceramic thickness effects on the fatigue behavior of partially- and fully-stabilized zirconia. Dent. Mater. https://doi.org/10.1016/j.dental.2021.01.001

Maeder, M., Pasic, P., Ender, A., Özcan, M., Benic, G.I., Ioannidis, A., 2019. Load-bearing capacities of ultra-thin occlusal veneers bonded to dentin. J. Mech. Behav. Biomed. Mater. 95, 165–171. https://doi.org/10.1016/j.jmbbm.2019.04.006

May, L.G., Kelly, J.R., Bottino, M.A., Hill, T., 2012. Effects of cement thickness and bonding on the failure loads of CAD / CAM ceramic crowns : Multi-physics FEA modeling and monotonic testing. Dent. Mater. 28, e99–e109. https://doi.org/10.1016/j.dental.2012.04.033

Miranda, J.S., de Carvalho, R.L.A., de Carvalho, R.F., Borges, A.L.S., Bottino, M.A., Özcan, M., Melo, R.M. de, Souza, R.O. de A. e., 2019. Effect of different loading pistons on stress distribution of a CAD/CAM silica-based ceramic: CAD-FEA modeling and fatigue survival analysis. J. Mech. Behav. Biomed. Mater. 94, 207–212.

https://doi.org/10.1016/j.jmbbm.2019.03.011

Nakamura, K., Harada, A., Inagaki, R., Kanno, T., Niwano, Y., Milleding, P., Örtengren, U., 2015. Fracture resistance of monolithic zirconia molar crowns with reduced thickness. Acta Odontol. Scand. 73, 602–608. https://doi.org/10.3109/00016357.2015.1007479

Nordahl, N., Vult von Steyern, P., Larsson, C., 2015. Fracture strength of ceramic monolithic crown systems of different thickness. J. Oral Sci. 57, 255–261. https://doi.org/10.2334/josnusd.57.255

Pereira, G.K.R., Graunke, P., Maroli, A., Zucuni, C.P., Prochnow, C., Valandro, L.F., Caldas, R.A., Bacchi, A., 2019. Lithium disilicate glass-ceramic vs translucent zirconia polycrystals bonded to distinct substrates: Fatigue failure load, number of cycles for failure, survival rates, and stress distribution. J. Mech. Behav. Biomed. Mater. 91, 122–130. https://doi.org/10.1016/j.jmbbm.2018.12.010

Pereira, G.K.R., Guilardi, L.F., Dapieve, K.S., Kleverlaan, C.J., Rippe, M.P., Valandro, L.F., 2018. Mechanical reliability, fatigue strength and survival analysis of new polycrystalline translucent zirconia ceramics for monolithic restorations. J. Mech. Behav. Biomed. Mater. 85, 57–65. https://doi.org/10.1016/j.jmbbm.2018.05.029

Piconi, C., Maccauro, G., 1999. Zirconia as a ceramic biomaterial. Biomaterials 20, 1–25. https://doi.org/10.1016/S0142-9612(98)00010-6

Scherer, M.M., Prochnow, C., Venturini, A.B., Pereira, G.K.R., Burgo, T.A. de L., Rippe, M.P., Valandro, L.F., 2018. Fatigue failure load of an adhesively-cemented lithium disilicate glass-ceramic: Conventional ceramic etching vs etch & prime one-step primer. Dent. Mater. 34, 1134–1143. https://doi.org/10.1016/j.dental.2018.04.012

Stawarczyk, B., Keul, C., Eichberger, M., Figge, D., Edelhoff, D., Lümkemann, N., 2017. Three generations of zirconia: From veneered to monolithic. Part I. Quintessence Int. 48, 369–380. https://doi.org/10.3290/j.qi.a38057

Straface, A., Rupp, L., Gintaute, A., Fischer, J., Zitzmann, N.U., Rohr, N., 2019. HF etching of CAD/CAM materials: Influence of HF concentration and etching time on shear bond strength. Head Face Med. 15, 1–10. https://doi.org/10.1186/s13005-019-0206-8

Thompson, J.Y., Anusavice, K.J., Naman, A., Morris, H.E., 1994. Fracture Surface Characterization of Clinically Failed All-ceramic Crowns. J. Dent. Res. 73, 1824–1832. https://doi.org/10.1177/00220345940730120601

Tinschert, J., Zwez, D., Marx, R., Anusavice, K.J., 2000. Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics. J. Dent. 28, 529–535. https://doi.org/10.1016/S0300-5712(00)00030-0

Tribst, J.P.M., Dal Piva, A.M. de O., Penteado, M.M., Borges, A.L.S., Bottino, M.A., 2018. Influence of ceramic material, thickness of restoration and cement layer on stress distribution of occlusal veneers. Braz. Oral Res. 32, e118. https://doi.org/10.1590/1807-3107bor-2018.vol32.0118

Velho, H.C., Dapieve, K.S., Pereira, G.K.R., Fraga, S., Valandro, L.F., Venturini, A.B., 2020. Journal of the Mechanical Behavior of Biomedical Materials Accelerated loading frequency does not influence the fatigue behavior of polymer infiltrated ceramic network or lithium disilicate glass-ceramic restorations. J. Mech. Behav. Biomed. Mater. 110, 103905. https://doi.org/10.1016/j.jmbbm.2020.103905

Venturini, A.B., Prochnow, C., Pereira, G.K.R., Segala, R.D., Kleverlaan, C.J., Valandro, L.F., 2019. Fatigue performance of adhesively cemented glass-, hybrid- and resin-ceramic materials for CAD/CAM monolithic restorations. Dent. Mater. 35, 534–542. https://doi.org/10.1016/j.dental.2019.01.013

Venturini, A.B., Prochnow, C., Pereira, G.K.R., Werner, A., Kleverlaan, C.J., Valandro, L.F., 2018. The effect of hydrofluoric acid concentration on the fatigue failure load of adhesively cemented feldspathic ceramic discs. Dent. Mater. 34, 667–675. https://doi.org/10.1016/j.dental.2018.01.010

Wiskott, H.W., Nicholls, J.I., Belser, U.C., 1995. Stress fatigue: basic principles and prosthodontic implications. Int. J. Prosthodont. 8, 105–16.

Yan, J., Kaizer, M.R., Zhang, Y., 2018. Load-bearing capacity of lithium disilicate and ultra-translucent zirconias. J. Mech. Behav. Biomed. Mater. 88, 170–175. https://doi.org/10.1016/j.jmbbm.2018.08.023

Zhang, Y., Kelly, J.R., 2017. Dental Ceramics for Restoration and Metal Veneering. Dent. Clin. North Am. 61, 797–819.

Zhang, Y., Lawn, B.R., 2018. Novel Zirconia Materials in Dentistry. J. Dent. Res. 97, 140–147. https://doi.org/10.1177/0022034517737483

Zhang, Y., Mai, Z., Barani, A., Bush, M., Lawn, B., 2016. Fracture-resistant monolithic dental crowns. Dent. Mater. 32, 442–449. https://doi.org/10.1016/j.dental.2015.12.010

Zimmermann, M., Egli, G., Zaruba, M., Mehl, A., 2017. Influence of material thickness on fractural strength of CAD/CAM fabricated ceramic crowns. Dent. Mater. J. 36, 778–783. https://doi.org/10.4012/dmj.2016-296

Zucuni, C.P., Pereira, G.K.R., Valandro, L.F., 2020. Grinding, polishing and glazing of the occlusal surface do not affect the load-bearing capacity under fatigue and survival rates of bonded monolithic fully-stabilized zirconia simplified restorations. J. Mech. Behav. Biomed. Mater. 103, 103528. https://doi.org/10.1016/j.jmbbm.2019.103528

TABLES

Table 1. Commercial name, manufacturer, batch number and composition (including Elastic moduli and Poisson ratio to ceramic and substrate materials) of the materials used in this in vitro study.

Material	Commercial name, manufacturer (batch number)	Composition	Elastic moduli/Poisson ratio
Feldspathic ceramic	VITA Mark II, VITA Zahnfabrik (43800)	SiO_2 56-64 wt%, Al_2O_3 20-23 wt%, K_2O 6-8 wt%, Na_2O 6-9 wr%, other and coloring oxide 0-0.6 wt%	48.7GPa/0,22
Polymer infiltrated ceramic network	VITA Enamic, VITA Zahnfabrik (49620)	SiO_2 58-63 wt%, Al_2O_3 20-23 wt%, K_2O 4-6 wt%, Na_2O 6-11 wt%, B_2O_3 0,5-2 wt%, other and coloring oxide 0-1 wt%	30GPa/0,28
Lithium disilicate	IPS CAD, Ivoclar Vivadent (W89815)	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%	95GPa/0,25
Yttria-stabilized Zirconia	IPS ZirCAD MT Multi, Ivoclar Vivadent (W82689)	ZrO_2 86 – 93.5 wt%, Y_2O_3 > 6.5 % – ≤ 8.0 wt%, HfO_2 ≤ 5.0 wt%, Al_2O_3 ≤ 1.0 wt%, other oxides ≤ 1.0 wt%	200GPa/0,31
Dentin analogue material	Epoxy Plate 150 Plate 150 × 350 x 2.0 mm; Carbotec GmbH & Co. KG (229861)	Continuous filament woven fiberglass bonded with epoxy resin	14.9GPa/0,31
Metallic alloy	4all, Ivoclar Vivadent (T32718)	Ni 61.4%, Cr 25.7%, Mo 11.0%, Si 1.5%, Mn <1.0%, Al <1.0%, C <1.0%	220GPa/0,33
Dual cure resin cement	Multilink Automix, Ivoclar Vivadent (W07285)	Dimethacrylates, HEMA, barium glass filler, Ba-Al-Fluoro-Silicate glass, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments	7.5GPa/0,3
10% hydrofluoric acid	Condac Porcelana, FGM (060519)	10% hydrofluoric acid, water, thickener, surfactant and coloring	-
5% hydrofluoric acid	IPS Ceramic Etching Gel, Ivoclar Vivadent (W14921)	< 5% hydrofluoric acid HF	-
Silane coupling agent	Monobond Plus, Ivoclar Vivadent (W90329)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate	-
Primer	Multilink Primer (A and B), Ivoclar Vivadent (W88902/W4494)	Primer A: water, initiators; primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabilizer	-
Aluminum oxide (metal treatment)	Bio-Art Equipamentos Odontológicos Ltda. (58221)	Aluminum Oxide (120 µm)	-
Aluminum oxide (zirconia treatment)	Polidental Indústria e Comércio (44493)	Aluminum Oxide (45 µm)	-

Table 2. Surface treatments for ceramic material and substrate

Material	Surface treatment procedures	
	Etching Strategy	**Adhesive Strategy**
Feldspathic ceramic	Etching with 5% hydrofluoric acid 60 s, washing 30 s, ultrasonic cleaning 5 min (distilled water).	A primer containing multiple bond promoters (Monobond Plus, Ivoclar Vivadent) was applied for 60 s (scrubbed on the bonding surfaces off all zirconia discs for 15 s and then kept reacting for 45 s).
Polymer infiltrated ceramic network		
Lithium disilicate	Etching with 5% hydrofluoric acid 20 s, washing 30 s, ultrasonic cleaning 5 min (distilled water).	
Yttria-stabilized zirconia	Air-abrasion with aluminum oxide particles (45 µm, 10 mm distance, 2.8 bars) 10 s, ultrasonic cleaning 5 min (distilled water).	
Epoxy resin	Etching with 10% hydrofluoric acid 60 s, washing 30 s, ultrasonic cleaning 5 min (distilled water).	Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the treated surfaces of all substrate discs for 30 s and air-dried until a thin layer was obtained
Metallic alloy	Air-abrasion with aluminum oxide particles (120 µm, 10 mm distance, 5 bars) 10 s, ultrasonic cleaning 5 min (distilled water).	

Table 3. Results for fatigue failure load in Newtons (FFL) and number of cycles until failure (CFF).

Groups' codes	Restorative Materials	Substrate	FFL (Mean – 95%CI)	Weibull Modulus for FFL (Mean – 95%CI)	Cycles (Mean – 95%CI)	Weibull Modulus for Cycles (Mean – 95%CI)
FEL.ER	**Feldspathic ceramic**	**Epoxy Resin**	627 (574 – 679) E	8.42 (6.44 – 11.02) BCD	31,333 (28,720 – 33,947) E	8.42 (6.44 – 11.02) BCD
FEL.MA		**Metallic Alloy**	1960 (1882 – 2154) C	16.90 (12.62 – 22.63) A	98,000 (94,080 – 101,920) C	16.90 (12.62 – 22.63) A
PICN.ER	**Polymer infiltrated ceramic network**	**Epoxy Resin**	1280 (1154 – 1406) D	5.71 (4.01 – 8.14) CD	64,115 (57,896 – 70,333) D	5.76 (4.05 – 8.20) CD
PICN.MA		**Metallic Alloy**	2213 (2132 – 2294) B	17.37 (12.44 – 24.26) A	111,333 (107,114 – 115,553) B	16.65 (11.77 – 23.55) A
LD.ER	**Lithium disilicate glass-ceramic**	**Epoxy Resin**	1227 (1142 – 1311) D	9.52 (7.32 – 12.39) BC	61,333 (57,114 – 65,554) D	9.52 (7.32 – 12.39) BC
LD.MA		**Metallic Alloy**	2707 (2554 – 2860) A	13.56 (8.95 – 20.56) AB	135,333 (127,679 – 142,987) A	13.56 (8.95 – 20.56) AB
YZ.ER	**Yttria-stabilized zirconia ceramic**	**Epoxy Resin**	1267 (1120 – 1413) D	5.28 (3.90 – 7.15) D	62,717 (55,618 – 69,816) D	5.35 (3.95 – 7.24) D
YZ.MA		**Metallic Alloy**	2733 (2663 – 2804) A	25.72 (17.11 – 38.68) A	136,668 (133,128 – 140,205) A	25.72 (17.11 – 38.68) A

Different letters in each column indicate statistically significant differences ($P < .05$) using maximum likelihood estimations for Weibull Modulus, and Kaplan Meier followed by Mantel-cox post-hoc test for FFL and CFF.

Groups	Steps of load to failure and steps of number of cycles until failure													
	200N 10 x 10^3	**400N 20 x 10^3**	**600N 30 x 10^3**	**800N 40 x 10^3**	**1000N 50 x 10^3**	**1200N 60 x 10^3**	**1400N 70 x 10^3**	**1600N 80 x 10^3**	**1800N 90 x 10^3**	**2000N 100 x 10^3**	**2200N 110 x 10^3**	**2400N 120 x 10^3**	**2600N 130 x 10^3**	**2800N 140 x 10^3**
FEL.ER	1	0.93 (0.06)	0.20 (0.10)	0.0	-	-	-	-	-	-	-	-	-	-
FEL.MA	1	1	1	1	1	1	1	1	0.60 (0.13)	0.20 (0.10)	0.0	-	-	-
PICN.ER	1	1	1	0.93 (0.06)	0.73 (0.11)	0.53 (0.13)	0.20 (0.10)	0.0	-	-	-	-	-	-
PICN.MA	1	1	1	1	1	1	1	1	1	0.73 (0.11)	0.33 (0.12)	0.0	-	-
LD.ER	1	1	1	1	0.80 (0.10)	0.27 (0.11)	0.07 (0.06)	0.0	-	-	-	-	-	-
LD.MA	1	1	1	1	1	1	1	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.87 (0.09)	0.87 (0.09)	0.73 (0.11)
YZ.ER	1	1	1	0.93 (0.06)	0.67 (0.12)	0.46 (0.13)	0.13 (0.09)	0.13 (0.09)	0.0	-	-	-	-	-
YZ.MA	1	1	1	1	1	1	1	1	1	1	1	0.87 (0.09)	0.80 (0.10)	0.80 (0.10)

* Kaplan Meier and Mantel-Cox tests.
** The symbol '-' indicates absence of specimens being tested on the respective step.

Table 4. Survival rates – probability of specimens to exceed the respective load to failure (FFL) and number of cycles until failure (CFF) steps, and its respective standard error values.

FIGURES

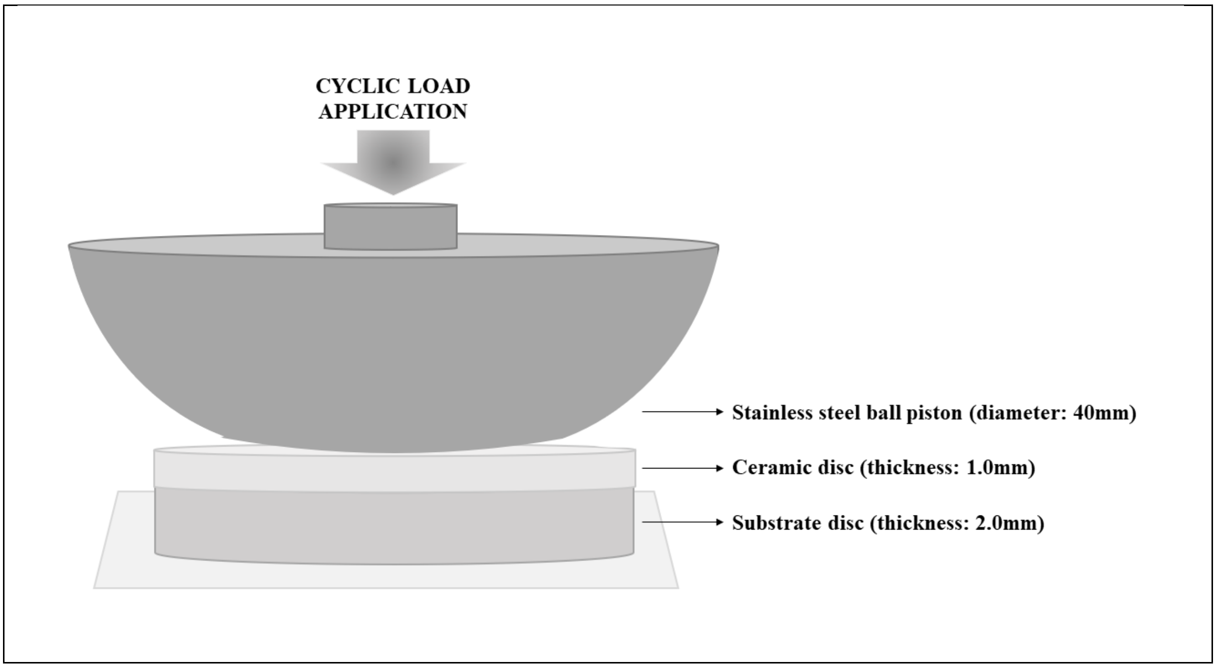


Figure 1. Fatigue test setup.

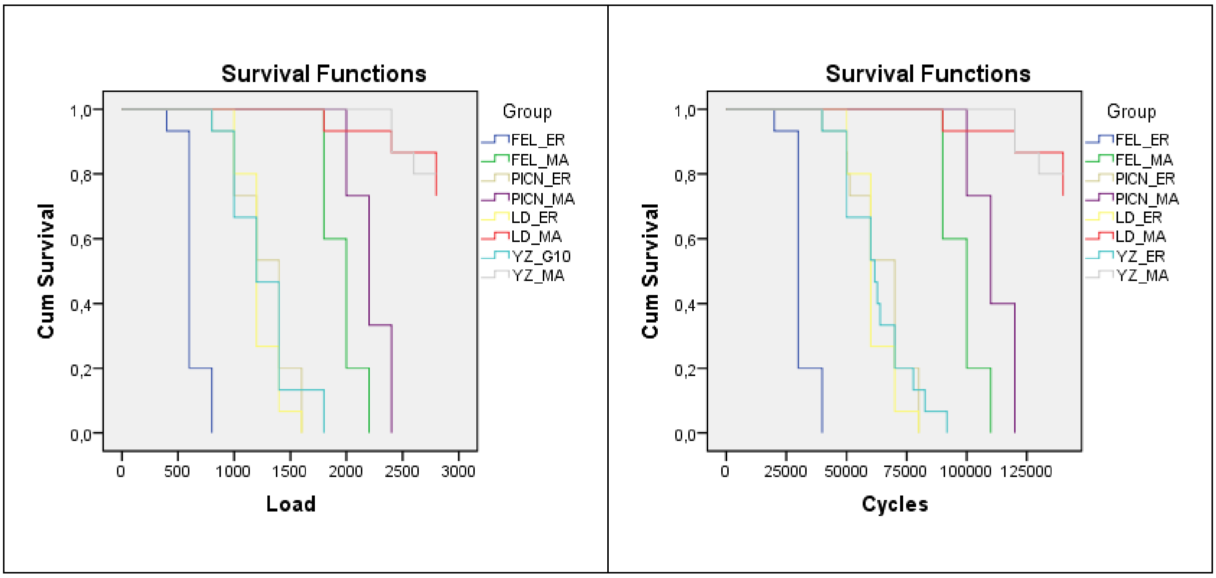


Figure 2. Survival curves according to the steps of fatigue failure load (left) and number of cycles (right) in which each disc failed, obtained by Kaplan–Meier and Log-rank tests.

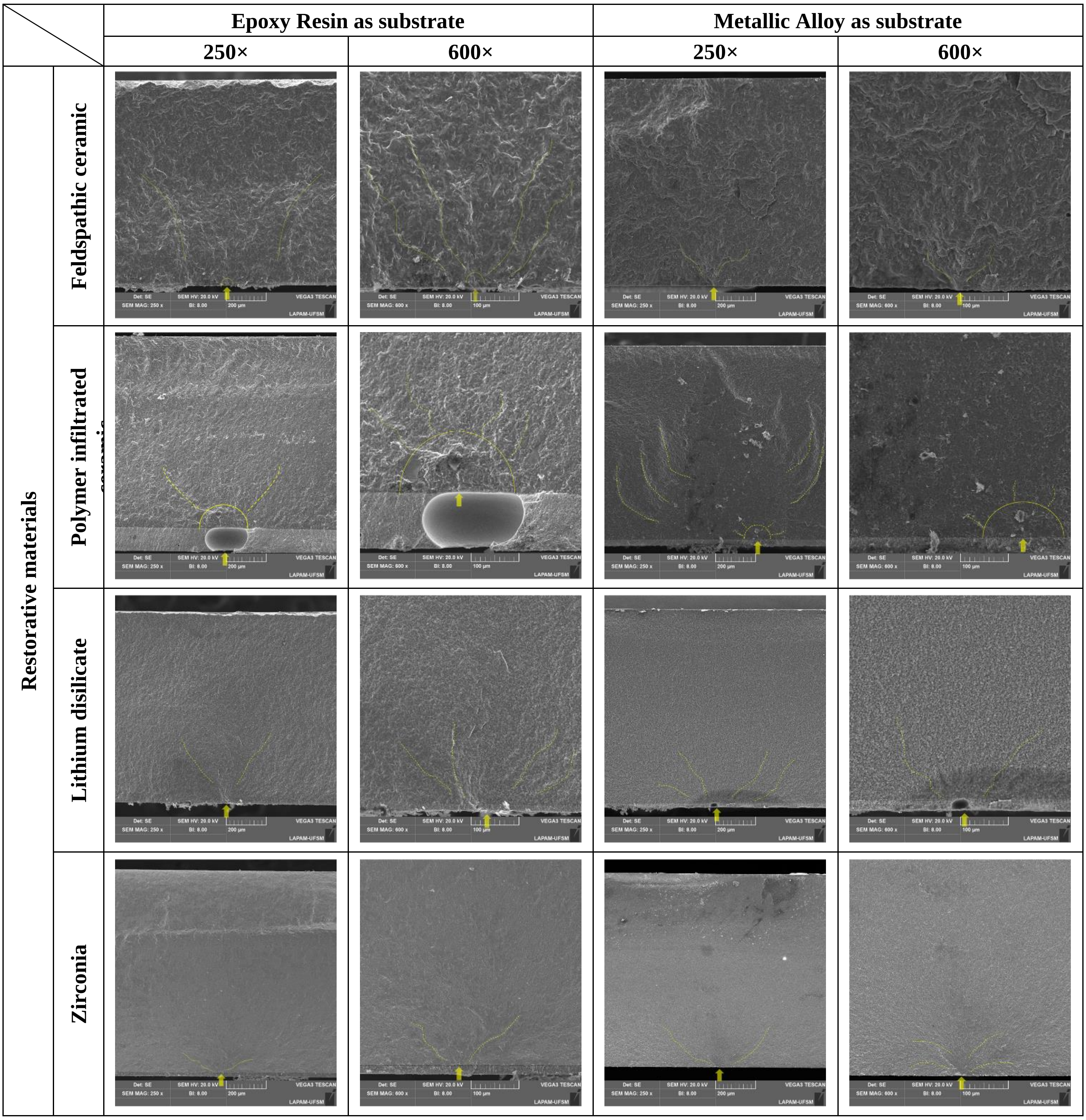


Figure 3. Representative SEM images (250× and 600×) of ceramic discs with failures originated at cementation surface from defects present at the ceramic surface, pointed by yellow arrows, and then propagated to the opposite side (occlusal/top surface). The dashed lines demonstrate the propagation of the failure.

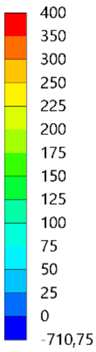
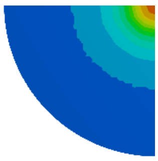
Restorative Materials	Surface view	Substrates				Scale
		Epoxy Resin		Metallic Alloy		
		FEA Images	MPS	FEA Images	MPS	
Feldspathic ceramic	Side Section view		184.56 MPa		141.35 MPa	
	Inner surface view					
Polymer infiltrated ceramic	Side Section view		127.44 MPa		86.41 MPa	
	Inner surface view					
Lithium disilicate glass-ceramic	Side Section view		278.83 MPa		232.15 MPa	
	Inner surface view					
Yttria-stabilized polycrystalline zirconia	Side Section view		395.88 MPa		341.91 MPa	
	Inner surface view					

Figure 4. FEA analysis demonstrates the tensile stress in the inner surface of the restorations (Maximum Principal Stress – MPS). Under the same applied load, the stress concentration on the inner surface of ceramic discs decreased on a stiffer substrate. In addition, more rigid restorative materials lead to a higher concentration of stress on the internal surface when the same substrate was considered

4. ARTIGO 3 – INFLUENCE OF SURFACE TREATMENT OF RESIN COMPOSITE PROSTHETIC CORE ON THE LOAD-BEARING CAPACITY UNDER FATIGUE OF LITHIUM DISILICATE MONOLITHIC SIMPLIFIED RESTORATIONS

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Influence of surface treatment of resin composite prosthetic core on the load-bearing capacity under fatigue of lithium disilicate monolithic simplified restorations

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Short title: Effect of resin composite substrate surface treatment on the fatigue behavior of lithium disilicate restorations.

ABSTRACT

This study evaluated the influence of surface treatments of resin composite substrate on the fatigue behavior of adhesively cemented lithium disilicate glass-ceramic simplified restorations. CAD/CAM lithium disilicate ceramic blocks were shaped into discs (N= 60, Ø= 10 mm; thickness= 1.0 mm). Resin composite discs (N= 60, Ø= 10 mm, thickness= 2 mm) were allocated into four groups considering the "surface treatment" factor: Ctrl – no surface treatment; Bur – grinding with coarse diamond bur (#3101G, KG Sorensen); PA – etching with 37% phosphoric acid (15 s); AA – air abrasion with alumina particles (45 µm, 10 mm distance, 2.8 bars, 10 s). The surface topography, the roughness, the fractal dimension (estimated by the box counting method) and the contact angle analyses were performed after the surface treatments. The lithium disilicate discs were etched (5% hydrofluoric acid, 20 s), silanized and adhesively cemented (Multilink N, Ivoclar Vivadent) on the resin composite discs. The samples (bonded restoration set) were subjected to a step-stress fatigue test at 20 Hz, 10,000 cycles/step with a step-size of 100 N applied on the ceramic surface, having ceramic up and resin composite down. Fractographic analysis was performed. The fatigue data (Fatigue Failure Load – FFL; and Cycles for Failure – CFF) were analyzed by Kaplan Meier with Mantel-Cox log-rank post-hoc tests ($\alpha = 0.05$). No statistical difference for fatigue performance could be found among the groups (FFL means: 820 – 867 N; CFF means: 53195 – 61090 cycles). The bur group showed higher surface roughness and contact angle values. The PA group the highest average fractal dimension. Therefore, the resin composite surface treatment for core build-up induces topographical changes, however it has no effect on the fatigue behavior of lithium disilicate restorations.

Keywords: Ceramics, Surface conditioning, Adhesion, Fatigue phenomena, Survival analysis.

Highlights:

- The resin composite surface treatment has no influence on the fatigue behavior of bonded lithium disilicate restorations.
- The grinding and particle air-abrasion promote greater roughness and topographical changes of the resin composite surface.
- The phosphoric acid etching has no effect on the roughness or topographical changes of resin composite.

1. Introduction

The prosthetic treatment of pulpless teeth with resin composite core and fiber posts has been widely used as an alternative to traditional metallic cast-and-cores [1]. The main benefit is its elastic modulus being closest to the root dentin, which allows better stress distribution of masticatory loads through the dental element avoiding catastrophic root fractures [2]. In addition, resin composite cores enable more esthetic results due to the possibility of choosing a material with a color similar to that of the tooth as opposed to dark metallic cores, which require opacifiers and thick restorations to be masked and avoid interference of the substrate in the final color of the restoration [3].

Lithium disilicate glass-ceramics are good options for crowns, combining excellent aesthetic properties and adequate mechanical strength [4]. The adhesive bonding of etchable glass-ceramics is an important key for the proper functional performance and longevity of the restoration-tooth complex [5]. Surface treatments and the adhesion to resin composite luting agents have been widely studied and present predictable results, being a highly important point for the restoration's success [6–8]. Likewise, the choice for resin composite cores can be another important benefit due to the improved bond strength between resin cement and resin composites [9]. These materials present similar characteristics and compositions; even so, it is important to note that the resin cores are submitted to different operative stages before the definitive cementation of the restoration, i.e. the preparation stages of the prosthetic abutment, impression, provisional cementation and finishing are necessary before the ceramic restoration can be definitively seated on the prosthetic core [10]. In this sense, previous studies have reported that the different resin composite core build-up surface treatments can modify the bond strength between the resin composite and the resin cement [10,11].

Resin composite surfaces generally become rougher when treated, and plays a remarkable role for the bond improvements of the resin composite to resin cements [7]. However, it is important to understand how this improvement can influence the mechanical behavior of ceramic restorations cemented on these substrates. In addition, it must be also taken into account that monolithic ceramic restorations are usually subjected to the oral environment with application of intermittent cyclical loads which mainly generate stress on the inner surface of the restoration [12,13]. Therefore, studies have been developed evaluating the influence of different ceramic restoration bonding surface treatments on its fatigue behavior, demonstrating the importance of improved adhesion to obtain favorable results in mechanical outcomes [7,14,15]. Nevertheless, these studies disregard the resin cement-substrate interface upon which the crown will be settled/bonded; therefore, little is known about the influence of the substrate surface treatment on the fatigue behavior of lithium disilicate restorations.

In view of the aforementioned issues, the present study aims to evaluate the influence of resin composite surface treatments for core build-up on the fatigue behavior of lithium disilicate glass-ceramic monolithic simplified restorations bonded to the resin composite substrate. The hypothesis is that: resin composite surface treatment can affect the fatigue findings.

2. Materials and Methods

The general description of the materials used in the present study, their manufacturers, main composition, and batch numbers are presented in Table 1.

2.1 Sample assembly and study design

A simplified geometry test based on previous studies was used to simulate the fatigue behavior of ceramic restorations for posterior teeth [16–18]. To do so, resin composite discs indicated for core build-up (Tetric N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) were prepared and submitted to the distinct surface treatments prior to adhesive cementation of lithium disilicate glass-ceramic discs (IPS e.Max CAD, Ivoclar Vivadent).

This study was designed to have 1 control group and 3 testing groups, considering distinct surface treatments of the resin composite substrate (n = 15) (Table 2):

- **Control group (CTRL)**: no surface treatment, with ultrasonic cleaning for 5 min (distilled water) only;
- **Bur group:** grinding with coarse diamond bur (3101G, 151 µm);
- **PA group**: acid etching (37% phosphoric acid, 15 s);
- **AA group:** air-abrasion with 45 µm aluminum oxide powder (10 s, 10 mm distance, 2.8 bars).

2.2 Sample preparation

2.2.1 Ceramic discs

Lithium disilicate glass-ceramic blocks (IPS e.Max CAD HT A2, Ivoclar Vivadent) were shaped into cylinders with silicon carbide papers (600- and 1200-grit) (3M, Sumaré, Brazil) under water-cooling (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA). Next, slices were obtained in a cutting machine under water-cooling (Isomet 1000, Buehler). The discs were polished with 600- and 1200-grit silica carbide papers (3M) to remove surface irregularities inherent to the cutting and later crystallized (Vacumat 6000 MP, Vita Zahnfabrik, Bad Sackingen, Germany) as recommended. The final dimensions of the ceramic discs were 10 mm in diameter and 1 mm in thickness.

2.2.2 Resin composite discs

A total of 60 resin composite discs (Tetric N-Ceram, Ivoclar Vivadent) were prepared using a metallic matrix (Ø= 10 mm; thickness= 2 mm). An increment (± 2 mm) was inserted using a #1 spatula (Golgran, São Caetano do Sul, Brazil), and a polyester strip was subsequently positioned over the increment and compressed using a glass slide to obtain a flat surface. Then, the resin composite was light-activated for 20 s (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia) through the glass. The cementation surface of the discs was ground with 120-grit silicon carbide paper (3M) applied under light digital pressure with circular movements by a single trained operator (ACCR) in two rotatory movements (clockwise and counterclockwise) to simulate the roughness of a coarse diamond bur commonly used for dental preparations [11]. A provisory cement (Temp-Bond NE, Kerr Corporation, Romulus, USA) was mixed according to the manufacturer's instructions (equal lengths of base and accelerator mixed for approximately 30 s) and applied onto the surface of the resin composite discs. The discs were stored in distilled water for 14 days (37 ºC) to simulate the period between dental preparation and final cementation of the ceramic restoration.

2.3 Surface treatments of the resin composite

The provisory cement was removed from the resin composite surface with manual instruments, the surface was cleaned with pumice using a low-speed handpiece (Kavo Dental, Biberach, Germany) and subsequently submitted to an ultrasonic bath (Vitasonic, Vita Zahnfabrik, Bad Sackingen, Germany) with distilled water for 5 min. Next, the surface treatments were performed as described in Table 2.

2.4 Roughness analysis

The treated surfaces of all resin composite specimens (N= 60) were analyzed through a surface roughness tester (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Japan) to determine the surface roughness according to the International Organization for Standardization [19]. The arithmetic means of four surface roughness measurements (two in the grinding direction and two in the opposite direction) were calculated for each sample and the Ra parameter was obtained (cut-off of 5; λC of 0.8 mm; λS of 2.5 µm). Ra is defined as the arithmetic mean of the absolute values of peaks and valleys measured from a mean plane (in µm).

2.5 Cementation procedure

After the surface treatments of the resin composite and ceramic discs (Table 2), a dual-cure resin cement (Multilink N, Ivoclar Vivadent) was manipulated and applied onto the treated ceramic surface, followed by settlement on the corresponding resin composite disc under constant load of 2.5 N applied for 10 min on the occlusal surface of ceramic disc. The resin cement excesses were removed with a microbrush and light-activation (1200 mW/cm^2, Radii-Cal, SDI) was performed for five exposures of 20 s each (0º, 90º, 180º, 270º and on the top surface). All the cemented specimens were stored in distilled water (12 days up to 18 days; 37 ºC) until the fatigue test.

2.6 Step-stress fatigue test

The fatigue failure load was established using the Step-stress method on a mechanical testing machine (INSTRON Electropuls E3000, Instron Corporation, Norwood, USA). Each sample was stabilized on a flat steel base under water and loaded by a hemispherical stainless-steel piston with 40 mm in diameter, positioned in the center of the ceramic's occlusal surface. An adhesive tape (110 μm) was placed on the occlusal surface of each specimen before testing to reduce contact stress concentration and to prevent contact surface damage [20].

Load pulses were applied at a frequency of 20 Hz [21] for 10,000 cycles at each load increment (100 N). The test started with an initial load of 200 N (5.000 cycles) to adjust the sample/piston contact followed by crescent load steps (400 N, 500 N, 600 N, 700 N and up to its failure). The specimens were checked for cracks every 10,000 cycles by light oblique transillumination [22]. A cylindrical metal ring (internal diameter ≅ 10 mm allowing passive insertion) was attached to the steel base ensuring that the sample was always positioned in the same place after checking and that the load was applied to the center of the ceramic disc. The radial crack was considered as the main outcome and the fatigue test was ended once detected for this sample. The failure data (fatigue failure load – FFL, and cycles for fatigue failure – CFF) of each sample were recorded for data analysis.

2.7 Fractographic analysis

All failed specimens were inspected by oblique light transillumination after testing and in a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) at 10× magnification to visually analyze for initial cracks. For this, the ceramic fragments were detached to expose the fractured surface. Next, one representative specimen per group was selected and submitted to Scanning Electron Microscopy (SEM, JSM-6360, JEOL, Tokyo, Japan) (Acceleration Voltage: 15.00 kV; Detector: SE – Secundary electron) at 50× magnifications to illustrate the failure origin and characterize the fractographic features.

2.8 Contact angle analysis, fractal dimension and topographic analysis

Additional treated resin composite specimens (n= 4) were produced to perform these analyses. The contact angle test was performed in using a goniometer (Drop Shape analysis, model DSA 30S, Kruss GmbH, Hamburg, Germany) connected to a computer software program (DSA3, V1 .0.3-08, Kruss) via the sessile drop technique. One drop of distilled water (11 μl) was dropped using a needle at the center of the specimen. The mean of five measurements per specimen was registered after 5 s.

The topography pattern was analyzed (n= 1) by Atomic Force Microscopy (AFM - Park Systems NX10 equipment, Suwon, Korea), with the surface area and fractal dimension being obtained through analyzing the AFM micrographs. The analysis was performed through the non-contact mode and using a reflective aluminum backside-coated highly doped monolithic silicon probe (Nanosensors PPP-NCHR, Neuchâtel, Switzerland). An area of 10 × 10 μm was considered and the box-counting method was used

[23]. AFM data analysis was performed using Park XEI software (version 4.3.4 Build22.RTM1). The fractal dimension was obtained at five different regions of each specimen followed by evaluating the average of all these measurements. The topographic pattern was also evaluated through stereomicroscopy (Zeiss Stemi SV6; Carl Zeiss, Jena, Germany) at 7.5× and SEM topographical analysis (JSM-6360, JEOL, Tokyo, Japan) at 100× magnification.

2.9 Data analysis

A statistical software program (IBM SPSS Software; IBM, Armonk, NY, USA) was used with a significance level of 0.05. The FFL and CFF data were subjected to a Kaplan Meier analysis with post-hoc Mantel-Cox (log-rank) test ($\alpha= 0.05$), and the survival probability was tabulated for each step of the test.

Fractographic and topographic analysis were qualitatively evaluated. No statistical analysis was performed for the fractal dimension, because an increase in 10% in the fractal dimension causes the surface stiffness to decrease by more than one order of magnitude in solid surfaces [24]. One-way ANOVA and Tukey's test ($\alpha= 0.05$) were performed for contact angle data.

3. Results

No statistical difference ($P> 0.05$) was found among the groups regarding the fatigue behavior (Table 3). Also, similar survival rates could be found (Table 4 and Figure 1).

The fractographic analysis (Figure 2) shows radial cracks initiating at the bonding surface of the ceramic discs. The crack progression zone is evident through the semi-circular signs in the upper half of the samples.

The bur group presented the highest surface roughness value, followed by the AA group (Bur > AA > PA = Ctrl) (Table 3). AA presented the more complex surface for the fractal dimension (higher fractal dimension), followed by the Ctrl, Bur and PA groups, respectively (Table 3).

The surface alterations induced by grinding with the bur can be visually seen under a small magnification (7.5×), while the microretentions created by the air-abrasion are visible under SEM (100×) (Figure 3). The etching with 37% phosphoric acid led to no surface change. The contact angle measurement shows statistically significant differences, with the highest average for the Bur groups (Bur > Ctrl > PA = AA) (Figure 3).

4. Discussion

The hypothesis was rejected since the surface treatments of the resin composite had no statistically significant effect on the fatigue findings. Even with evident differences in terms of topographical alterations by surface treatment (Bur and AA groups), this 'disc-disc' test set-up under fatigue having ceramic up and resinous substrate down induces tensile stress peak mainly on the inner surface of the ceramic [16,25,26], with the surfaces subjected to the conditionings (resin composite) in this case receiving lower tensile stress, therefore, having no influence on the fatigue failure.

According to our fractographic analysis, the failures of the ceramics occurred from the cementation surface (Fig. 2), in accordance with what is described by the literature as clinically relevant outcome [8]. In a previous study, it was observed that the increase in roughness generated by grinding with diamond bur on an epoxy resin substrate generated better results in fatigue when compared to the polished substrate [18]. However, the same was not observed in the present study, where all groups had similar FFL, CFF and survival rates (Table 3 and 4, Figure 1). In the previous study, the control group was polished with low-grain silica carbide paper, while we chose to submit all groups to roughness simulation, mimicking the changes caused by the burs used in prosthetic core preparation [11]. In this case, it suggested that polished surfaces may have a negative influence on the mechanical behavior of bonded ceramics, and therefore studies with polishing protocols of the prosthetic core may be carried out for this purpose. In any case, we conclude that our unpolished substrate surfaces was enough to generate the necessary roughness to improve the adhesion between substrate and resin cement, which did not occur in the aforementioned study.

In terms of surface features, some differences could be found. For instance, the air-abrasion and grinding procedures promoted rougher resin composite surface, as well as remarkable surface micromorphological changes (Table 3, Fig 1). From the micromechanical standpoint, the increase in roughness is beneficial for resin interlocking and bonding improvements [9]. On the other hand, phosphoric acid appears to be inert on the resinous surface (no relevant surface topographic change), being indicated as a cleaner of the surface [27]. Even though no surface alterations could be found with acid etching, this cleaning was relevant when considering the contact angle data (Fig. 1), which refers to wettability: ability of fluids to spread on the solid surface [28]. Thus, adhesive systems are expected to have good spreading for better bond strength, and therefore the use of phosphoric acid may have been important in this regard. However, it should be noted that the contact angle measurements as performed without roughness corrections. Rougher surfaces modify wettability as they are susceptible to trapping air into deep valleys [29]. This effect can be observed in the Bur group, where the defects created by the diamond bur resulted in a high contact angle due to the negative wettability effect. However, we must take into consideration the benefits of a rough surface for bond strength [9], where there is greater adhesive area and the possibility of micromechanical retention of the cementing agent. Therefore, the wettability data must be evaluated with caution.

The fractal dimension quantitatively provides the complexity of the adhesive surface after the evaluated protocols [24]. The AA group presented the highest value, since the abrasion with the aluminum oxide particles leads to a surface modification by the impact of particles making the surface more complex. In turn, the Bur group’s mean was similar to the Ctrl group due to the repetitive pattern generated by the diamond bur, which results in similar values to those obtained by grinding simulation made with abrasive silica carbide paper in all groups before provisory cementation.

Not having an as-mirror polished condition (no topographic changes) as a negative control can be pointed out as a limitation of our study, even though our target was to evaluate the surface treatments for which the resin composite can be clinically subjected to (leading to micromorphological changes), inspecting its effects on the fatigue performance of bonded glass-ceramics [11]. From this viewpoint, our study indicates that the alumina particles air-abrasion and grinding with diamond bur of the resin composite leads to relevant topographical changes, but does not have an influence on the fatigue behavior of tested glass-ceramic when bonded onto the substrate. Another limitation is the absence of the sample calculation. However, the calculation based on the averages obtained in our studies suggests 1216 samples per group to detect a statistical difference among conditions considering our experimental design, which is unfeasible for laboratory studies like this one, and points to the scarce probability to observe the influence of such factor on clinical practices. Finally, thermal aging could have been considered in our study. Anyway, we still consider our findings relevant even without considering aging procedures.

5. Conclusion

- The assessed surface treatments of resin composite promotes topographical changes; however, they have no influence on the fatigue behavior of bonded lithium disilicate glass-ceramic restorations.

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REFERENCES

[1] Marchionatti AME, Wandscher VF, Rippe MP, Kaizer OB, Valandro LF. Clinical performance and failure modes of pulpless teeth restored with posts: a systematic review. Braz Oral Res 2017;31:e64. https://doi.org/10.1590/1807-3107BOR-2017.vol31.0064.

[2] Santos AFV, Meira JBC, Tanaka CB, Xavier TA, Ballester RY, Lima RG, et al. Can fiber posts increase root stresses and reduce fracture? J Dent Res 2010;89:587–91. https://doi.org/10.1177/0022034510363382.

[3] Bacchi A, Boccardi S, Alessandretti R, Pereira GKR. Substrate masking ability of bilayer and monolithic ceramics used for complete crowns and the effect of association with an opaque resin-based luting agent. J Prosthodont Res 2019;63:321–6. https://doi.org/10.1016/j.jpor.2019.01.005.

[4] da Silva LH, de Lima E, Miranda RB de P, Favero SS, Lohbauer U, Cesar PF. Dental ceramics: A review of new materials and processing methods. Braz Oral Res 2017;31:133–46. https://doi.org/10.1590/1807-3107BOR-2017.vol31.0058.

[5] Scherrer SS, de Rijk WG, Belser UC, Meyer JM. Effect of cement film thickness on the fracture resistance of a machinable glass-ceramic. Dent Mater 1994;10:172–7. https://doi.org/10.1016/0109-5641(94)90028-0.

[6] Prochnow C, Venturini AB, Grasel R, Gundel A, Bottino MC, Valandro LF. Adhesion to a lithium disilicate glass ceramic etched with hydrofluoric acid at distinct concentrations. Braz Dent J 2018;29:492–9. https://doi.org/10.1590/0103-6440201802080.

[7] Dapieve KS, Machry RV, Pilecco RO, Kleverlaan CJ, Rocha Pereira GK, Venturini AB, et al. One-step ceramic primer as surface conditioner: Effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations. J Mech Behav Biomed Mater 2020;104:103686. https://doi.org/10.1016/j.jmbbm.2020.103686.

[8] Nogueira I de O, Oliveira PFG de, Magno MB, Ferreira DMTP, Maia LC, Rabello TB. Does the application of an adhesive layer improve the bond strength of etched and silanized glass ceramics to resin-based materials? A systematic review and meta-analysis. J Prosthet Dent 2020:1–9. https://doi.org/10.1016/j.prosdent.2019.12.005.

[9] Machry R V., Fontana PE, Bohrer TC, Valandro LF, Kaizer OB. Effect of Different Surface Treatments of Resin Relined Fiber Posts Cemented With Self-adhesive Resin Cement on Push-out and Microtensile Bond Strength Tests. Oper Dent 2020;45:E185–95. https://doi.org/10.2341/19-108-L.

[10] Klosa K, Shahid W, Aleknonyte-Resch M, Kern M. Cleaning and conditioning of contaminated core build-up material before adhesive bonding. Materials (Basel) 2020;13:1–15. https://doi.org/10.3390/ma13122880.

[11] Cotes C, Cardoso M, de Melo RM, Valandro LF, Bottino MA. Effect of composite surface treatment and aging on the bond strength between a core build-up composite and a luting agent. J Appl Oral Sci 2015;23:71–8. https://doi.org/10.1590/1678-775720140113.

[12] Kelly JR, Rungruanganunt P, Hunter B, Vailati F. Development of a clinically validated bulk failure test for ceramic crowns. J Prosthet Dent 2010;104:228–38. https://doi.org/10.1016/S0022-3913(10)60129-1.

[13] Bonfante EA, Coelho PG. A Critical Perspective on Mechanical Testing of Implants and Prostheses 2016. https://doi.org/10.1177/0022034515624445.

[14] Prochnow C, Pereira GKR, Venturini AB, Scherer MM, Rippe MP, Bottino MC, et al. How does hydrofluoric acid etching affect the cyclic load-to-failure of lithium disilicate restorations? J Mech Behav Biomed Mater 2018;87:306–11. https://doi.org/10.1016/j.jmbbm.2018.07.040.

[15] Fraga S, de Jager N, Kleverlaan CJ, Campos F, Valandro LF. Does luting strategy affect the fatigue behavior of bonded Y-TZP ceramic? J Adhes Dent 2018;20:307–15. https://doi.org/10.3290/j.jad.a40988.

[16] Chen C, Trindade FZ, De Jager N, Kleverlaan CJ, Feilzer AJ. The fracture resistance of a CAD/CAM Resin Nano Ceramic (RNC) and a CAD ceramic at different thicknesses. Dent Mater 2014;30:954–62. https://doi.org/10.1016/j.dental.2014.05.018.

[17] de Kok P, Pereira GKR, Fraga S, de Jager N, Venturini AB, Kleverlaan CJ. The effect of internal roughness and bonding on the fracture resistance and structural reliability of lithium disilicate ceramic. Dent Mater 2017;33:1416–25. https://doi.org/10.1016/j.dental.2017.09.018.

[18] Cadore-Rodrigues AC, Machry RV, Burgo TA de L, Venturini AB, May LG, Valandro LF. Fatigue behavior of bonded lithium disilicate glass-ceramic simplified restorations is not damaged by the finishing/grinding of the bonding surface of dentin analogue material. Int J Adhes Adhes 2021;107:102824. https://doi.org/10.1016/j.ijadhadh.2021.102824.

[19] ISO 4287, 1997. Geometrical Product Specifications (GPS). International Organization for Standardization n.d.

[20] Kelly JR. Clinically relevant approach to failure testing of all-ceramic restorations. J Prosthet Dent 1999;81:652–61. https://doi.org/10.1016/S0022-3913(99)70103-4.

[21] Velho HC, Dapieve KS, Pereira GKR, Fraga S, Valandro LF, Venturini AB. Journal of the Mechanical Behavior of Biomedical Materials Accelerated loading frequency does not influence the fatigue behavior of polymer infiltrated ceramic network or lithium disilicate glass-ceramic

restorations. J Mech Behav Biomed Mater 2020;110:103905. https://doi.org/10.1016/j.jmbbm.2020.103905.

[22] Dibner AC, Kelly JR. Fatigue strength of bilayered ceramics under cyclic loading as a function of core veneer thickness ratios. J Prosthet Dent 2016;115:335–40. https://doi.org/10.1016/j.prosdent.2015.09.017.

[23] Schestatsky R, Zucuni CP, Venturini AB, de Lima Burgo TA, Bacchi A, Valandro LF, et al. CAD-CAM milled versus pressed lithium-disilicate monolithic crowns adhesively cemented after distinct surface treatments: Fatigue performance and ceramic surface characteristics. J Mech Behav Biomed Mater 2019;94:144–54. https://doi.org/10.1016/j.jmbbm.2019.03.005.

[24] Buzio R, Boragno C, Biscarini F, Buatier De Mongeot F, Valbusa U. The contact mechanics of fractal surfaces. Nat Mater 2003;2:233–6. https://doi.org/10.1038/nmat855.

[25] Machry R V, Cadore-Rodrigues AC, Luiz A, Borges S, Pereira GKR, Kleverlaan CJ, et al. Fatigue resistance of simplified CAD – CAM restorations : Foundation material and ceramic thickness effects on the fatigue behavior of partially- and fully-stabilized zirconia. Dent Mater 2021. https://doi.org/10.1016/j.dental.2021.01.001.

[26] Pereira GKR, Graunke P, Maroli A, Zucuni CP, Prochnow C, Valandro LF, et al. Lithium disilicate glass-ceramic vs translucent zirconia polycrystals bonded to distinct substrates: Fatigue failure load, number of cycles for failure, survival rates, and stress distribution. J Mech Behav Biomed Mater 2019;91:122–30. https://doi.org/10.1016/j.jmbbm.2018.12.010.

[27] Fawzy AS, El-Askary FS, Amer MA. Effect of surface treatments on the tensile bond strength of repaired water-aged anterior restorative micro-fine hybrid resin composite. J Dent 2008;36:969–76. https://doi.org/10.1016/j.jdent.2008.07.014.

[28] Marshall SJ, Bayne SC, Baier R, Tomsia AP, Marshall GW. A review of adhesion science. Dent Mater 2010;26:11–6. https://doi.org/10.1016/j.dental.2009.11.157.

[29] Wenzel R. Resistance of solid surfaces to wetting by water. Ind Eng Chem 1936;28:988–994. https://doi.org/10.1017/cbo9781316146743.

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, Schaan, Liechtenstein (LOT W89815)	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 oxides 0-12 wt%
Resin composite	Tetric N-Ceram, Ivoclar Vivadent (LOT Z009GG)	Dimethacrylates, fillers (barium glass, ytterbium trifluoride, mixed oxide and copolymers), additives, catalysts, stabilizers and pigments. Particle size: 40-3000 nm
Temporary cement	Temp Bond NE, Kerr Corporation, Romulus, USA (LOT 5-1166)	Zinc Oxide and poly organo acids
Resin cement	Multilink N, Ivoclar Vivadent (LOT Z0074R)	Dimethacrylates, HEMA, barium glass filler, Ba-Al-Fluoro-Silicate glass, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments
5% Hydrofluoric acid	Condac porcelana 5%, FGM, Joinville, Brazil (LOT 020819)	Hydrofluoric acid at 5%, water, thickener, surfactant and colouring
37% Phosphoric acid	Condac 37%, FGM, Joinville, Brazil (LOT 050919)	Phosphoric acid at 37%, thickener, pigment and deionized water
Diamond bur	3101G, KG Sorensen, Cotia, Brazil (LOT 14763)	Stainless steel and diamond grains (particle size: 151 µm)
Aluminum oxide	Polidental, Cotia, Brazil (LOT 44493)	Aluminum Oxide. Particle size: 45 µm
Coupling agent	Monobond N, Ivoclar Vivadent (LOT Y19262)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate

Table 2. Surface treatments for ceramic material and finishing procedures for resin composite substrate.

	Lithium disilicate	Resin composite
Surface treatments	Etching with 5% hydrofluoric acid 20 s, washing 30 s, ultrasonic cleaning 5 min (distilled water).	**Ctrl**: no surface treatment; ultrasonic cleaning 5 min (distilled water) only.
		Bur: Grinding using diamond burs (3101G; changed every 2 specimens) with a contra-angle handpiece (T2 REVO R170 up to 170,000 rpm, Sirona, Bensheim, Germany) coupled to a low speed motor (Kavo Dental, Biberach, Germany) under constant water cooling (~30mL/min) according to the protocol described by Cadore-Rodrigues el al. (2021). Ultrasonic cleaning 5 min (distilled water) [18].
		PA: Etching with 37% phosphoric acid for 15 s, washing 30 s, ultrasonic cleaning 5 min (distilled water).
		AA: Air-abrasion with aluminum oxide particles (45 µm, 10 mm distance, 2.8 bars) for 10 s and air-jet cleaning.
Adhesive Strategy	A primer containing multiple bond promoters (Monobond N, Ivoclar Vivadent) was scrubbed on the bonding surfaces of the treated ceramic discs for 15 s and kept reacting for 45 s.	Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the treated surfaces of resin composite discs for 30 s and air-dried until a thin layer was obtained.

Table 3. Results from the survival of fatigue data (Fatigue Failure Load – FFL, Cycles for fatigue failure – CFF), depicting mean and 95% confidence intervals (CI), and data from surface roughness analysis (µm – Ra parameter, mean and standard deviation - SD), mean and standard deviation (SD) of surface area (µm^2) and fractal dimension values estimated by the box counting (D_{BC}) method.

Group	FFL (Newton)*	CFF*	Surface Roughness (Ra)**	AFM analysis	
				Surface area (µm²)	D_{BC}
Ctrl	840 (774.2 – 905.7) A	58047 (51840 – 64254) A	0.641 (0.25) C	25.628 (0.26)	2.154 (0.03)
Bur	860 (818.1 – 901.9) A	59960 (55552 – 64368) A	5.731 (0.74) A	28.630 (3.18)	2.150 (0.06)
PA	867 (768.7 – 871.3) A	61090 (55635 – 66544) A	0.910 (0.37) C	26.205 (0.46)	2.131 (0.03)
AA	820 (818.9 – 874.4) A	53195 (44837 – 61553) A	2.529 (0.47) B	30.967 (3.97)	2.170 (0.02)

* The same letters in each column indicate no statistically significant differences (P < .05) using Kaplan Meier followed by Mantel-cox post-hoc test for FFL and CFF.
** Different letters in each column indicate statistically significant differences (P < .05) depicted by one-way ANOVA and Tukey post-hoc test for Surface Roughness and Surface area.

Table 4. Survival probabilities for fatigue failure load (FFL) and number of cycles for failure (CFF) with respective standard errors.

Group	Fatigue failure load (FFL)								
	400N	**500N**	**600N**	**700N**	**800N**	**900N**	**1,000N**	**1,100N**	**1,200N**
Ctrl	1	1	1	0.67 (0.12)	0.47 (0.13)	0.20 (0.06)	0.07 (0.06)	0.0	-
Bur	1	1	1	0.93 (0.06)	0.53 (0.13)	0.13 (0.09)	0.0	-	-
PA	1	1	1	0.93 (0.07)	0.40 (0.13)	0.20 (0.10)	0.07 (0.06)	0.07 (0.06)	0.0
AA	1	1	0.93 (0.07)	0.80 (0.10)	0.40 (0.13)	0.07 (0.06)	0.0	-	-
	Number of cycles for failure (CFF)								
	15 x 10^3	**25 x 10^3**	**35 x 10^3**	**45 x 10^3**	**55 x 10^3**	**65 x 10^3**	**75 x 10^3**	**85 x 10^3**	**95 x 10^3**
Ctrl	1	1	1	0.67 (0.12)	0.47 (0.13)	0.20 (0.10)	0.07 (0.06)	0.0	-
Bur	1	1	1	0.93 (0.06)	0.47 (0.13)	0.13 (0.09)	0.0	-	-
PA	1	1	1	0.93 (0.07)	0.40 (0.13)	0.20 (0.10)	0.07 (0.06)	0.07 (0.06)	0.0
AA	1	1	0.93 (0.07)	0.80 (0.10)	0.47 (0.13)	0.07 (0.06)	0.0	-	-

* Kaplan Meier and Mantel-Cox tests.
** The symbol '-' indicates absence of specimens being tested on the respective step.

FIGURES

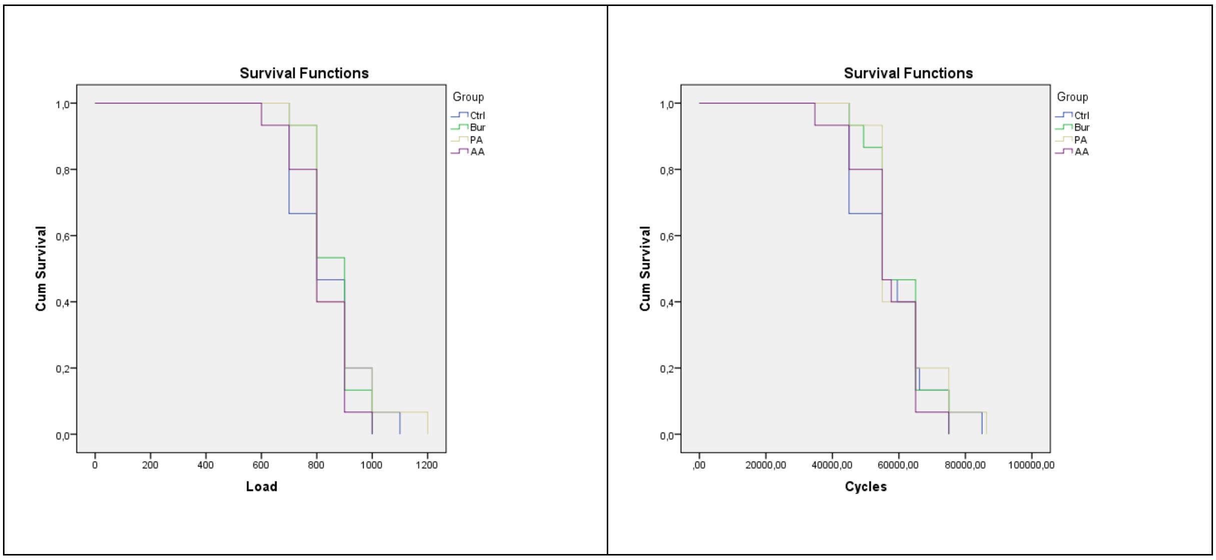
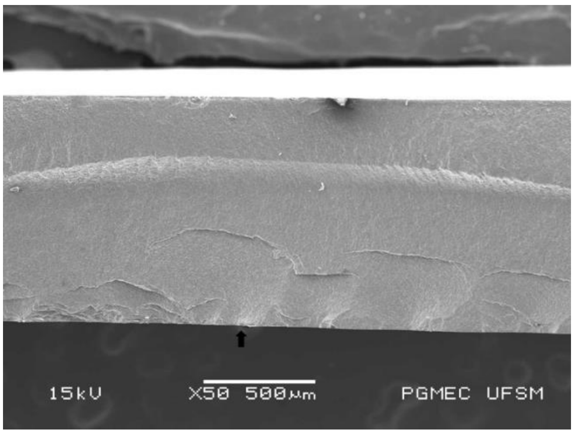
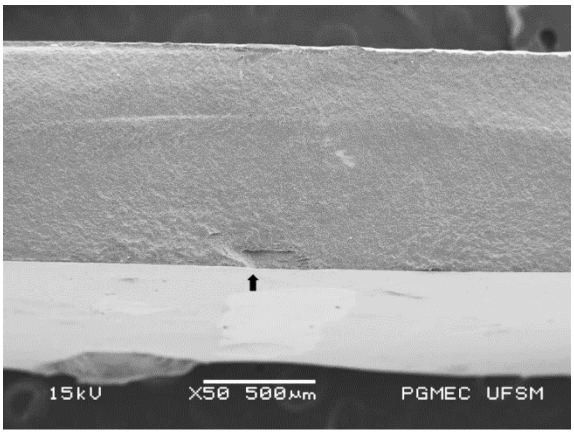


Figure 1. Survival curves according to the steps of fatigue failure load (FFL; left) and number of cycles (right) in which each disc failed obtained by Kaplan–Meier and Log-rank tests.

Ctrl	Bur
	
PA	**AA**

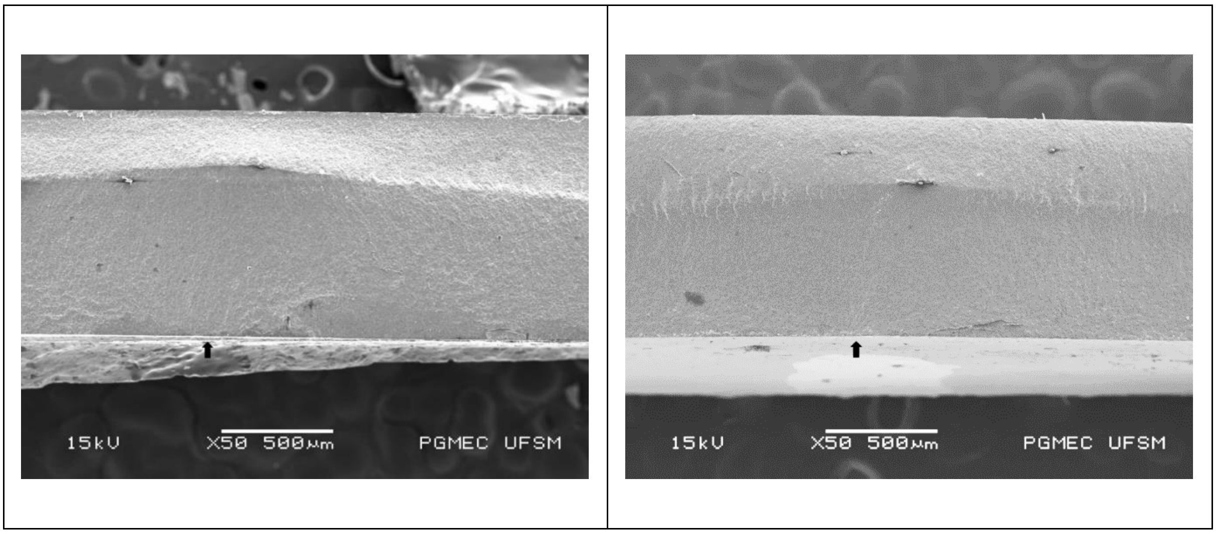
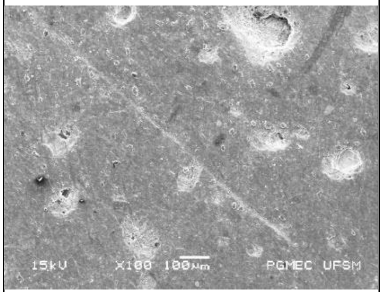
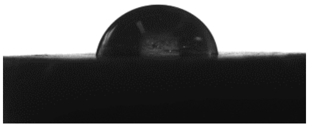
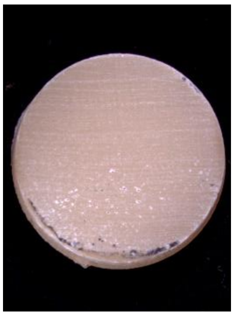
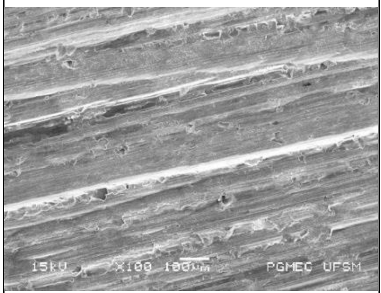
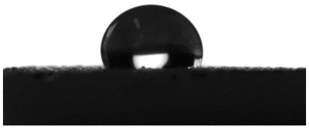
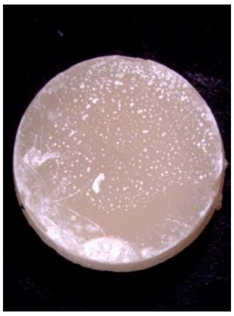
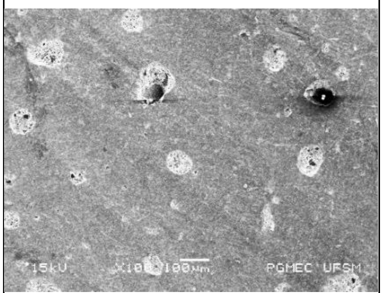
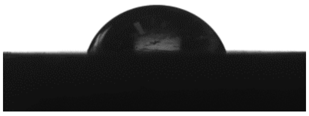
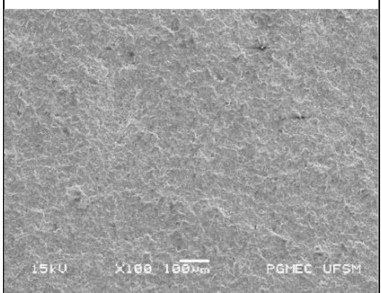
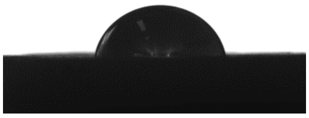


Figure 2. Representative SEM images of fractographic analysis at 50× magnification. The arrows indicate the failure origin on the cementation surface of the ceramic disc (region subjected to tensile stresses during the fatigue test). In addition, compression zones are observable at the top of the ceramic disc and the crack progression zone is evident through the semi-circular signs in the upper half of the samples.

Group	Topographic Images		Contact Angle	
	Stereomocroscopy (7.5×)	**SEM (100×)**	**Images**	**Mean (SD)***
Ctrl				92.92 (3.43) B
Bur				102.32 (4.74) A
PA				78.57 (4.14) C
AA				83.42 (4.49) C

*Different letters indicate statistically significant differences ($P < .05$) (one-way ANOVA and Tukey post-hoc tests).

Figure 3. Representative topographic images in stereomicroscopy (7.5×) and scanning electron microscopy (SEM; 100×). Representative images and mean (standard deviation - SD) of the contact angle measurement. The topographic images show a superficial pattern with macro- and micro-visible differences after the finishing protocols evaluated. The surface roughness of the Bur group leads to an increased contact angle, indicating less wettability. Smaller contact angles were obtained in the PA and AA groups.

5 DISCUSSÃO

O suporte adequado das restaurações cerâmicas monolíticas é imprescindível no reforço estrutural desses materiais, uma vez que são reconhecidamente resistentes à compressão, mas susceptíveis à falha em tração (DENRY; KELLY, 2008; KWON et al., 2018). No caso de coroas totais em prótese fixa, a zona submetida a tensões de tração está principalmente localizada na face interna da restauração (superfície de cimentação), na região imediatamente oposta a aplicação das cargas mastigatórias situada na face oclusal (KELLY, 1999). Ao longo dos anos em função, as restaurações são submetidas a cargas cíclicas e, nesse contexto, desfechos catastróficos podem ser iniciados a partir de falhas pré-existentes decorrentes do processo de usinagem, ajustes internos e externos e até mesmo pelo condicionamento da superfície para cimentação adesiva (CADORE-RODRIGUES et al., 2021b; PILECCO et al., 2021; PROCHNOW et al., 2018; RODRIGUES et al., 2018; ZUCUNI, et al., 2017). Mecanicamente, é possível compreender uma zona de compressão na área sendo submetida ao carregamento intermitente, causando a flexão da restauração e, como efeito, o deslocamento da zona oposta pela abertura repetitiva dos defeitos existentes nessa área e progressão das trincas através da cerâmica.

O fenômeno de formação e crescimento lento e subcrítico de trincas gera valores de resistência inferiores aos atribuídos por meio de ensaios mecânicos estáticos (BONFANTE; COELHO, 2016). Diversos estudos têm sido conduzidos para compreender a influência de aspectos que geram alteração topográfica na superfície de cimentação no comportamento em fadiga das cerâmicas monolíticas (CADORE-RODRIGUES et al., 2021b; DE KOK et al., 2017; PILECCO et al., 2021; PROCHNOW et al., 2018; RODRIGUES et al., 2018; ZUCUNI, et al., 2017). Entretanto, as metodologias aplicadas utilizam resinas compostas, dentes ou materiais análogos de dentina (resina epóxi) como substratos que, devido ao baixo módulo elástico desses materiais, tendem a sofrer deformação elástica durante a aplicação de carga. Em contrapartida, cerâmicas são materiais friáveis que suportam pouca ou nenhuma deformação (DA SILVA et al., 2017; SCHERRER et al., 2017). A conjunção de comportamentos distintos leva a concentração de tensões na região da interface adesiva e, consequentemente, falhas classificadas como trincas radiais, sendo essas reconhecidamente umas das principais origens das fraturas das restaurações cerâmicas monolíticas (KELLY, 2008).

Por outro lado, é possível que clinicamente as coroas totais sejam cimentadas sobre núcleos metálicos constituídos por ligas fundidas de alto módulo de elasticidade. Em oposição ao comportamento dos materiais resinosos, esses metais necessitam de cargas altas para sofrer algum grau de deformação. Sendo assim, impedem a flexão da cerâmica e, consequentemente, aumentam a capacidade da restauração de resistir à solicitação mecânica (CHEN et al., 2019; FACENDA et al., 2019; MACHADO et al., 2021). Nos dois primeiros estudos que compõem essa tese, foram encontrados melhores resultados mecânicos das cerâmicas odontológicas avaliadas quando cimentados sobre substratos metálicos comparado a substratos resinosos, o que corrobora o conceito levantado de que há influência positiva do maior módulo de elasticidade do substrato no comportamento em fadiga das cerâmicas odontológicas.

Tendo em vista esses resultados, pode-se afirmar que evitar a flexão da coroa protética através do suporte de substratos com maior módulo de elasticidade diminui a formação de tensões de tração na sua superfície interna. No entanto, é ainda importante considerar que a similaridade de módulo elástico entre os materiais (restauração e substrato) permite melhor distribuição das tensões ao longo do conjunto (DAPIEVE et al., 2021). No segundo estudo, a cerâmica reforçada por uma rede polimérica (*Polymer-Infiltrated Ceramic Network* – PICN) apresentou comportamento similar ao dissilicato de lítio e à zircônia de terceira geração quando cimentado a um material análogo de dentina (resina epóxi reforçado por fibra de vidro). Sabidamente, tanto a cerâmica vítrea quanto a policristalina apresentam resistência flexural superior ao PICN (DELLA BONA; CORAZZA; ZHANG, 2014; FACENDA; BORBA; CORAZZA, 2018; VENTURINI et al., 2019b), no entanto, a menor discrepância entre o módulo de elasticidade da restauração cerâmico-polimérica e o substrato resinoso favoreceu o comportamento do material restaurador frente as cargas cíclicas (FACENDA et al., 2019). Esse achado é corroborado pela análise de elemento finitos do estudo, no qual fica evidente a menor tensão na superfície de cimentação do PICN e, portanto, menor solicitação estrutural do material nessa condição.

Ainda sobre a comparação entre materiais restauradores, o estudo um avaliou também duas gerações de zircônia em diferentes espessuras. A classificação em gerações atribui-se às modificações microestruturais que foram executadas ao longo dos anos com intuito de melhorar as propriedades ópticas do material (STAWARCZYK et al., 2017). Através da incorporação de maior quantidade de ítria na composição foi possível a estabilização dos cristais de zircônia na forma cúbica (ZHANG, Y.; LAWN, 2018). Entretanto, houve uma redução na resistência desses materiais atribuída a incapacidade de transformação de fase na presença de trincas (mecanismos de tenacificação), que fez com que essa geração de zircônia fosse chamada de *fully-stabilized* (completamente estabilizada) por alguns autores (CADORE-RODRIGUES et al., 2020; CARDOSO et al., 2020; PEREIRA et al., 2018; SULAIMAN et al., 2017; ZUCUNI; PEREIRA; VALANDRO, 2020). Embora ainda possa ser sugerido que ocorra alguma transformação de fase mesmo na presença de cristais na forma cúbica, considera-se que o mecanismo de tenacificação seja bastante reduzido (MAO et al., 2018). De fato, os resultados do primeiro estudo comprovam o pior comportamento mecânico em fadiga da zircônia com maior quantidade de ítria quando dados do mesmo material de substrato e espessura da restauração foram avaliados.

Além do módulo de elasticidade, é de se esperar que haja diferença na resistência de união entre o cimento resinoso utilizado na cimentação adesiva e os diferentes materiais de substratos selecionados para os dois primeiros estudos, devido a questões de propriedades química e mecânica inerentes a cada um deles (AL-HARBI et al., 2016; JAMEL; NAYIF; ABDULLA, 2019; SABATINI; PATEL; D'SILVA, 2013; TSUJIMOTO et al., 2017). No entanto, a avaliação isolada dessas variáveis dentro do conjunto complexo de fatores que envolve a comparação entre eles é complexa. Por sua vez, mesmo para cada um desses substratos são possíveis diferentes protocolos podem ser clinicamente adotados no que diz respeito ao preparo do núcleo protético e tratamento de superfície. Nesse sentido, Cadore-Rodrigues et al. (2021) avaliaram diferentes pontas diamantadas de preparo e acabamento utilizados no preparo do material análogo de dentina. Entretanto, os resultados demonstraram não haver interferência no comportamento em fadiga da restauração cerâmica cimentadas sobre eles entre os protocolos testados, embora todas tenham apresentado resultados superiores quando comparados ao grupo controle polido (CADORE-RODRIGUES et al., 2021a).

No que diz respeito aos núcleos protéticos confeccionados com resina composta, Cotes et al. (2015) concluíram que aqueles que são asperizados com uma ponta diamantada antes do processo de cimentação melhoram a resistência de união com o cimento resinoso. O estudo avaliou diferentes métodos de finalização da resina composta do núcleo simulando o preparo do substrato após a remoção da restauração provisória (COTES et al., 2015). Assim sendo, o terceiro estudo dessa tese buscou aprimorar esses achados transladando esse conhecimento para o ponto de vista mecânico. Restaurações monolíticas de dissilicato de lítio foram adesivamente cimentadas sobre substratos de resina composta submetidos a diferentes protocolos de preparo e finalização da superfície pra cimentação adesiva e os protocolos avaliados geraram características distintas na resina composta. No entanto, não foram encontradas diferenças no comportamento mecânico das restaurações nos resultados de fadiga. Durante a incidência das cargas na face oclusal da cerâmica, a concentração de tensões ocorre sobretudo na sua superfície de cimentação. O estresse é dissipado pelo cimento resinoso e pouco ocorre na interface adesiva com o substrato que, portanto, tem pouca influência no desempenho mecânico do conjunto.

Tendo em vista o exposto, parece lícito afirmar que no que diz respeito aos diferentes materiais de substrato, o comportamento da cerâmica monolítica está principalmente atrelado as diferenças de módulo de elasticidade entre eles. Ainda assim, é preciso considerar que os estudos que compõem essa tese foram executados a partir de uma metodologia de restauração simplificada, isto é, discos cerâmicos cimentados adesivamente sobre substratos também em formato de disco. Essa condição de ensaio foi desenvolvida com o objetivo de mimetizar restaurações monolíticas em dentes molares, levando em consideração pontualmente a distribuição de tensões ocorrendo na interface de cimentação (CHEN, C. et al., 2014). A partir dessa distribuição de tensões localizada, busca-se simular trincas radiais originadas a partir dessa região, tendo sido esse o desfecho considerado nos três estudos. Porém, entende-se que clinicamente as coroas monolíticas pode apresentar outras origens de falha catastrófica, podendo ser falhas oriundas de defeitos da superfície oclusal das restaurações (denominadas falhas em cone, ou *Hertzian cone cracks*) decorrente de defeitos de processamento do material, desgaste da superfície oclusal realizado pelo cirurgião-dentista devido a ajustes dos pontos de contatos, efeito da carga cíclica intermitente da mastigação provocando fadiga e deslizamento da cúspide antagonista gerando uma zona de tensão mecânica durante o carregamento; e lascamento de regiões não suportadas pelo substrato devido particularidade anatômicas das restaurações (LAWN et al., 2004; SCHERRER et al., 2007; SORNSUWAN; ELLAKWA; SWAIN, 2011; YI; KELLY, 2008; ZHANG, Yu; SAILER; LAWN, 2013).

Assim, é necessário que todos esses desfechos sejam considerados para que haja completo entendimento do comportamento mecânico dessas restaurações frente a questão de pesquisa instaurada e, portanto, estudos que aprofundem a temática abordada ainda se fazem necessários. De qualquer maneira, os dados obtidos a partir dessa tese contribuem com a construção do conhecimento científico sobre os materiais restauradores no que diz respeito ao comportamento mecânico em fadiga e, especificamente, em relação à influência do substrato nesse desfecho.

6 CONCLUSÃO

Baseado nos achados dessa tese, foi encontrada clara relação entre o módulo de elasticidade do substrato e o comportamento mecânico das restaurações cerâmicas adesivamente cimentadas. Além disso, o tratamento de superfície do substrato de resina composta notoriamente altera aspectos topográficos do material, porém sem causar interferência no desempenho em fadiga das restaurações a base de cerâmica de dissilicato de lítio.

As diferentes cerâmicas odontológicas avaliadas nos dois primeiros estudos apresentam características específicas inerentes à microestrutura e composição de cada uma delas e, consequentemente, resultados particulares frente aos ensaios realizados como, por exemplo, as diferenças encontradas entre as duas zircônias de diferentes gerações no primeiro estudo. Entretanto, todos os materiais avaliados apresentaram melhor desempenho quando adesivamente cimentadas sobre o material com maior módulo de elasticidade (liga metálica), sendo essa uma clara evidência da conclusão acima proposta. Individualmente, destaca-se o comportamento do material a base de rede cerâmica reforçada por uma rede polimérica (*Polymer-Infiltrated Ceramic Network* - PICN) que apresentou resultados similares à zircônia de terceira geração e dissilicato de lítio quando cimentados sobre material análogo de dentina sobretudo devido à compatibilidade do módulo de elasticidade do material restaurador e substrato.

REFERÊNCIAS

ABOUSHELIB, M. N.; SLEEM, D. Microtensile bond strength of lithium disilicate ceramics to resin adhesives. **The Journal of Adhesive Dentistry**, v. 16, n. 6, p. 547–552, 2014.

AL-HARBI, F. A. *et al.* In vitro shear bond strength of Y-TZP ceramics to different core materials with the use of three primer/resin cement systems. **Journal of Prosthetic Dentistry**, v. 115, n. 1, p. 84–89, 2016.

AMARAL, M. *et al.* Fatigue behavior of Y-TZP ceramic after surface treatments. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 57, p. 149–156, 2016.

ANUSAVICE, K. J.; SHEN, C.; RAWLS. **Phillips Materiais Dentários**. 12. ed. Rio de Janeiro: Elsevier, 2013.

BASSO, G. R. *et al.* Reliability and failure behavior of CAD-on fixed partial dentures. **Dental Materials**, v. 32, n. 5, p. 624–630, 2016.

BELLI, R. *et al.* Fracture Rates and Lifetime Estimations of CAD/CAM All-ceramic Restorations. **Journal of Dental Research**, v. 95, n. 1, p. 67–73, 2016.

BELLI, R. *et al.* Chairside CAD/CAM materials. Part 1: Measurement of elastic constants and microstructural characterization. **Dental Materials**, v. 33, n. 1, p. 84–98, 2017.

BONFANTE, E. A.; COELHO, P. G. A Critical Perspective on Mechanical Testing of Implants and Prostheses. **Advances in Dental Research**, v. 28, n. 1, p. 18–27, 2016.

BOTTINO, M. A. *et al.* Inlays made from a hybrid material: Adaptation and bond strengths. **Operative Dentistry** , v. 40, n. 3, p. E83–E91, 2015.

CADORE-RODRIGUES, A. C. *et al.* Fatigue behavior of bonded lithium disilicate glass-ceramic simplified restorations is not damaged by the finishing/grinding of the bonding surface of dentin analogue material. **International Journal of Adhesion and Adhesives**, v. 107, n. 102824, 2021.

CADORE-RODRIGUES, A. C. *et al.* Fatigue performance of fully-stabilized zirconia polycrystals monolithic restorations: The effects of surface treatments at the bonding surface. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 110, n. 103962, 2020.

CADORE-RODRIGUES, A. C. *et al.* Grinding and polishing of the inner surface of monolithic simplified restorations made of zirconia polycrystals and lithium disilicate glass-ceramic: Effects on the load-bearing capacity under fatigue of the bonded restorations: Grinding and polishing on . **Journal of the Mechanical Behavior of Biomedical Materials**, v. 124, n. 104833, 2021.

CAMPOS, F. *et al.* Adhesive cementation promotes higher fatigue resistance to zirconia crowns. **Operative Dentistry**, v. 42, n. 2, p. 215–224, 2017.

CHEN, C. *et al.* The fracture resistance of a CAD/CAM Resin Nano Ceramic (RNC) and a CAD ceramic at different thicknesses. **Dental Materials**, v. 30, n. 9, p. 954–962, 2014.

CHEN, S. E. *et al.* Fracture Resistance of Various Thickness CAD Lithium Disilicate

Crowns Cemented on Different Supporting Substrates: An In Vitro Study. **Journal of Prosthodontics**, v. 28, n. 9, p. 997–1004, 2019.

COLDEA, A.; SWAIN, M. V.; THIEL, N. Mechanical properties of polymer-infiltrated-ceramic-network materials. **Dental Materials**, v. 29, n. 4, p. 419–426, 2013.

COTES, C. *et al.* Effect of composite surface treatment and aging on the bond strength between a core build-up composite and a luting agent. **Journal of Applied Oral Science**, v. 23, n. 1, p. 71–78, 2015.

DA SILVA, L. H. *et al.* Dental ceramics: A review of new materials and processing methods. **Brazilian Oral Research** , v. 31, p. 133–146, 2017.

DAL PIVA, A. M. de O. *et al.* CAD-FEA modeling and analysis of different full crown monolithic restorations. **Dental Materials**, v. 34, n. 9, p. 1342–1350, 2018.

DAPIEVE, K. S. *et al.* Alumina particle air-abrasion and aging effects: Fatigue behavior of CAD/CAM resin composite crowns and flexural strength evaluations. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 121, n. 104592, 2021.

DE KOK, P. *et al.* The effect of internal roughness and bonding on the fracture resistance and structural reliability of lithium disilicate ceramic. **Dental Materials**, v. 33, n. 12, p. 1416–1425, 2017.

DELLA BONA, A.; CORAZZA, P. H.; ZHANG, Y. Characterization of a polymer-infiltrated ceramic-network material. **Dental Materials**, v. 30, n. 5, p. 564–569, 2014.

DENRY, I.; KELLY, J. R. State of the art of zirconia for dental applications. **Dental Materials** , v. 24, n. 3, p. 299–307, 2008.

DENRY, I. L.; HOLLOWAY, J. A.; ROSENSTIEL, S. F. Crystallization kinetics of a low-expansion feldspar glass for dental applications. **Journal of Biomedical Materials Research**, v. 41, n. 3, p. 398–404, 1998.

FACENDA, J. C. *et al.* Effect of supporting substrate on the failure behavior of a polymer-infiltrated ceramic network material. **Journal of Prosthetic Dentistry**, v. 121, n. 6, p. 929–934, 2019.

FACENDA, J. C.; BORBA, M.; CORAZZA, P. H. A literature review on the new polymer-infiltrated ceramic-network material (PICN). **Journal of Esthetic and Restorative Dentistry**, v. 30, n. 4, p. 281–286, 2018.

FRAGA, S. *et al.* Does luting strategy affect the fatigue behavior of bonded Y-TZP ceramic? **Journal of Adhesive Dentistry**, v. 20, n. 4, p. 307–315, 2018.

FRAGA, S. *et al.* Impact of machining on the flexural fatigue strength of glass and polycrystalline CAD/CAM ceramics. **Dental Materials**, v. 33, n. 11, p. 1286–1297, 2017.

GONZAGA, C. C. *et al.* Slow crack growth and reliability of dental ceramics. **Dental Materials**, v. 27, n. 4, p. 394–406, 2011.

GUARDA, G. B. *et al.* Effects of surface treatments, thermocycling, and cyclic loading on the bond strength of a resin cement bonded to a lithium disilicate glass ceramic. **Operative Dentistry**, v. 38, n. 2, p. 208–217, 2013.

GUILARDI, L. F. *et al.* Effect of zirconia surface treatment, resin cement and aging on the load-bearing capacity under fatigue of thin simplified full-contour Y-TZP restorations. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 97, p. 21–29, 2019.

JAMEL, R. S.; NAYIF, M. M.; ABDULLA, M. A. Influence of different surface treatments of nickel chrome metal alloy and types of metal primer monomers on the tensile bond strength of a resin cement. **Saudi Dental Journal**, v. 31, n. 3, p. 343–349, 2019.

KELLY, J.R.; DENRY, I. Stabilized zirconia as a structural ceramic: An overview. **Dental Materials**, v. 24, n. 3, p. 289–298, 2008.

KELLY, J R *et al.* Fracture surface analysis of dental ceramics: clinically failed restorations. **The International journal of prosthodontics**, v. 3, n. 5, p. 430–440, 1990.

KLOSA, K. *et al.* Cleaning and conditioning of contaminated core build-up material before adhesive bonding. **Materials**, v. 13, n. 12, p. 1–15, 2020.

KWON, S. J. *et al.* Comparison of the mechanical properties of translucent zirconia and lithium disilicate. **Journal of Prosthetic Dentistry**, v. 120, n. 1, p. 132–137, 2018.

LAWN, B. R. *et al.* Materials design in the performance of all-ceramic crowns. **Biomaterials**, v. 25, n. 14, p. 2885–2892, 2004.

MACHADO, P. S. *et al.* Load-bearing capacity under fatigue and FEA analysis of simplified ceramic restorations supported by peek or zirconia polycrystals as foundation substrate for implant purposes. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 123, n. 104760, 2021.

MAO, L. *et al.* Graded Ultra-Translucent Zirconia (5Y-PSZ) for Strength and Functionalities. **Journal of Dental Research**, v. 97, n. 11, p. 1222–1228, 2018.

MAY, L. G. *et al.* Effects of cement thickness and bonding on the failure loads of CAD/CAM ceramic crowns : Multi-physics FEA modeling and monotonic testing. **Dental Materials**, v. 28, n. 8, p. e99–e109, 2012.

NAKAMURA, K. *et al.* Fracture resistance of monolithic zirconia molar crowns with reduced thickness. **Acta Odontologica Scandinavica**, v. 73, n. 8, p. 602–608, 2015.

NISHIOKA, G. *et al.* Fatigue strength of several dental ceramics indicated for CAD-CAM monolithic restorations. **Brazilian Oral Research**, v. 32, p. 1–8, 2018.

NORDAHL, N.; VULT VON STEYERN, P.; LARSSON, C. Fracture strength of ceramic monolithic crown systems of different thickness. **Journal of Oral Science**, v. 57, n. 3, p. 255–261, 2015.

PEREIRA, G. K. R. *et al.* Mechanical reliability, fatigue strength and survival analysis of new polycrystalline translucent zirconia ceramics for monolithic restorations. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 85, p. 57–65, 2018.

PEREIRA, G. K. R. *et al.* Lithium disilicate glass-ceramic vs translucent zirconia polycrystals bonded to distinct substrates: Fatigue failure load, number of cycles for failure, survival rates, and stress distribution. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 91, p. 122–130, 2019.

PICONI, C.; MACCAURO, G. Zirconia as a ceramic biomaterial. **Biomaterials**, v. 20, n. 1, p. 1–25, 1999.

PILECCO, R. O. *et al.* In-lab simulation of CAD/CAM milling of lithium disilicate glass-ceramic specimens: Effect on the fatigue behavior of the bonded ceramic. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 121, n. 104604, 2021.

PROCHNOW, C. *et al.* Hydrofluoric acid concentrations: Effect on the cyclic load-to-failure of machined lithium disilicate restorations. **Dental Materials**, v. 34, n. 9, p. e255–e263, 2018.

QUINN, J. B. *et al.* Fractographic analyses of three ceramic whole crown restoration failures. **Dental Materials**, v. 21, n. 10, p. 920–929, 2005.

RODRIGUES, C. da S. *et al.* Internal adjustments decrease the fatigue failure load of bonded simplified lithium disilicate restorations. **Dental Materials**, v. 34, n. 9, p. e225–e235, 2018.

ROJPAIBOOL, T.; LEEVAILOJ, C. Fracture Resistance of Lithium Disilicate Ceramics Bonded to Enamel or Dentin Using Different Resin Cement Types and Film Thicknesses. **Journal of Prosthodontics**, v. 26, n. 2, p. 141–149, 2017.

SABATINI, C.; PATEL, M.; D'SILVA, E. In vitro shear bond strength of three self-Adhesive resin cements and a resin-modified glass lonomer cement to various prosthodontic substrates. **Operative Dentistry**, v. 38, n. 2, p. 186–196, 2013.

SAILER, I. *et al.* All-ceramic or metal-ceramic tooth-supported fixed dental prostheses (FDPs)? A systematic review of the survival and complication rates. Part I: Single crowns (SCs). **Dental Materials** , v. 31, n. 6, p. 603–623, 2015.

SCHERRER, S. S. *et al.* ADM guidance—Ceramics: guidance to the use of fractography in failure analysis of brittle materials. **Dental Materials**, v. 33, n. 6, p. 599–620, 2017.

SCHERRER, S. S. *et al.* Fractographic ceramic failure analysis using the replica technique. **Dental Materials**, v. 23, n. 11, p. 1397–1404, 2007.

SEYDLER, B.; SCHMITTER, M. Clinical performance of two different CAD/CAM-fabricated ceramic crowns: 2-Year results. **Journal of Prosthetic Dentistry**, v. 114, n. 2, p. 212–216, 2015.

SHEMBISH, F. A. *et al.* Fatigue resistance of CAD/CAM resin composite molar crowns. **Dental Materials**, v. 32, n. 4, p. 499–509, 2016.

SORNSUWAN, T.; ELLAKWA, A.; SWAIN, M. V. Occlusal geometrical considerations in all-ceramic pre-molar crown failure testing. **Dental Materials**, v. 27, n. 11, p. 1127–1134, 2011.

SORRENTINO, R. *et al.* In vitro analysis of the fracture resistance of CAD-CAM monolithic zirconia molar crowns with different occlusal thickness. **Journal of the Mechanical**

Behavior of Biomedical Materials, v. 61, p. 328–333, 2016.

STAWARCZYK, B. *et al.* Three generations of zirconia: From veneered to monolithic. Part I. **Quintessence international**, v. 48, n. 5, p. 369–380, 2017.

SULAIMAN, T. A. *et al.* Effect of different treatments on the flexural strength of fully versus partially stabilized monolithic zirconia. **Journal of Prosthetic Dentistry**, v. 118, n. 2, p. 216–220, 2017.

THOMPSON, J. Y.; ANUSAVICE, K. Effect of Surface Etching on the Flexure Strength and Fracture Toughness of Dicor® Disks Containing Controlled Flaws. **Journal of Dental Research**, v. 73, n. 2, p. 505–510, 1994.

TINSCHERT, J. *et al.* Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics. **Journal of Dentistry**, v. 28, n. 7, p. 529–535, 2000.

TRIBST, J. P. M. *et al.* Influence of ceramic material, thickness of restoration and cement layer on stress distribution of occlusal veneers. **Brazilian oral research**, v. 32, p. e118, 2018.

TSUJIMOTO, A. *et al.* Interfacial characteristics and bond durability of universal adhesive to various substrates. **Operative Dentistry**, v. 42, n. 2, p. e59–e70, 2017.

VENTURINI, A. B. *et al.* Fatigue performance of adhesively cemented glass-, hybrid- and resin-ceramic materials for CAD/CAM monolithic restorations. **Dental Materials**, v. 35, n. 4, p. 534–542, 2019.

WISKOTT, H. W.; NICHOLLS, J. I.; BELSER, U. C. Stress fatigue: basic principles and prosthodontic implications. **The International journal of prosthodontics**, v. 8, n. 2, p. 105–116, 1995.

YAN, J.; KAIZER, M. R.; ZHANG, Y. Load-bearing capacity of lithium disilicate and ultra-translucent zirconias. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 88, p. 170–175, 2018.

YI, Y. J.; KELLY, J. R. Effect of occlusal contact size on interfacial stresses and failure of a bonded ceramic: FEA and monotonic loading analyses. **Dental Materials**, v. 24, n. 3, p. 403–409, 2008.

ZHANG, F. *et al.* Strength, toughness and aging stability of highly-translucent Y-TZP ceramics for dental restorations. **Dental Materials**, v. 32, n. 12, p. e327–e337, 2016.

ZHANG, Y.; LAWN, B. R. Novel Zirconia Materials in Dentistry. **Journal of Dental Research**, v. 97, n. 2, p. 140–147, 2018.

ZHANG, Y. *et al.* Damage accumulation and fatigue life of particle-abraded ceramics. **The International journal of prosthodontics**, v. 19, n. 5, p. 442–448, 2006.

ZHANG, Y.; KELLY, J. R. Dental Ceramics for Restoration and Metal Veneering. **Dental Clinics of North America**, v. 61, p. 797–819, 2017.

ZHANG, Y.; SAILER, I.; LAWN, B. R. Fatigue of dental ceramics. **Journal of Dentistry**, v. 41, n. 12, p. 1135–1147, 2013.

ZIMMERMANN, M. *et al.* Influence of material thickness on fractural strength of CAD/CAM fabricated ceramic crowns. **Dental Materials Journal**, v. 36, n. 6, p. 778–783, 2017.

ZUCUNI, C. P. *et al.* CAD/CAM machining Vs pre-sintering in-lab fabrication techniques of Y-TZP ceramic specimens: Effects on their mechanical fatigue behavior. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 71, p. 201–208, 2017.

ZUCUNI, C. P.; PEREIRA, G. K. R.; VALANDRO, L. F. Grinding, polishing and glazing of the occlusal surface do not affect the load-bearing capacity under fatigue and survival rates of bonded monolithic fully-stabilized zirconia simplified restorations. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 103, n. 103528, 2020.

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

Introduction

Authors are requested to submit their original manuscript and figures via the online submission and editorial system for Dental Materials. Using this online system, authors may submit manuscripts and track their progress through the system to publication. Reviewers can download manuscripts and submit their opinions to the editor. Editors can manage the whole submission/review/revise/publish process.
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