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Helder Callegaro Velho

**AVALIAÇÃO DOS PARÂMETROS DE TESTES EMPREGADOS PARA
INDUZIR FADIGA CÍCLICA EM MATERIAIS RESTAURADORES
DENTÁRIOS**

Santa Maria, RS
2021

Helder Callegaro Velho

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

Orientadora: Prof. Dr^a. Andressa Borin Venturini
Coorientador: Prof. Dr^o. Luiz Felipe Valandro

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Helder Callegaro Velho

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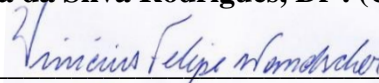
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RESUMO

AVALIAÇÃO DOS PARÂMETROS DE TESTES EMPREGADOS PARA INDUZIR FADIGA CÍCLICA EM MATERIAIS RESTAURADORES DENTÁRIOS

AUTOR: Helder Callegaro Velho
ORIENTADORA: Andressa Borin Venturini
COORIENTADOR: Luiz Felipe Valandro

A presente dissertação está dividida em 2 estudos. O estudo 1 revisou sistematicamente a literatura para identificar os métodos e parâmetros de testes utilizados para induzir fadiga cíclica em espécimes não anatômicos de materiais restauradores dentários disponíveis comercialmente e discutir as indicações, limitações e implicações dos diferentes parâmetros de testes e métodos adotados. Foram selecionados estudos *in vitro* escritos em língua inglesa que avaliaram espécimes não anatômicos de materiais restauradores dentários disponíveis comercialmente, submetidos à fadiga cíclica. A busca foi realizada nas bases de dados *PubMed*, *Scopus* e *Web of Science* por 2 pesquisadores independentes. A busca inicial resultou em 1.848 artigos, dos quais 92 foram incluídos. Dentre os estudos incluídos, a maioria avaliou cerâmicas odontológicas, testadas sem a presença de um agente de cimentação ou substrato de suporte, utilizando ensaios de fadiga acelerados, frequências ≤ 2 Hz, pistões de aço inoxidável com ponta esférica, carregamento axial e ambiente de teste úmido. Assim, foi possível concluir que a definição da configuração do teste e do método de fadiga deve estar estritamente relacionada ao objetivo do estudo, e os achados devem ser compatíveis com o padrão de falha clínica ou conforme descrito nas diretrizes internacionais de testes mecânicos, onde a análise fractográfica é recomendada. O estudo 2 teve por objetivo avaliar a influência da frequência de carregamento no comportamento mecânico à fadiga de restaurações simplificadas de cerâmica infiltrada por polímeros (PICN) e dissilicato de lítio (LD). Trinta discos ($\varnothing = 10$ mm; espessura = 1,0 mm) de cada cerâmica foram cimentados adesivamente em discos de material análogo à dentina ($\varnothing = 10$ mm; espessura = 2,0 mm). Os espécimes foram alocados aleatoriamente em 4 grupos ($n = 15$) de acordo com o material cerâmico (LD ou PICN) e com a frequência de carregamento utilizada (2 ou 20 Hz). O teste de fadiga *stepstress* (carga inicial = 200 N; incremento = 100 N; 10.000 ciclos) foi executado e os dados foram analisados por testes de sobrevivência (Kaplan Meier e Mantel-Cox) e análise de Weibull. Nenhuma diferença estatística foi detectada em relação a carga para falha em fadiga e número de ciclos para falha entre os grupos do mesmo material cerâmico para as diferentes frequências avaliadas. Todas as falhas foram trincas radiais partindo da superfície de cimentação. Portanto, o uso de uma frequência de carregamento de 20 Hz é uma alternativa viável para acelerar os testes de fadiga, sem alterar os valores de carga para falha por fadiga, número de ciclos para falha e o padrão de falha de restaurações simplificadas de cerâmica infiltrada por polímeros e dissilicato de lítio.

Palavras-chave: Análise de sobrevivência. Cerâmicas odontológicas. Fadiga. Materiais dentários. Parâmetros de testes de fadiga. Resina composta.

ABSTRACT

EVALUATION OF TESTS PARAMETERS USED TO INDUCE CYCLIC FATIGUE IN DENTAL RESTORATIVE MATERIALS

AUTHOR: Helder Callegaro Velho
PROMOTER: Andressa Borin Venturini
CO-PROMOTER: Luiz Felipe Valandro

The present dissertation is divided into two studies. Study 1 systematically reviewed the literature to identify what are the methods and testing parameters used to induce fatigue on non-anatomic specimens of commercially available dental restorative materials, to discuss their indication, limitations, and implications. In vitro studies written in English which evaluated commercially available non-anatomic dental restorative material samples subjected to mechanical cyclic fatigue were selected. The search was performed in the PubMed, Scopus, and Web of Sciences databases by 2 independent researchers. The initial search yielded 1,848 articles, of which 92 were included. Among the studies included, most evaluated dental ceramic materials, tested without the presence of a luting agent, or supporting substrate, evaluated the materials using accelerated fatigue tests, frequencies ≤ 2 Hz, stainless steel indenters with a spherical tip, axial loading, and a wet test environment. Thus, it was possible to conclude that the definition of the test configuration and the fatigue method must be strictly related to the objective of the study, and the findings must be compatible with the clinical failure pattern or as described in the international standards for mechanical tests, in which fractographic analysis is recommended. Study 2 aimed to evaluate the influence of loading frequency on the mechanical fatigue behavior of polymer-infiltrated ceramic-network (PICN) and lithium disilicate (LD) simplified restorations. Thirty discs ($\varnothing = 10$ mm; thickness = 1.0 mm) of each ceramic material were adhesive cemented onto dentin analogue discs ($\varnothing = 10$ mm; thickness = 2.0 mm). The specimens were randomly allocated in 4 groups (n = 15) according to the ceramic (LD or PICN) and the loading frequency used (2 Hz or 20 Hz). Stepstress fatigue test (initial load = 200 N; increment = 100 N; 10,000 cycles) was performed and the data were analyzed by survival tests (Kaplan Meier and Mantel-Cox) and Weibull analysis. No statistical difference was detected in relation to fatigue failure load and number of cycles for failure between the groups of the same ceramic for the different frequencies evaluated. All failures were radial cracks in the cementation surface. Therefore, the use of a 20 Hz loading frequency shows to be a viable alternative to accelerate fatigue tests without changing the fatigue failure load values, number of cycles for failure and the failure pattern of simplified restorations made of lithium disilicate glass ceramic or polymer infiltrated ceramic network.

Keywords: Dental ceramics. Dental Materials. Fatigue. Fatigue test parameters. Resin composite. Survival analysis.

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1 INTRODUÇÃO GERAL

Os materiais restauradores dentários estão em constante evolução, visto que os avanços da odontologia adesiva e a demanda por estética têm favorecido o desenvolvimento de novos compósitos para substituir o amálgama, assim como cerâmicas reforçadas por partículas em troca das restaurações metalocerâmicas (BAYNE et al., 2019). Aliado a isso, a incorporação da tecnologia CAD/CAM (*Computer-Aided Design/Computer-Aided Manufacturing*) e o desenvolvimento de materiais restauradores em blocos manufaturados industrialmente para usinagem têm permitido a confecção de restaurações com maior confiabilidade mecânica devido à redução da presença de defeitos e poros no interior do material (ZHANG; KELLY, 2017).

No ambiente bucal, os materiais restauradores estão expostos à presença de umidade, cargas mastigatórias, alterações de temperatura e de pH (DENRY; HOLLOWAY, 2010). Além disso, esses materiais geralmente são submetidos à cargas muito abaixo de sua carga crítica, de forma contínua ou repetitiva, estando sujeitos à falha por fadiga (KELLY et al., 2017). Neste sentido, a avaliação *in vitro* prévia dos materiais restauradores dentários por meio de ensaios em fadiga é importante para estimar os valores de resistência, risco de falha ou comparar as variantes do material, e assim, estimar a capacidade mecânica destes e a sua compatibilidade com as demandas exigidas quando em função na cavidade oral (KELLY, 1999).

Fadiga pode ser definida como um processo de degradação (enfraquecimento) de um material, sob a influência de estresse mecânico, químico ou biológico, ou uma combinação destes (KELLY et al., 2017). O processo de falha por fadiga inicia com o aparecimento da trinca a partir de defeitos estruturais do material ou superficiais (desgaste com pontas abrasivas ou padrão topográfico promovido pelo processamento), resultando no crescimento dessa trinca e por fim, a falha catastrófica do material (ROSENTRITT; BEHR; PREIS, 2016). Dois mecanismos relevantes estão envolvidos neste processo, decorrentes ou do crescimento 'subcrítico' ou 'lento' de trincas (SCG) e/ou de efeitos cíclicos adicionais (KELLY et al., 2017). SCG é um processo que envolve o crescimento de trincas a partir de defeitos pré-existentes em níveis de tensão inferiores ao necessário para levar à fratura do material (TASKONAK et al., 2008). No que diz respeito ao processo de corrosão sob tensão assistido quimicamente por água, a difusão de moléculas de água na ponta de uma trinca causa hidrólise das ligações de siloxano (Si-O-Si) nas cerâmicas vítreas (CHARLES, 1958; FREIMAN; WIEDERHORN; MECHOLSKY, JR., 2009; MICHALSKE; FREIMAN, 1993; WIEDERHORN, 1967;

WIEDERHORN; BOLZ, 1970). Considerando as resinas compostas, a absorção de água da matriz resinosa pode levar à hidrólise das interfaces carga-matriz, acelerando o processo de falha (TAKESHIGE et al., 2007).

Neste sentido, os ensaios de fadiga são uma alternativa para a avaliação do comportamento mecânico dos materiais restauradores dentários a longo prazo, pois eles simulam um cenário mais próximo do que ocorre clinicamente (WISKOTT; NICHOLLS; BELSER, 1995). Os ensaios de fadiga podem ser classificados de acordo com o modo de aplicação de tensão ou deformação em: estático (permanecendo constante com o tempo), dinâmico (aplicado com uma taxa constante) ou cíclico (tensão ou magnitude de deformação variando com o tempo) (BARAN; BOBERICK; MCCOOL, 2001). No entanto, o carregamento cíclico é o teste de fadiga mais clinicamente relevante, pois produz uma melhor percepção sobre a resposta do material para uma vida útil completa (KELLY et al., 2017). Diversos ensaios de fadiga cíclica podem ser executados utilizando diferentes geometrias de teste e metodologias de ensaio de fadiga dependendo do objetivo do estudo em questão.

Geometrias de teste de flexão (resistência flexural 3-pontos, 4-pontos ou biaxial) são importantes para a caracterização e avaliação da confiabilidade dos materiais por meio da determinação de fadiga e limites de resistência, quando submetidos a fatores específicos (por exemplo, como um tratamento de superfície afeta uma propriedade do material, ou um método de processamento, entre outros) (KELLY et al., 2017). Por outro lado, o uso de geometrias de teste com espécimes no formato de coroas anatômicas ou não anatômicas (discos, placas ou coroas planas) cimentadas em um substrato de suporte (dentina, material análogo à dentina, metal) são uma alternativa para a aproximação dos ensaios laboratoriais com a realidade clínica, onde variáveis como cimentação adesiva passam a ser consideradas. Um ponto importante a ser observado neste tipo de geometria cimentada, é que os modos de falha sejam semelhantes aos padrões encontrados clinicamente (KELLY et al., 2010).

A respeito das metodologias de ensaios de fadiga, métodos como a determinação de gráficos de ciclos de tensões até a falha (curvas S-N) são notoriamente os que consomem mais tempo para aquisição dos dados, apesar de produzirem melhores percepções sobre a resposta do material para uma vida útil completa em relação aos ensaios de fadiga acelerados (KELLY et al., 2017). Uma alternativa a esses testes é o uso de métodos de fadiga acelerados como *Staircase*, *Stepstress* ou *Boundary*. Nesses ensaios, as amostras são submetidas a condições de teste mais severas (níveis de tensões superiores aos encontrados clinicamente) de forma a

acelerar a falha, porém mantendo os modos de falha observados clinicamente (BONFANTE; COELHO, 2016; NELSON, 2004).

Após a definição da geometria do teste e do método de ensaio de fadiga, algumas variáveis do teste precisam ser definidas, como o pistão a ser utilizado para aplicar carga (material, formato e dimensão), frequência de carregamento (número de ciclos por segundo), número de ciclos e ambiente de teste (úmido ou seco). Em especial, a frequência de carregamento é uma das variáveis que pode influenciar no tempo necessário para a execução dos ensaios de fadiga cíclica. Na perspectiva de aproximação com a realidade clínica, onde a frequência mastigatória varia de 0,95 Hz a 2,15 Hz (PO et al., 2011), o uso de frequências próximas a 2 Hz parece ser a escolha ideal. No entanto, a aquisição dos dados torna-se muito lenta ao utilizar estas frequências. Neste sentido, alguns estudos vêm avaliando o uso que frequências de carregamento mais altas (10, 20 e 40 Hz) como alternativa para acelerar os ensaios de fadiga em diferentes materiais cerâmicos (FRAGA et al., 2016, 2020; JOSHI et al., 2014; KELLY et al., 2010). Maiores valores de carga para falha por fadiga foram encontrados para uma frequência de 20 Hz em comparação a 2 Hz em uma cerâmica a base de alumina infiltrada por vidro (KELLY et al., 2010). Por outro lado, Fraga et al. (2016) constataram que é possível acelerar os ensaios de fadiga em uma zircônia parcialmente estabilizada por óxido de ítrio (Y-TZP) utilizando uma frequência de 20 Hz sem influenciar os valores de resistência a fadiga. No que diz respeito às cerâmicas vítreas de fluorapatita (JOSHI et al., 2014) e reforçada por leucita (FRAGA et al., 2020), a frequência de carregamento não influenciou os valores de resistência à fadiga nem o tempo necessário para falha em testes de sobrevivência. No entanto, a adoção de frequências de carregamento mais altas para os demais materiais cerâmicos ainda requer investigação.

Frente ao exposto em relação a importância da avaliação do comportamento à fadiga de materiais restauradores dentários, conhecer os principais métodos utilizados e a influência dos diferentes parâmetros de teste na obtenção dos dados, configura-se como uma temática relevante que requer investigação. Neste sentido a presente dissertação está fragmentada em 2 artigos.

ARTIGO 1: *What are the methods and testing parameters used to induce mechanical fatigue in non-anatomic dental restorative material specimens? A scoping review.* Apresenta como objetivo revisar sistematicamente a literatura a fim de identificar os métodos, parâmetros de testes e características usadas para induzir fadiga cíclica em espécimes não anatômicos de

materiais restauradores dentários, e discutir as indicações, limitações e implicações dos diferentes parâmetros de testes e métodos adotados.

ARTIGO 2: *Accelerated loading frequency does not influence the fatigue behavior of polymer infiltrated ceramic network or lithium disilicate glass-ceramic restorations.* O estudo visa avaliar se é possível acelerar os ensaios de fadiga aumentando a frequência de carregamento sem influenciar o comportamento mecânico à fadiga de restaurações de cerâmica infiltrada por polímeros e de dissilicato de lítio.

2 ARTIGO 1: WHAT ARE THE METHODS AND TESTING PARAMETERS USED TO INDUCE MECHANICAL FATIGUE IN NON-ANATOMIC DENTAL RESTORATIVE MATERIAL SPECIMENS? A SCOPING REVIEW.

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What are the methods and testing parameters used to induce mechanical fatigue in non-anatomic dental restorative material specimens? A scoping review

Helder Callegaro Velho^a, Kiara Serafini Dapieve^a, Gabriel Kalil Rocha Pereira^a, Luiz Felipe Valandro^a, Andressa Borin Venturini^{a*}

^aMSciD and PhD Post-Graduate Program in Oral Science, Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil

Authors' e-mail addresses

Helder Callegaro Velho (heldercvelho@hotmail.com)

Kiara Serafini Dapieve (kiara_s_d@hotmail.com)

Gabriel Kalil Rocha Pereira (gabrielkrpereira@hotmail.com)

Luiz Felipe Valandro (valandrolf@gmail.com)

Andressa Borin Venturini (andressa.venturini@hotmail.com)

***Corresponding author:** Andressa Borin Venturini, D.D.S, M.S.D., Ph.D., Assistant Researcher, Federal University of Santa Maria, Faculty of Odontology, MSciD-PhD Graduate Programs in Oral Science, Prosthodontics Unit, 1000 Roraima Av, T Street, Building 26F, Room 2360, UFSM Campus, 97105-900, Santa Maria, Brazil. E-mail: andressa.venturini@hotmail.com

Abstract

The aim of the present scoping review was to identify the methods, testing parameters, and characteristics used to induce cyclic fatigue on non-anatomic dental restorative material specimens, to discuss their indications, limitations, and implications. The protocol of this study, available online (<https://osf.io/m95cz/>), followed the Joana Briggs Institute guidelines, and the reporting was based on PRISMA Extension for Scoping Reviews. In vitro studies written in English which evaluated commercially available non-anatomic dental restorative material samples subjected to mechanical cyclic fatigue were selected. The search was performed in PubMed, Scopus and Web of Science databases by 2 independent researchers. Study screening was also undertaken by 2 independent researchers using the EndNote program. Next, a descriptive analysis was performed considering dental restorative material, luting agent, type of supporting substrate, test geometry, fatigue method, test parameters (loading frequency, material and shape/dimension of indenter, direction of load application), test environment conditions (dry or wet) and fractographic analysis (present or absent). The initial search yielded 1,848 articles, of which 92 were included. Based on the collected data, most of the included studies evaluated dental ceramic materials (n= 60, 65.2%); tested materials without the presence of a luting agent or supporting substrate (n= 60, 65.2%); used staircase (n= 52, 55.9%) or step-stress (n= 22, 23.7%) accelerated fatigue tests, loading frequencies below 2 Hz (n= 43, 42.6%), stainless steel (n= 19, 19.2%) or tungsten carbide (n= 14, 14.1%) load applicators with spherical shaped tip 40 mm diameter (n= 17, 18.3%) or <7 mm diameter (n= 12, 12.9%); applied only axial loads (n= 87, 92.6%); and considered a wet testing environment (n= 73, 75.3%). Although the flexural test configurations, axial loading and accelerated fatigue tests are the most used, the definition of test configuration, loading direction and fatigue method must be strictly related to the study objective. Loading frequencies below 2 Hz are the most used. Despite that, higher frequencies (e.g. 20 Hz) can be an alternative to accelerate the tests of materials such as Y-TZP ceramic, polymer infiltrated ceramic network and lithium disilicate glass-ceramic. Load applicators of different materials and shape/dimensions are described in the literature, but they should not alter the failure mode that the setup is mimicking, thus a careful fractographic analysis is essential. A wet test environment was described in most of the studies, which is an important tool to simulate the hydrolytic degradation of materials observed in clinical scenarios.

Keywords: Cyclic fatigue. Dental ceramics. Resin composites. Test variables.

1. Introduction

In vitro evaluation of dental restorative materials is essential to assess their mechanical capacity and compatibility with the physiological demands when in function in the oral cavity. Hence, material's resistance values, risk of failure or comparing the material variants [1]. Dental restorative materials in the oral environment are subjected to challenging conditions such as humidity, temperature and pH variations [2]. In addition, these materials are usually loaded far below their critical load, either continuously or repetitively, meaning they are subjected to fatigue [3].

Fatigue is often defined as the degradation (weakening) of a structural component under the influence of mechanical, chemical, or biological stress, and in most cases a combination of them. This process mostly involves two major relevant mechanisms, arising either from 'subcritical' or 'slow' crack growth (SCG) and/or cyclic effects [3]. SCG is a process that involves the stable growth of pre-existing flaws at stress intensity factor (KI) levels lower than that necessary for the flaw to become unstable (K_{Ic}) [4]. In relation to the stress-corrosion process chemically assisted by water, the diffusion of water molecules at the tip of a crack causes hydrolysis of the siloxane bonds (Si-O-Si) in glass ceramics [5–9]. In addition, the mechanical degradation in ceramic materials is favored by the friction between crack walls during cyclic loading due to the change in the stress concentrated at the crack tip from tensile to compression (cyclic effects) [10]. Considering resin composite materials, water absorption from the resin matrix may lead to the hydrolysis of filler-matrix interfaces, accelerating the failure process [11].

Fatigue-induced failure mechanisms depend on the type of material [12]. Pre-existing flaws in ceramics originating from processing serve as nuclei for crack propagation. The crack propagates catastrophically when it has grown to a critical size and the stress states at the crack tip exceeds the materials' fracture toughness [13]. The direct measurement of the crack velocity as a function of stress intensity factor is a way to determine SCG [14]. Several mechanisms may participate in fatigue-induced damage in composites. Among them, filler type [15], matrix cracking, matrix deformation, void formation, multidirectional cracking, filler debonding and filler failure [12]. A crack will start in either the matrix or the filler particles (or the interface), with the crack propagation and direction being continuously modified during the fatigue process, due to changes in the distribution of internal stresses [12].

In this sense, fatigue tests are an alternative for assessing the long-term behavior of dental restorative materials [16]. The mode of stress or strain application in fatigue tests may be static (remaining constant with time), dynamic (applied at some constant rate), or cyclic (stress or strain magnitude varying with time) [12]. However, the cyclic loading is the most clinically relevant fatigue test as they produce the best insight in the material response for a complete service life [3]. Thus, it

is necessary that the tests are planned to simulate the different aspects to which dental restorative materials are subjected in order to accurately predict the clinical behavior and failure modes that occur clinically [1]. It is also necessary to define the test geometry, fatigue method and some test parameters such as, loading frequency, load applicator type (shape and dimension), test environment conditions and fractographic analysis. So far, there is no standardization for choosing these fatigue test parameters. Furthermore, the effect of using different parameters in cyclic fatigue tests on the mechanical behavior and failure mode of dental restorative materials is not completely clear. In addition, a compiled of literature would help to discuss the existing methods and guide future studies.

As mentioned, a wide range of variables which characterize cyclic fatigue tests associated with the large number of studies found in the literature makes the variability among studies very large, which in turn makes it difficult to compare their findings. Thus, the aim of the present scoping review was to systematically review the literature to identify the methods, testing parameters, and characteristics used to induce cyclic fatigue on non-anatomic restorative dental material specimens, to discuss their indications, limitations, and implications.

2. Materials and methods

The protocol of this study was based on the framework proposed by Peters et al. [17] according to the Joana Briggs Institute and is available at the following link: (<https://osf.io/m95cz/>). In addition, the reporting of this scoping review was based on PRISMA Extension for Scoping Reviews [18].

2.1 Inclusion criteria

In vitro studies which evaluated commercially available dental restorative material samples induced to mechanical cyclic fatigue were selected. Studies which evaluated single-unit restorations with simplified geometry (non-anatomic) were included, regardless of the type of dental restorative material or the presence of luting agents, and/or the type of adopted supporting substrate (e.g., tooth remnant tissues: enamel, dentin; or even simplified in vitro assemblies using resin composite, epoxy resin, metal alloys, ceramic alloys; among others). Studies which evaluated the mechanical fatigue performance of the restorative material following normative guidelines were also considered (i.e. disc- or bar- shaped tested under bending tests, among other methods). Studies which did not conduct a fatigue test until failure were not considered, for example studies which used mechanical cycling for aging, and then executed a static test.

2.2 Search

The search was performed in three databases (PubMed, Scopus and Web of Science) without date restrictions (last executed on May 07, 2020) and was limited to articles written in the English

language. The search strategy was based on Mesh terms and free-text specific terms of PubMed and adapted for the Scopus and Web of Science databases (Table 1).

2.3 Screening

The search was initially undertaken using the EndNote program (EndNote X9, Thomson Reuters, New York, NY). Two researchers (H.C.V. and K.S.D.) independently identified articles by first analyzing titles and abstracts for relevance and the presence of the eligibility criteria. Retrieved records was classified as include, exclude, or uncertain. The full-text articles of the included and uncertain records were selected for further eligibility screening by 2 reviewers (H.C.V. and G.K.R.P) (acting independently). Discrepancies in screening of titles/abstracts and full text articles were resolved through a discussion.

2.4 Charting the results

A form was created using the Excel program (Microsoft Excel, Redmond, WA), which was tested by four reviewers to reach a consensus for data collections. Then, two reviewers (H.C.V. and K.S.D.) extracted the data and the other two reviewers (A.B.V. and G.K.R.P.) checked it. The following data were collected: dental restorative material, luting agent and type of supporting substrate (if present), test geometry, fatigue method, test parameters (loading frequency, type of load applicator – material and shape/dimension, load application direction), in accordance with normative guidelines (ISOs, ASTMs, or other), test environment conditions (dry or wet), and fractographic analysis (present or absent). Each data was considered separately in data collection for studies which used more than one restorative material, test method or parameter. Meanwhile, only new information was considered for studies in which data were based on previous studies (i.e. duplicate data were excluded).

2.5 Data analysis

The synthesis focused on describing the main characteristics of the studies regarding the collected data. In addition, qualitative analysis was performed and presented using tables and illustrations.

3. Results

The initial search resulted in 1848 studies. A total of 157 articles were considered eligible for full-text assessment after duplicate removal and assessment of titles and abstracts, from which 92 records were included for qualitative analysis (Figure 1). The bibliographic references of the included studies can be consulted in the supplementary material (List of included studies). The studies by Joshi et al. [19] and Joshi et al. [20] presented data which appeared to have been collected from the same

group of specimens. Therefore, the data from the study by Joshi et al. [20] were excluded from the qualitative analysis to avoid the risk of duplicates.

Regarding the main characteristics of the included studies, most studies evaluated dental ceramics (n= 60; 65.2% – Table 2), whereas the remaining studies considered resin composites (n= 28; 30.4% - Table 2) or glass ionomer as restorative material (n= 4; 4.4% – Table 2). Most cyclic fatigue studies only tested the restorative material as test geometry (n= 57; 62% – Table 3), and only a few studies used simplified cemented geometries such as disc on disc (trilayer cemented assembly) (n= 25; 27.2% – Table 3) or simplified non-anatomic crowns (n= 7; 7.6% – Table 3). Figure 2 illustrates the specimen format used for each test geometry.

It is important to highlight that changes have been detected in the execution of cyclic fatigue tests over time. The use of support substrates and luting agents become more common after 2007 (Figure 3A). Regarding the fatigue methods, only the staircase approach (n= 14; 66.7%) and fatigue cycling until failure (n= 7; 33.3%) were reported until 2007. Other reported methods (e.g. step stress approach and boundary technique) have been used (Figure 3B) from that time forward. It can be emphasized that accelerated fatigue tests such as the staircase (n= 52; 55.9% - Table 4) and step-stress approaches (n= 22; 23.7% - Table 4) were the most used until 2020. The cycling until failure methods were only used in 16.1% (n= 15) of the studies (Table 4). A detailed description of the test methodologies performed by each study including fatigue method, loading frequency, material and shape of the load applicator, load application direction, test environment conditions, and use of normative guidelines are described in Table 4.

The load applicator material was not reported in 44.4% (n= 44) of the included studies, and the format/dimension was not reported in 46.2% studies (n= 43). Stainless steel (n= 19; 19.2%) and tungsten carbide (n= 14; 14.1%) were the most reported materials used for the load applicator. Different load applicator materials were compared in only two studies: Anderson et al. [21] compared load applicators made of nanohybrid composite resin dentures, unfilled acrylic resin dentures, tungsten carbide and a pressed leucite glass ceramic; meanwhile, Stijacic et al. [22] considered an interpenetrating polymer network resin-based dentures, heat-pressed leucite glass-ceramic on supporting a high noble alloy substructure, heat-pressed lithium disilicate and a zirconium dioxide load applicator. Spherical/hemispherical-shaped tip load applicator with a diameter of 40 mm in diameter were the most used (n= 17; 18.3%). The cylinder/circular-shaped tips (<3 mm) were used by 14.0% (n= 13) and spherical-shaped tips (<7 mm) by 12.9% (n= 12) of the studies.

Regarding load application mode, axial load application was reported by most studies (n= 87; 92.6%), while rotary was reported in 3.2% (n= 3) and sliding by 4.3% (n= 4) of the studies, respectively. A wet test environment was the most reported by the studies (n= 73; 75.3%). A dry test environment was reported for only 7.2% (n= 7) of the studies. Information about the test environment

was not reported in 17.5% (n= 17) of studies. A fractographic analysis was performed by 78.5% (n= 73) of studies. A description of implementing normative guidelines for the fatigue test design was not reported in most of the included studies (n= 65; 69.8%). The ISO 6872 normative guideline was the most cited (n= 26; 28.0%).

4. Discussion

The present study is the first which attempts to provide a synthesis of information considering the test characteristics, parameters and methods used to induce cyclic fatigue in non-anatomical restorative dental material specimens. The data collected highlight that several test parameters and methodologies have been used to conduct fatigue tests on dental restorative materials. Therefore, it is important to discuss the methodological implications and the accurate approximation of the clinical reality.

In vitro tests grant the researcher the possibility of isolating factors and addressing specific parameters which may influence a desired outcome. On the other hand, in vitro tests may also enable adopting more complex testing scenarios which attempt to simulate almost all factors that a restoration may be exposed to in the clinical environment. This latter scenario is undeniably more complex [23]. Thus, the researcher should consider which experimental design and method are suitable to the study objective in order to access the outcome, isolating the desired study factors or associating them to do so. The use of non-anatomical specimens avoids the influence of different thicknesses of restorative material on the cusps, facilitating the control of the loads to predict stress distribution. In addition, the reduced time for sample preparation and the low cost are advantages of using non-anatomical specimens. In this sense, we believe that this is the main reason for the common use of non-anatomical specimens, and why this scoping review attempts to collect data and guide future research of such scenarios. Undoubtedly, a scoping review of anatomic samples may further contribute to the knowledge of this specific scientific field in the future.

4.1 Test geometry

The International Standardization Guidelines are available to guide the execution of mechanical tests in specimens of strict restorative materials with specific formats, such as the International Organization for Standardization - ISO 6872 (2015) for ceramic materials, ISO 10477 (2018) for polymer-based crown and veneering materials, and the American Society for Testing and Materials - ASTM e-647-93 (1994), which describe standard methods to estimate fatigue crack growth rates. Despite the existence of International Standardization Guidelines for non-anatomic specimens in flexural test configurations, only 26 studies reported the use of such guidelines from the

46 included studies which explored this scenario. Compliance with the guidelines outlined by an international association ensures that all factors of the fracture mechanics of the material are respected, avoiding potential masking of the effect of the studied factor on the outcome. It also enhances the reproducibility and comparability between studies. Therefore, it is mandatory to obey these guidelines when using such geometries. However, there are no guidelines to be followed when considering cemented specimens which involve more complex testing geometries. Thus, the recommendation that has been widely followed is that the mechanical tests should induce failures observed in a clinical scenario, which highlights the importance of fractographic analysis in both scenarios [13,23].

Most of the studies in non-anatomic specimens have evaluated materials in flexural test configurations (e.g. 3-point, 4-point, or biaxial flexural strength) (Table 3). These tests are important in characterizing and assessing the reliability of materials through determining fatigue, endurance limits and S-N curves submitted to specific factors (i.e. how a surface treatment affects a material property, or a processing method, among others) [3]. However, the specimens are loaded without being supported by any substrate in these tests, being exposed to more aggressive conditions than would occur clinically [24]. Restorations in the clinical setting are bonded to a support substrate (i.e. dental structure or metal core) and different stress distributions can be expected, depending on the modulus of elasticity of the restorative and substrate materials [25–27]. In addition, fatigue tests using cemented specimens can also evaluate the role of adhesive cementation in the mechanical behavior of restorations, since appropriate resin adhesion improves the fracture strength [28,29].

4.2 Fatigue method

Although cyclic fatigue tests are time-consuming experiments for data acquisition, they are the most clinically relevant approach and produce a better perception of the material's response for a complete useful life [3]. Most studies have used accelerated fatigue tests (i.e. staircase, step-stress or boundary technique) (Table 4), which are an alternative to obtain data more quickly. The samples are subjected to more aggressive test conditions in these tests to accelerate the failure. Although they may be carefully executed to promote failure modes which occur clinically [23,30], it is important to consider the obtained data, the method used and its limitations. The staircase method determines the average fatigue limit value of the material for a determined useful life (number of cycles). However, the pre-defined number of cycles does not allow for predictions on different amounts of cycles and this test is not conducted in a wide force range or extreme stress values, which could mask performance under worst case scenarios [23]. On the other hand, each specimen in the step-stress method is loaded with increasing levels of load, meaning that after a certain period (number of cycles) the load is increased step-by-step until its failure or end of the test profiles. This method considers

the cumulative effect of the applied stresses, estimating the probability of survival over time and predicting the reliability for a specific period [31,32]. Another accelerated fatigue test reported was the boundary technique. In this method, two groups of specimens are evaluated for each number of determined cycles, using two stress levels, one corresponding to a low probability of failure and the other with a stress amplitude corresponding to a high probability of failure. An advantage of this methodology is that the data obtained may be used to predict stresses corresponding to low probabilities of failure, providing more precision on estimating the stress range of clinical interest [33–35]. Despite being more efficient approaches (in terms of time and effort), the accelerated methods may limit any phenomenological insight of the material's behavior. In addition, they provide a limited image of the S-N curve and the slow crack growth parameters (n and A) [3]. In this case, the most comprehensive approach is to determine stress-cycles-to-failure plots (S–N, Wöhler curve) [3]. Thus, the choice of the fatigue method must consider the necessary parameters to answer the study objectives.

4.3 Test parameters

The test parameters must then be defined after choosing the test geometry and the fatigue method, including the loading frequency, the type of load applicator (material, shape, and dimension), the load application direction and the test environment. The researcher should consider the material microstructure, the way the crack propagates into it and the influence mechanism of each of these parameters in the fatigue induction to select these parameters.

Regarding the loading frequency, it was observed herein that frequencies below 2 Hz were the most reported by the studies (Table 4), with these values being close to the masticatory frequency (1-2 Hz) [36]. However, the use of low loading frequencies can make data acquisition very time consuming. As mentioned, the friction between the crack walls during cyclic loading can contribute to the crack propagation (mechanical degradation) [10]. This mechanism can be influenced by the loading frequency adopted by the cyclic testing, since the time available for opening and closing the crack may vary according to the frequency adopted.

It is important to highlight that the way the crack propagates changes according to the material's microstructure [3,37,38]. In this sense, adopting higher frequencies (e.g. 10, 20 or 40 Hz) must be carefully considered. It was possible to accelerate the fatigue tests in yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP), polymer infiltrated ceramic network and lithium disilicate glass-ceramic using a frequency of up to 20 Hz [39,40]. However, no differences were observed in the time required for failure using frequencies of 2 or 10 Hz in fluorapatite glass-ceramic [20]. Likewise, a frequency of 20 Hz did not decrease the time required for failure in a leucite-reinforced glass-ceramic, since it required a greater number of cycles for failure for a frequency of

20 Hz compared to 2 Hz [41]. Higher fatigue failure load values were also observed when a glass-infiltrated alumina ceramic was tested using a frequency of 20 Hz compared to 2 Hz [42]. In addition, loading frequencies below 5 Hz have been adopted for resin composites, it is advocated that, due to their viscoelastic properties, high loading frequencies could lead to internal heating during fatigue testing, which may affect results [15,43]. Despite that we did not find conclusive data comparing different loading frequencies for resin composite or other ceramic materials. Therefore, future studies evaluating and comparing this factor are still suggested.

In addition to frequency, the load applicator choice is also a factor which influences the fatigue tests, since the material, shape and dimensions are important variables for failure modes and stress distribution [1]. Despite this, data regarding the load applicator used were not presented in most of the studies (Table 4), which makes reproducibility and comparison between studies difficult. The use of materials with a high modulus of elasticity (i.e. stainless steel and tungsten carbide) for the load applicator may overestimate resistance values [1,44], as it may induce greater contact pressure and consequently higher stress concentrations [42]. Another factor which can also influence the test results is the shape and dimension of load applicator. According to Kelly et al. [1], spheres which are 40 mm to 1 m in diameter would be necessary to generate clinically realistic occlusal contact areas. It is important to pay attention to this aspect, because in decreasing the load applicator diameter, the contact area between load applicator and restorative material is consequently also reduced, generating very high contact pressures which are not reflected in clinically relevant failures [1]. Moreover, flat load applicators should be avoided because they can become edge-loading applicators when the tested material deforms slightly beneath the load applicator [1], with the exception of studies that adopt the International Standardization Guidelines, such as ISO 6872(2015) which standardizes the use of a flat load applicators (1.4 ± 0.2 mm).

Sliding load application was little explored by the included studies when the load application direction is considered, whereas the majority used only axial loads (Table 4). This finding is completely expected, since anatomical specimens are commonly used with this complex loading scenario, but which were excluded from this scoping review limiting the discussion on this topic. Even so, the purpose of using sliding loads is to simulate inclination of the posterior dental cusp and the occlusal contacts assumed during chewing [45]. Among the included studies herein, only one compared axial loading and sliding loading on the reliability and fracture patterns of zirconia cores veneered with pressable porcelain. In this study they concluded that the reliability was not influenced by the load orientation (uni-axial vs. off-axis/sliding), but incorporation of sliding resulted in more aggressive damage to the veneer (partial cone cracks) [46].

Another important aspect is the test environment, as the presence of humidity may influence the fatigue phenomena through a chemical degradation process [3], which reinforces the necessity of

a wet environment during testing. A wet environment was the most used by studies in the current scoping review (Table 4), which denotes the authors' attention in this regard. It should be noted that this chemical degradation process occurs in conjunction with the mechanical degradation caused by cyclic loading over time. In this sense, the use of higher frequencies decreases the action time of the water, which can hinder the chemical degradation process [47].

We highlight the importance of the fractographic analysis of failed specimens throughout this review, replicating and enabling comparison with the clinically found flaws or predicted in the fractography guidance. Additionally, the guidance for fractographic analysis by Scherrer et al. [13] highlights the importance of fractographic analysis to characterize the observed flaws, serving to elucidate the failure origin, failure causes, loading conditions and the crack direction. Cracks in specimens tested in flexural test configurations are expected to start from defects on the tensile side (bottom) towards the compression side (top), where a compression curl is found [13]. Non-anatomical cemented test geometries simulate a scenario closer to that of clinical restorations to predict mechanical performance, where failure modes should mimic those which are clinically reported [3]. Some of the commonly observed clinical fracture modes include cracks initiating from the cementation interface surface opposite the load application [42] and in the crown margin region, juxtaposed at the end of the restoration preparation [48]. Defects introduced by processing in both scenarios would be responsible for triggering stress concentrations during mechanical loading that would induce the coalescence of these defects and subsequently catastrophic crack propagation [49]. Another region responsible for the origin of failures is the occlusal surface [50], as these failures would start from defects introduced by milling and grinding procedures, which are part of the fabrication and fitting/adjusting processes restorations [51,52]. In addition, failures can also initiate from wear facets, where wear related microcracks can form [53].

The limitations of this scoping review mainly include the great heterogeneity among the studies included regarding the considered materials, the specimen formats, testing characteristics, parameters and methods. Even though, this allows a discussion and guidance for future studies, the absence of primary studies comparing the influence of such factors in acquiring fatigue data makes it difficult to indicate one parameter or another. In this sense, further studies are required. In addition, we did not conduct a risk of bias assessment of the included studies, because this is a scoping review. In the future, a risk of bias assessment may become feasible when a systematic review and meta-analysis of the influence of each factor is possible. Despite this, our scoping review successfully points out that researchers have given importance to assessing the fatigue behavior of dental restorative materials and that studies have explored different methods and test configurations as an alternative to predict the clinical reality. Furthermore, our scoping review is the first to provide a

discussion of the effects of each testing parameter and method and provides tools for the researcher to select the best method and parameters which suit the study objective.

5. Conclusion

- Flexural test configurations, axial loading and accelerated fatigue tests are the most used. However, the definition of test configuration, loading direction and fatigue method must be strictly related to the study objective.

- Loading frequencies below 2 Hz are the most used. Despite that, higher loading frequencies (i.e., 20 Hz) prove to be an alternative to accelerate the tests of materials such as Y-TZP ceramic, polymer infiltrated ceramic network and lithium disilicate glass-ceramic.

- Load applicators of different materials and shape/dimensions are described in the literature, but they should not alter the failure mode that the setup is mimicking, thus a careful fractographic analysis is essential.

- A wet test environment was described in most of the studies, which is an important tool to simulate the hydrolytic degradation of materials observed in clinical scenarios.

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TABLES

Table 1. Search strategy according to each database.

<p>PUBMED <i>("Composite Resins"[Mesh] OR Composite resin OR resin-ceramic OR ceramic/polymer material OR polymer-infiltrated ceramic OR hybrid ceramic OR resin nano ceramic OR polymer-infiltrated-ceramic-network material OR CAD/CAM composite OR "ceramics" [Mesh] OR ceramic OR "dental porcelain" [Mesh] OR porcelain OR Glass ceramic OR polycrystalline ceramic OR Feldspathic OR Lithium disilicate OR lithium silicate OR zirconia OR yttrium polycrystalline tetragonal zirconia OR Leucite OR CAD/CAM OR CAD-CAM OR computer aided design OR computer aided machine) AND (Fatigue[Mesh] OR Fatigue) NOT (review OR wear OR shear OR retention OR tensile)</i></p>
<p>SCOPUS <i>("Composite resin" OR "resin-ceramic" OR "ceramic/polymer material" OR "polymer-infiltrated ceramic" OR "hybrid ceramic" OR "resin nano ceramic" OR "polymer-infiltrated-ceramic-network material" OR "CAD/CAM composite" OR "ceramics" OR "ceramic" OR "porcelain" OR "Glass ceramic" OR "polycrystalline ceramic" OR "Feldspathic" OR "Lithium disilicate" OR "lithium silicate" OR "zirconia" OR "yttrium polycrystalline tetragonal zirconia" OR "Leucite" OR "CAD/CAM" OR "CAD-CAM" OR "computer aided design" OR "computer aided machine") AND ("Fatigue") AND NOT ("review" OR "wear" OR "shear" OR "retention" OR "tensile") AND (LIMIT-TO (SUBJAREA , "DENT"))</i></p>
<p>Web of Science <i>(TS=(Composite resin) OR TS=(resin-ceramic) OR TS=(ceramic/polymer material) OR TS=(polymer-infiltrated ceramic OR TS=(hybrid ceramic) OR TS=(resin nano ceramic) OR TS=(polymer-infiltrated-ceramic-network material) OR TS=(CAD/CAM composite) OR TS=(ceramic) OR TS=(porcelain) OR TS=(Glass ceramic) OR TS=(polycrystalline ceramic) OR TS=(Feldspathic) OR TS=(Lithium disilicate) OR TS=(lithium silicate) OR TS=(zirconia) OR TS=(yttrium polycrystalline tetragonal zirconia) OR TS=(Leucite) OR TS=(CAD/CAM) OR TS=(CAD-CAM) OR TS=(computer aided design) OR TS=(computer aided machine)) AND (TS=(fatigue)) NOT (TS=(review) OR TS=(wear) OR TS=(shear) OR TS=(retention) OR TS=(tensile)) AND SU=(Dentistry)</i></p>

Table 2. Description of the materials used in the included studies.

Materials	n (%)
<i>Dental restorative material*</i>	
Dental ceramics	60 (65.2)
Resin composites	28 (30.4)
Glass ionomer as restorative material	4 (4.4)
<i>Luting agent</i>	
Not applicable	60 (65.2)
Resin cement	31 (33.7)
Zinc phosphate	1 (1.1)
<i>Supporting substrate</i>	
Not applicable	60 (65.2)
Dentin analogue	24 (26.1)
Resin composite	8 (8.7)

Table 3. Description of the test geometries used in the included studies.

Test geometry	n (%)
<i>Only restorative material</i>	57 (62.0)
Biaxial flexural strength	20 (21.7)
3-point flexure strength	14 (15.2)
4-point flexure strength	12 (13.0)
Rotating cantilever beam test	3 (3.3)
Flexural Strength according Bream et al. (1994)	2 (2.2)
Diametral compression	2 (2.2)
Compressive loading	2 (2.2)
Compact-tension	1 (1.1)
Bending cantilever beam test	1 (1.1)
<i>Restorative material cemented on substrate</i>	32 (34.8)
Trilayer cemented assembly*	25 (27.2)
Simplified non-anatomic crowns	7 (7.6)
<i>Unclear geometry</i>	3 (3.2)

Table 4. Description of the fatigue methods and test parameters used by the included studies.

Fatigue method	n (%)
Staircase	52 (55.9)
Step stress	22 (23.7)
Fatigue cycling until failure	15 (16.1)
Boundary technique	3 (3.2)
Unclear	1 (1.1)
Fatigue test parameters*	
<i>Material of load applicator</i>	
Not reported	44 (44.4)
Stainless Steel	19 (19.2)
Tungsten Carbide	14 (14.1)
G10 epoxy resin	5 (5.1)
Not applicable	5 (5.1)
Other materials	5 (5.1)
Ceramic	4 (4.0)
Steel	3 (3.0)
<i>Shape/dimension of load applicator</i>	
Not reported	43 (46.2)
Spherical > 40mm diameter	17 (18.3)
Cylinder/circular > 3mm diameter	13 (14.0)
Spherical < 7mm diameter	12 (12.9)
Not applicable	5 (5.4)
Unclear	3 (3.2)
<i>Loading Frequency</i>	
≤ 2Hz	43 (42.6)
>2 and ≤10Hz	25 (24.8)
> 10Hz	24 (23.8)
Not reported	5 (5.0)
Unclear	3 (3.0)
Load contact was 0-7s out of a 2s cycle	1 (1.0)
<i>Type/Direction of load application</i>	
Axial	87 (92.6)
Sliding	4 (4.3)
Rotary	3 (3.2)
<i>Test environment conditions</i>	
Wet	73 (75.3)
Not reported	17 (17.5)
Dry	7 (7.2)
<i>Fractographic analysis</i>	
Present	73(78.5)
Absent	20(21.5)
<i>In accordance to normative guidelines</i>	
Not reported	65 (69.9)
ISO 6872	26 (28.0)
ISO 10477	1 (1.0)
ASTM E647-93	1 (1.0)

*Each data was considered separately in studies that used more than one parameter.

FIGURES

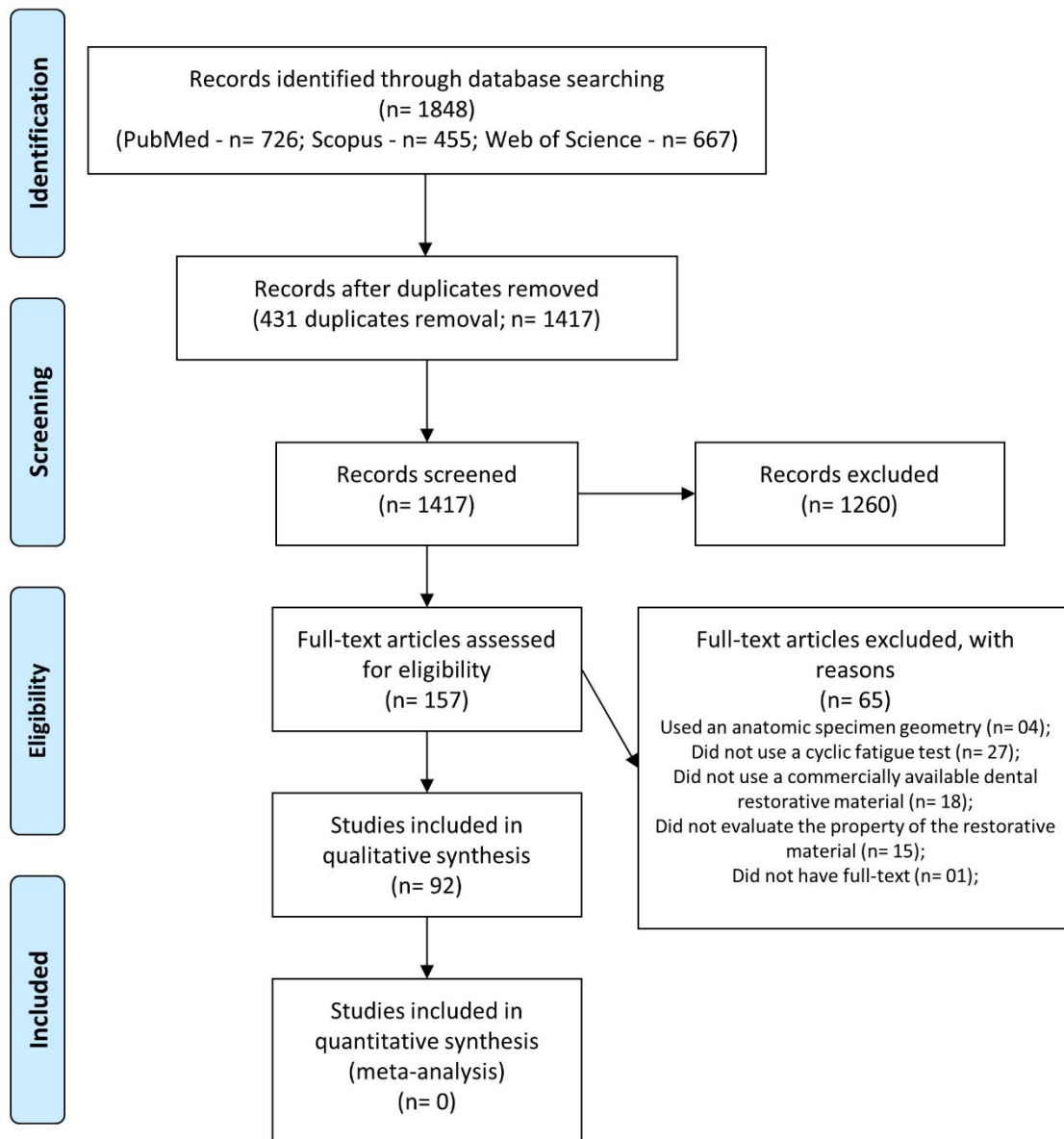


Figure 1. Flowchart of study selection.




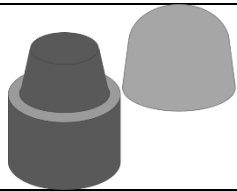
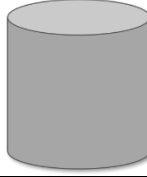

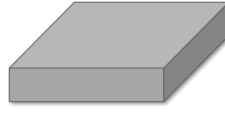
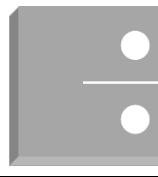
			
-3-point flexure strength; -4-point flexure strength; -Flexural strength; -Bending cantilever beam test	-Biaxial flexural strength	-Trilayer cemented assembly	-Simplified non-anatomic crowns
			
-Diametral compression	-Rotating cantilever beam test	-Compressive loading	-Compact-tension

Figure 2. Specimens format illustrations for each test geometry (dimensions differed among studies). In simplified trilayer assembly and simplified crowns, the restorative material was cemented in a dentin analogue material represented in the image by the darker color.

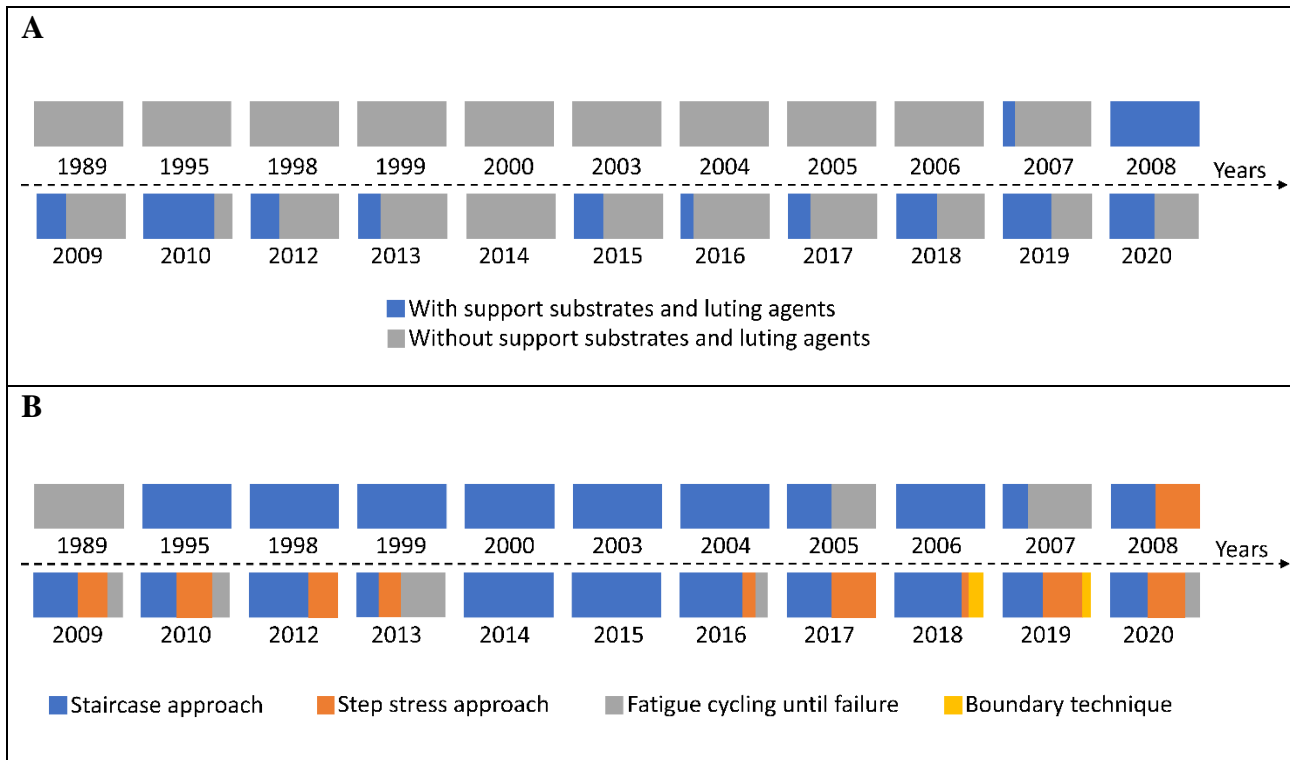


Figure 3. Changes in the execution of the cyclic fatigue test over time. **A** – Use of support substrates and luting agents; **B** – Fatigue methods adopted by studies.

SUPPLEMENTARY MATERIAL

LIST OF INCLUDED STUDIES

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3 ARTIGO 2: ACCELERATED LOADING FREQUENCY DOES NOT INFLUENCE THE FATIGUE BEHAVIOR OF POLYMER INFILTRATED CERAMIC NETWORK OR LITHIUM DISILICATE GLASS-CERAMIC RESTORATIONS

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Accelerated loading frequency does not influence the fatigue behavior of polymer infiltrated ceramic network or lithium disilicate glass-ceramic restorations

Helder Callegaro Velho^a, Kiara Serafini Dapieve^a, Gabriel Kalil Rocha Pereira^a, Sara Fraga^b, Luiz Felipe Valandro^a, Andressa Borin Venturini^a

^a*MSciD and PhD Post-Graduate Program in Oral Science, Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil*

^b*Department of Conservative Dentistry, Federal University of Rio Grande do Sul (UFRGS), Porto Alegre, Rio Grande do Sul State, Brazil*

Authors' e-mail addresses

Helder Callegaro Velho (heldercvelho@hotmail.com)

Kiara Serafini Dapieve (kiara_s_d@hotmail.com)

Gabriel Kalil Rocha Pereira (gabrielkrpereira@hotmail.com)

Sara Fraga (sara.odonto@yahoo.com.br)

Luiz Felipe Valandro (valandrolf@gmail.com)

Andressa Borin Venturini (andressa.venturini@hotmail.com)

Corresponding author:

Luiz Felipe Valandro, D.D.S., M.Sci.D., Ph.D., Associate Professor, Federal University of Santa Maria, Faculty of Odontology, MSciD-PhD Graduate Programs in Oral Science, Prosthodontics Unit, 1000 Roraima Av, T Street, Building 26F, Room 2360, UFSM Campus, 97105-900, Santa Maria, Brazil. E-mail: lvalandro@hotmail.com (Dr LF Valandro).

Short title: 20 Hz loading frequency as an alternative for fatigue testing

Highlights

- The influence of cyclic loading frequency on fatigue properties was evaluated.
- Loading frequency does not affect the fatigue behavior of lithium disilicate ceramic.
- Loading frequency does not affect the fatigue behavior of polymer infiltrated ceramic.
- Loading frequency does not influence the failure pattern.
- The use of 20Hz frequency is validated to accelerate fatigue tests on such materials.

Abstract

This study aimed to evaluate the influence of loading frequency on the fatigue mechanical behavior of adhesively cemented polymer-infiltrated ceramic-network (PICN) and lithium disilicate (LD) simplified monolithic restorations. Thirty (30) disc-shaped specimens ($\varnothing=10\text{mm}$; thickness=1.0mm) of each ceramic material (PICN – Enamic, Vita Zahnfabrik or LD – IPS e.max CAD, Ivoclar Vivadent) were produced and adhesively cemented onto dentin analogue discs made of fiber and epoxy resin material ($\varnothing=10\text{mm}$; thickness=2.0mm). PICN and LD cemented assemblies were randomly allocated into 2 groups ($n=15$) according to the loading frequency used for the fatigue testing (20 Hz or 2 Hz), composing the PICN_20, PICN_2, LD_20 and LD_2 testing groups. Fatigue tests were run using the step-stress approach (initial load=200 N; step-size=100 N; 10,000 cycles per step) and the collected data (fatigue failure load – FFL and number of cycles for failure – CFF) were analyzed by survival tests (Kaplan Meier and Mantel-Cox) and Weibull analysis. Fractographic analysis of failed specimens were also performed. No statistically significant differences were detected in relation to FFL and CFF between the groups within the same ceramic material (PICN_20: 1127 N / 102,667 cycles = PICN_2: 1120 N / 102,000 cycles; LD_20: 980 N / 88,000 cycles = LD_2: 900 N / 80,000 cycles). All failures were radial cracks in the cementation surface. Therefore, the use of a 20 Hz loading frequency shows to be a viable alternative to accelerate cyclic fatigue tests without affecting the fatigue mechanical behavior and the failure pattern of simplified restorations made of lithium disilicate glass ceramic or polymer infiltrated ceramic network bonded to the dentin analogue.

Keywords: Glass-ceramics. Hybrid ceramics. Fatigue phenomena. Mechanical cycling. Fatigue testing parameters.

1. Introduction

In order to attend the high aesthetic demand of the patients, new ceramic materials for metal-free restorations have been developed combining esthetics, biocompatibility and better mechanical properties (Bajraktarova-valjakova et al., 2018). In general, dental glass ceramics stand out for its excellent aesthetics, translucency, high strength, biocompatibility, wear resistance and, in certain cases, these materials are bioactive (Kargozar et al., 2019; Montazerian and Zanotto, 2017).

Lithium disilicate (LD) glass-ceramic has been widely used due to an association of factors which enable excellent aesthetic results (glass matrix), high resistance (adding 70% of the volume by elongated and interlocked crystals) (Guess et al., 2011; Kruzic et al., 2018; Zogheib et al., 2011) and thus versatility for clinical indications (Tysowsky, 2009). Another material which has been expanding its use in clinical restorations is the polymer-infiltrated ceramic-network (PICN). PICN allegedly has similar mechanical (elastic modulus and hardness) and aesthetic characteristics to the dental structure, since it combines composite resin characteristics with those of a glass ceramic (He and Swain, 2011). In fact, the development of PICN was tailored to diminish its brittleness and consequently its risk to fracture and antagonist tooth wear (Belli et al., 2017; Wendler et al., 2018, 2017). Despite this, both materials (LD and PICN) are still considered brittle materials, and fracture is one of the commonly reported technical problems (Sailer et al., 2015; Spitznagel et al., 2020).

In vitro tests to evaluate the mechanical behavior of restorative materials should simulate different aspects of the oral environment in order to produce failure modes close to those seen clinically (Kelly, 1999). The mechanical failure of dental restorations occurs after years in function, indicating a failure due to fatigue instead of acute overload through a monotonic test (Zhang and Lawn, 2004). Thus, fatigue tests are essential to predict the long-term mechanical behavior of restorative materials, as they simulate a mechanical scenario closer to what occurs clinically (Wiskott et al., 1995). The load in fatigue tests is cyclically applied with a previously defined frequency (number of cycles per second) for a specified number of cycles. Some studies have been conducted with low loading frequencies such as 1-2Hz (Belli et al., 2013; El Zhawi et al., 2016; Paula et al., 2015), since it is an approximation of the clinical masticatory frequency (Po et al., 2011). However, low frequency fatigue tests at 2 Hz may become very time consuming. Thus, the use of high loading frequencies to enable faster data acquisition is always a must for the scientific community.

High loading frequencies (such as 20 Hz) make in vitro tests more feasible and drastically reduce the time needed to perform them, which would enable testing a greater number of specimens over a high number of cycles and consequently increase the clinical relevance of the results. However, higher fatigue failure load values were observed with a frequency of 20 Hz compared to 2 Hz in a glass-infiltrated alumina ceramic (Kelly et al., 2010). Furthermore, Fraga et al. (2016) stated that it is possible to accelerate the fatigue tests of a Y-TZP ceramic using frequencies up to 20 Hz without influencing the fatigue strength data. Regarding glass-ceramics, it has been reported that loading frequency does not influence the fatigue strength nor the time necessary to promote failure in lifetime studies (Fraga et al., 2020; Joshi et al., 2014). Thus, this theme is still

unanswered in literature and more studies are encouraged to assess if loading frequency can be increased without influencing the acquired data.

Considering the absence of studies supporting the use of higher frequencies in fatigue tests of adhesively cemented PICN or LD ceramic restorations and the relevance of this topic for accelerating fatigue testing, the present study aimed to assess whether it is possible to accelerate fatigue tests by increasing loading frequency without influencing the fatigue mechanical behavior of these restorations. The study assumed a null hypothesis that there would not be a difference in the fatigue behavior between 2 and 20 Hz loading frequencies for both materials.

2. Material and methods

The materials used in this study, their chemical composition and manufacturers are described in Table 1.

2.1 Specimen preparation

The specimens were produced using a prior validated method which uses a simplified tri-layer setup (ceramic, cement and epoxy resin adhesively luted to one another) to simulate a monolithic restoration of a posterior molar tooth (Chen et al., 2014).

CAD/CAM blocks of PICN (Vita Enamic, VITA Zahnfabrik, Bad Säckingen, Germany) and LD ceramics (IPS e.Max CAD, Ivoclar Vivadent; Schaan, Liechtenstein) were shaped into cylinders using a diamond drill (internal diameter= 10 mm; Diamant Boart, Brussels, Belgium) coupled to a bench drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) under refrigeration. Next, the cylinders were sectioned under water-cooling in a cutting machine (Isomet 1000; Buehler, Lake Bluff, USA), resulting in 30 discs of each ceramic with an initial thickness of 1.1 mm and 10 mm in diameter. Both surfaces of all discs were ground (EcoMet/AutoMet 250, Buehler) with grit SiC papers (#400, #600 and #1200, 3M; Sumare, Brazil), obtaining a final thickness of 1.0 ± 0.02 mm. Additionally, the LD ceramic discs were crystallized in a specific oven (Vacumat 6000 MP, VITA Zahnfabrik) according to the manufacturer's guidelines.

The epoxy resin discs were obtained from 2.0 mm thick epoxy resin plates ($150 \times 350 \times 2.0$ mm; Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany) with a diamond drill in the same aforementioned way for the CAD/CAM blocks, obtaining 60 epoxy resin discs ($\varnothing = 10$ mm; thickness = 2.0 mm).

The ceramic discs were cleaned in an ultrasonic bath with isopropyl alcohol (5 min) prior to the surface treatments, while the epoxy resin discs were cleaned with distilled water (5 min).

2.2 Cementation procedure

The cementation surfaces of ceramic discs were treated as recommended by the respective manufacturer (Table 2), washed with air/water spray for 30 s, and cleaned in an ultrasonic bath with distilled water for 5 min. Afterwards, the coupling agent (Monobond N, Ivoclar Vivadent) was applied for 15 s on the treated surface and left to react for 45 s (for a total of 60 s), as recommended by the manufacturer.

The cementation surface of dentin analogue discs was etched with 10% hydrofluoric acid (Condac Porcelana, FGM, Joinville, Brazil) for 60 s (Kelly et al., 2010), followed by washing with air/water spray (30 s) and ultrasonic cleaning (5 min) with distilled water. After the etching procedure, Multilink A and B Primers (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed onto the treated surfaces (30 s), and air-dried until a thin layer was obtained.

Each ceramic disc was adhesively cemented to an epoxy resin disc with resin cement (Multilink Automix, Ivoclar Vivadent) under a constant load of 2.5N for 10 min. The resin cement excesses were removed and the assemblies were light-cured (1200 mW/cm², Radian-Cal, SDI, Bayswater, Australia) for five exposures of 20 s each surface (0°, 90°, 180°, 270° and occlusal surface). All specimens were stored in distilled water (37°C) for at least 24 h up to a maximum period of 7 days before performing the mechanical fatigue tests.

After the cementation procedure, the specimens of each ceramic material were randomly allocated (www.randomizer.org) into 2 groups (n= 15) according to the frequency used for the fatigue test (2 Hz or 20 Hz) (Table 2).

2.3 Step-stress fatigue test

The cemented assemblies (n= 15) were subjected to the step-stress fatigue approach in an electric machine (Instron ElectroPuls E3000, InstronCorp, Norwood, USA). Cyclical intermittent loads were applied with a 40 mm diameter stainless steel hemispheric piston (Kelly et al., 2010) under distilled water at a frequency of 2 Hz or 20 Hz. An adhesive tape (110 µm) was placed on the occlusal surface of the cemented assembly and a thin sheet of a non-rigid material (cellophane, 2.50 µm) was placed between the piston and the ceramic surface to reduce the stress concentration contact (Kelly, 1999). An initial load of 200 N per 10,000 cycles and incremental steps of 100 N per 10,000 cycles were applied until failure, being considered as a radial crack. The specimens were checked for cracks at the end of each step by light oblique transillumination (Dibner and Kelly, 2016). When failure was detected, the fatigue failure load (FFL) and the number of failure cycles (CFF) were recorded for further statistical analysis.

2.4 Fractographic analysis

The specimens were analyzed after the fatigue tests in a stereomicroscope to verify the type of macroscopic fracture. Representative samples (n= 1) were selected from each material. The specimens were longitudinally sectioned into two halves, perpendicularly to the radial crack direction with a diamond blade under water-cooling (Isomet 1000, Buehler). The fragments were subsequently ultrasonically cleaned with distilled water, gold sputtered and analyzed under scanning electron microscopy (SEM - Vega3, Tescan, Czech Republic) at 1,000× magnification (Secondary Electrons detector, 20 Kv acceleration voltage) to determine the fractographic characteristics.

2.5 Data analysis

FFL and the CFF data were subjected to survival analysis by Kaplan Meier and Mantel-Cox Log Rank test using IBM SPSS software (IBM, Armonk, NY, USA), and the survival rates relative to each testing step

were also tabulated. Additionally, FFL and CFF data were submitted to Weibull analysis using the Super SMITH Weibull 4.0k-32 software program (Wes Fulton, Torrance, United States) under the maximum likelihood method to describe structural reliability (Weibull modulus) of each tested condition. The representative failure pattern of the evaluated conditions was qualitatively analyzed.

3. Results

No statistically significant differences were detected for the load frequencies tested in PICN or LD restorations for FFL and CFF, as shown in Table 3. The Weibull analysis also showed no statistically significant difference between the frequencies in each material (Table 3).

Table 4 summarizes the survival rates of the restorations for FFL and CFF, while Fig. 1 shows the survival curves for the two aforementioned parameters.

Regarding the fractographic analysis, SEM images showed that the failures during the fatigue tests were radial cracks from the cementation interface (cement-ceramic), propagating parallel to the load application direction (Figure 2).

4. Discussion

The findings of the present study show that it is possible to accelerate the fatigue tests by a frequency of 20 Hz without affecting the fatigue mechanical behavior and fractographic features of an adhesively cemented PICN or LD ceramic restorations. Therefore, the assumed null hypothesis was accepted.

As already mentioned, the desire to accelerate fatigue tests is a necessity to make them more feasible and drastically reduce the time needed to perform them. However, this should be done without compromising the validity of the acquired data and the failure pattern observed in restorations submitted to a clinical environment (Nelson, 1980), meaning radial cracks originating on the intaglio ceramic-cement surface (Kelly et al., 2010, 1989; Thompson et al., 1994).

The existing non-consensual data in literature regarding the effect of testing frequencies are clear. Rosentritt et al. (2006) showed that the increase in the loading frequency (1.6 and 3 Hz) did not influence the fracture strength of LD all-ceramic fixed-partial dentures. Likewise, in evaluating the effect of loading frequencies (2 and 10 Hz) in a lifetime prediction of a fluorapatite glass-ceramic, Joshi et al. (2014) showed that the time to failure was not significantly different when applying 2 Hz or 10 Hz, which indicates that the use of a higher frequency did not optimize data collection in the lifetime test for a glass-ceramic. Similar results were reported for a leucite-reinforced glass-ceramic, in which the use of higher loading frequencies (such as 20 Hz) did not save time, since a higher number of cycles was necessary to promote the failure when compared to 2 Hz (Fraga et al., 2020). On the other hand, Kelly et al. (2010) investigated 2, 10 and 20 Hz frequencies in evaluating adhesively cemented glass-infiltrated alumina ceramic discs, finding an increase in the fatigue failure load when 20 Hz was used. Furthermore, Fraga et al. (2016) found similar results in investigating 2, 10, 20 and 40 Hz frequencies on the fatigue strength of a yttrium stabilized zirconia up to 20 Hz, with higher values when the 40 Hz frequency was applied.

The microstructure of the material undergoes important changes during cyclic loading due to friction between crack walls caused by the change in the stress concentrated in the crack tip from tensile to compression (Jian et al., 1993). In addition, it is known that the microstructure of a ceramic material influences their properties and the way that a crack propagates into its core (Chevalier et al., 2007; Kelly et al., 2017; Kruzic et al., 2018). The crack propagation in LD ceramics only occurs within the glass matrix, and the main toughening mechanism during crack growth is crack deflection and branching by the randomly oriented interlocked crystals (Apel et al., 2008). Meanwhile, crack propagation occurs through the continuous reinforced-glass phase, and the interpenetrated resin matrix is responsible to deflect and bridge the growing cracks in PICN material (Coldea et al., 2013a, 2013b). For polycrystalline ceramics (tested by the aforementioned studies - Kelly et al., 2010; Fraga et al., 2016), there is a combination of such mechanisms further allied with a potential transformation toughening by crystal phase transformation when stimuli is applied, which hinders crack propagation even more (Kelly et al., 2017; Kruzic et al., 2018). Thus, it is logical to assume that the testing frequency may trigger different effects depending on the material being evaluated and its respective susceptibility to slow-crack growth mechanisms.

All scenarios explored in the present study exceed the loads found clinically, which according to Kohyama et al. (2004) and Schindler et al. (1998) ranges from 20 to 140 N. It is important to note that the herein presented values were obtained through an accelerated fatigue test, where fatigue is induced in a faster and more reliable way. In these accelerated tests, the samples are subjected to more severe test conditions (stress levels higher than those found clinically) in order to accelerate the failure, while it is important to maintain the failure modes that occur clinically (Nelson, 1980). Fact that can be confirmed by the failure mode found in the fractographic analysis (radial cracks initiating from the cementation surface, underneath the load application point), which is described as the main failure reported in clinically failed ceramic crowns (Kelly et al., 2010, 1989; Thompson et al., 1994).

Finally, the data herein shows that it was possible to drastically reduce the time required (approximately 90%) for the fatigue tests using a frequency of 20 Hz rather than 2 Hz without changing the fatigue behavior of both simplified ceramic restorative materials tested (LD and PICN) and their fracture pattern. For instance, the time consumed to test one specimen of PICN reduced from 13h50min at 2 Hz to 1h20 min at 20 Hz, while for LD the time reduced from 1h at 2 Hz to 1h12min at 20 Hz. Therefore, although there is a decrease in the contact time of the piston with the materials tested using a frequency of 20 Hz, this decrease has no influence on the fatigue mechanical behavior.

It is pertinent to emphasize that the results of the present study were obtained through an accelerated fatigue method (step-stress) and the extrapolation of using higher frequencies for other fatigue testing methodologies requires prior assessment of each context, even though the same findings would probably be obtained. In addition, the results are limited to the evaluation of PICN and LD ceramic restorations, and therefore prior evaluation is required to indicate high loading frequencies for other ceramics.

5. Conclusion

The use of a 20 Hz frequency during fatigue testing shows to be a viable alternative to accelerate the test without interfering with the accessed fatigue mechanical behavior and the resulting failure pattern of lithium disilicate or polymer-infiltrated ceramic restorations.

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TABLES

Table 1. Description of materials, commercial name, manufacturer, composition and batch number.

Material	Commercial name/manufacturer	Composition	Batch number
Lithium disilicate glass ceramic	IPS e.Max CAD, Ivoclar Vivadent, Schaan, Liechtenstein	SiO ₂ 57-80%, Li ₂ O 11-19%, K ₂ O 0-13%, P ₂ O ₅ 0-11%, ZrO ₂ 0-8%, ZnO 0-8%, other and colouring oxides 0-12%.	W93126
Polymer infiltrated ceramic network	Vita Enamic, VITA Zahnfabrik	SiO ₂ 58-63%, Al ₂ O ₃ 20-23%, Na ₂ O 6-11%, K ₂ O 4-6%, B ₂ O ₃ 0,5-2%, other and/or colouring oxides 0-1%.	51880
Ceramic primer coupling agent	Monobond N, Ivoclar Vivadent	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate.	Y19262
5% hydrofluoric acid	IPS Ceramic Etching Gel, Ivoclar Vivadent	< 5% hydrofluoric acid.	W14921
10% hydrofluoric acid	Condac Porcelana, FGM, Joinville, Brazil	< 10% hydrofluoric acid.	010819
Dual cure resin cement	Multilink N, Ivoclar Vivadent	Silicate glass, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments.	W44613
Primer	Multilink Primer (A and B), Ivoclar Vivadent	Primer A: water, initiators; primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabilizer.	Primer A: W88902 Primer B: W44494
Epoxy resin	Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany	Continuous filament woven fiberglass bonded with epoxy resin.	-

*The chemical composition is described according to the manufacturers' information.

Table 2. Experimental design.

Group	Material	Loading Frequency	Surface treatment*
PICN_20	Polymer-infiltrated ceramic-network (PICN)	20 Hz	Hydrofluoric acid 5% for 60 s
PICN_2		2 Hz	
LD_20	Lithium disilicate ceramic (LD)	20 Hz	Hydrofluoric acid 5% for 20 s
LD_2		2 Hz	

*Recommended by the manufacturer.

Table 3. Mean fatigue failure loads (FFL) in Newtons, number of cycles for failure (CFF), and Weibull modulus of each evaluated condition, with their respective 95% confidence interval (CI).

Groups	FFL		CFF	
	Mean (CI)*	Weibull modulus (CI)**	Mean (CI)*	Weibull modulus (CI)**
PICN_20	1127 (1078 – 1175) ^A	12.33 (8.12 – 17.35) ^A	102,667 (97,803 – 107,531) ^A	11.27 (7.42 – 15.87) ^A
PICN_2	1120 (1034 – 1206) ^{AB}	8.86 (5.57 – 13.12) ^{AB}	102,000 (93,403 – 110,597) ^{AB}	8.05 (5.05 – 11.92) ^{AB}
LD_20	980 (884 – 1076) ^{BC}	5.68 (3.74 – 7.91) ^B	88,000 (78,398 – 97,602) ^{BC}	5.13 (3.38 – 7.17) ^{BC}
LD_2	900 (817 – 983) ^C	6.23 (4.04 – 8.91) ^{AB}	80,000 (71,663 – 88,338) ^C	5.56 (3.60 – 7.96) ^C

Different letters indicate statistical differences on each column for each considered outcome.
 * Kaplan-Meier and Mantel-Cox (log-rank) tests for FFL and CFF.
 ** Maximum -likelihood estimations for Weibull analysis, based on the absence of CI overlapping.

Table 4. Survival rates – probability of specimens to exceed the respective fatigue failure load (FFL) and number of cycles for failure (CFF) step without crack propagation, and its respective standard error values.

Groups	FFL (N) / CFF													
	200 / 10,000	300 / 20,000	400 / 30,000	500 / 40,000	600 / 50,000	700 / 60,000	800 / 70,000	900 / 80,000	1000 / 90,000	1100 / 100,000	1200 / 110,000	1300 / 120,000	1400 / 130,000	1500 / 140,000
PICN_20	1	1	1	1	1	1	1	1	0.80 (0.10)	0.33 (0.12)	0.13 (0.09)	0.00 (0.00)	-	-
PICN_2	1	1	1	1	1	1	0.87 (0.09)	0.87 (0.09)	0.67 (0.12)	0.53 (0.13)	0.27 (0.11)	0.00 (0.00)	-	-
LD_20	1	1	1	1	0.93 (0.06)	0.87 (0.09)	0.87 (0.09)	0.60 (0.13)	0.27 (0.11)	0.13 (0.09)	0.07 (0.07)	0.07 (0.07)	0.00 (0.00)	-
LD_2	1	1	1	1	1	0.73 (0.11)	0.60 (0.13)	0.40 (0.13)	0.20 (0.10)	0.07 (0.06)	0.00 (0.00)	-	-	-

* The symbol ‘-’ indicates absence of specimens being submitted to the respective category.

FIGURES

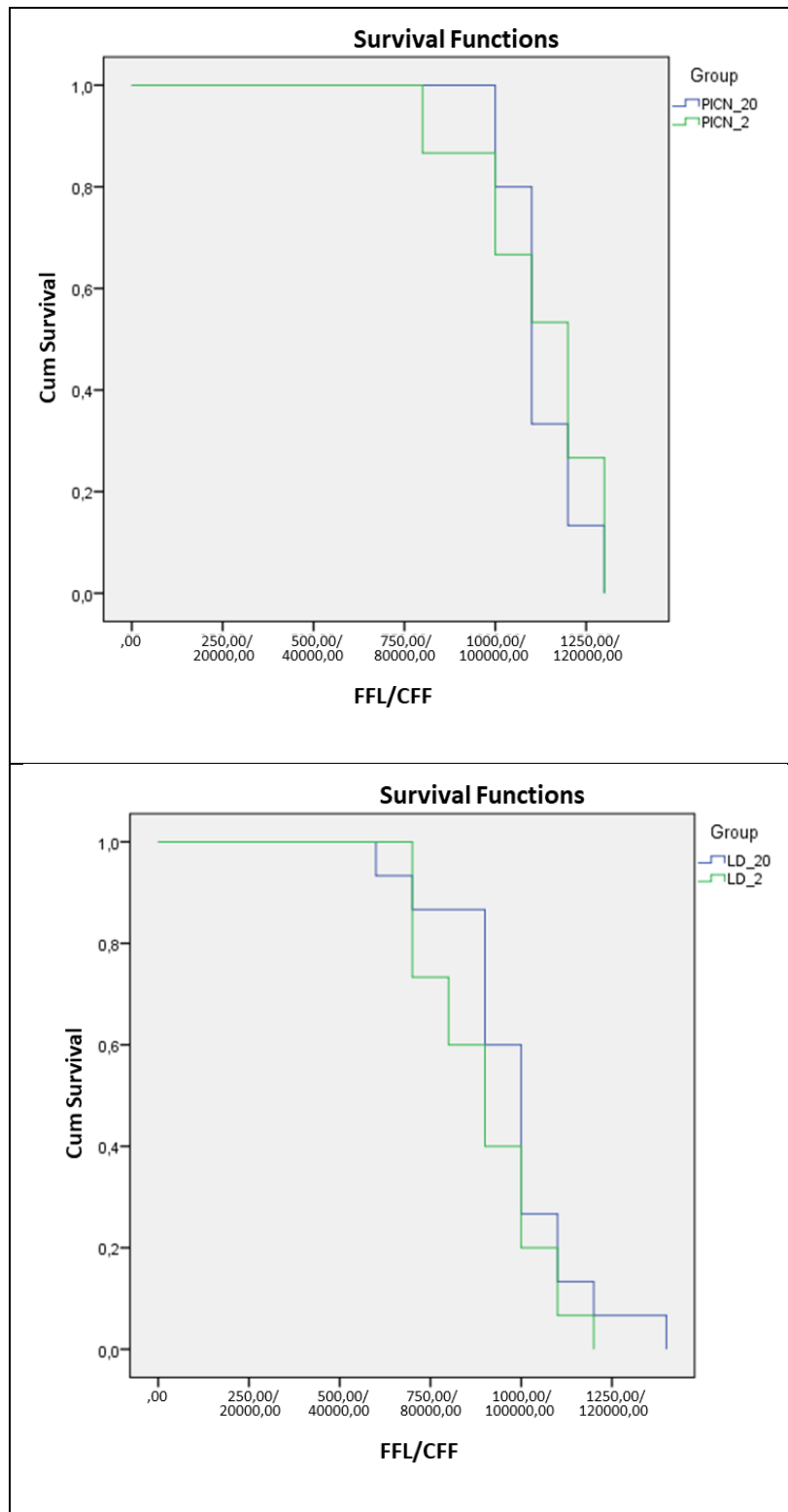


Figure 1. Survival curves according to the steps of fatigue failure loads (FFL) in Newtons, number of cycles for failure (CFF) in which each disc failed, obtained by Kaplan–Meier and Log-rank tests.

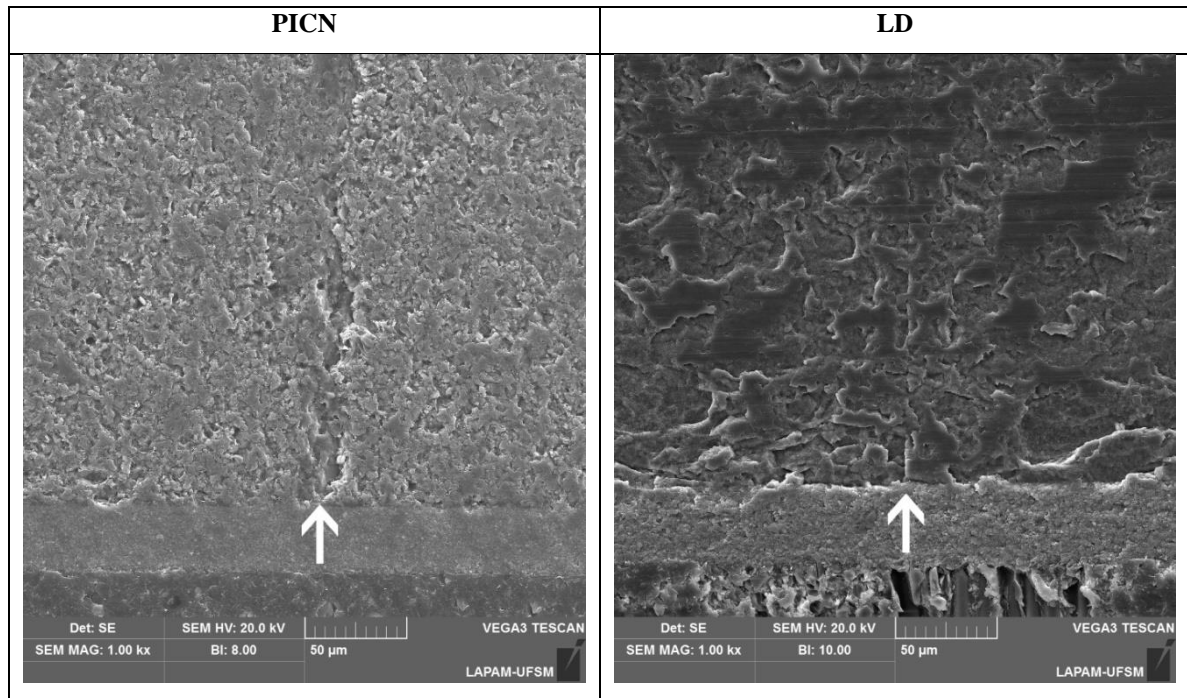


Figure 2. Micrographs obtained by SEM at 1000× magnification illustrating that all failures originated at cementation interface, region pointed by white arrows, which then propagated in a direction parallel to the load application into the opposite side surface.

4 DISCUSSÃO GERAL

Os estudos *in vitro* de materiais restauradores conferem ao pesquisador a possibilidade de isolar fatores e abordar parâmetros específicos que podem influenciar no desfecho estudado. Por outro lado, também podem permitir a adoção de cenários de testes mais complexos, que tentam simular muitos dos fatores a que uma restauração pode ser exposta no ambiente clínico (BONFANTE; COELHO, 2016). Assim, é importante que o pesquisador considere qual desenho experimental e método de teste são mais adequados para que os resultados consigam responder ao objetivo do estudo, seja isolando ou associando os fatores de estudo. Neste sentido, esta dissertação se propôs a investigar os parâmetros de testes adotados nos ensaios de fadiga cíclica de materiais restauradores.

Em um primeiro momento, uma revisão de escopo foi realizada com o propósito de identificar os parâmetros de teste, métodos e características usadas para induzir fadiga cíclica em espécimes não anatômicos de materiais restauradores dentários. Por meio dessa revisão foi possível observar que há uma grande heterogeneidade nos parâmetros de teste e metodologias que têm sido adotados para conduzir ensaios de fadiga cíclica em materiais restauradores dentários. Portanto, é importante discutir as implicações metodológicas e a aproximação com a realidade clínica.

A maioria dos estudos incluídos na presente revisão avaliaram os materiais restauradores através de geometrias de teste de flexão (resistência flexural 3-pontos, 4-pontos ou biaxial), empregadas para caracterização, avaliação da confiabilidade dos materiais ou avaliação de fatores específicos (métodos de processamento ou tratamento de superfície) e influência nas propriedades do material (KELLY et al., 2017). Já as geometrias de teste utilizando materiais restauradores cimentados a um substrato visam a simulação de um cenário mais próximo ao cenário clínico e diferentes distribuições de tensão podem ser esperadas, dependendo do módulo de elasticidade dos materiais restauradores e do substrato (DAL PIVA et al., 2018; FACENDA et al., 2019; OTTONI et al., 2018). Além disso, pode ser avaliado o papel da cimentação adesiva no comportamento mecânico à fadiga das restaurações quando submetidas a diferentes métodos de processamento ou tratamentos de superfície, por exemplo.

Quanto aos métodos de fadiga, métodos acelerados como *Staircase*, *Stepstress* ou *Boundary* foram os mais adotados pelos estudos, visto que são uma alternativa para obtenção dos dados de uma forma mais rápida e confiável (KELLY et al., 2017). No

entanto, é importante conhecer suas características e limitações. O método *Staircase* determina o valor médio do limite de fadiga do material para uma determinada vida útil (número de ciclos). No entanto, o número pré-definido de ciclos a que o espécime é exposto limita previsões em diferentes números de ciclos e este teste não é conduzido em uma ampla faixa de carga ou valores de estresse extremos, o que poderia mascarar o desempenho nos piores cenários (BONFANTE; COELHO, 2016). Por outro lado, cada corpo de prova no método *Stepstress* é submetido a níveis crescentes de carga, o que significa que após um certo período (número de ciclos) a carga é aumentada passo a passo até sua falha ou término dos perfis de teste. Este método considera o efeito cumulativo das tensões aplicadas, estimando a probabilidade de sobrevivência ao longo do tempo e prevendo a confiabilidade para um período específico (METTAS; VASSILIOU, 2002; NELSON, 1980). Já no método *Boundary*, dois grupos de corpos de prova são avaliados para cada número de ciclos determinados, usando dois níveis de tensão, um correspondendo a uma baixa probabilidade de falha e outro com uma amplitude de tensão correspondendo a uma alta probabilidade de falha. Uma vantagem desta metodologia é que os dados obtidos podem ser usados para prever tensões correspondentes a baixas probabilidades de falha, fornecendo mais precisão na estimativa da faixa de tensões de interesse clínico (HUYSMANS et al., 1992; MAENNIG, 1975; ZHANG; GRIGGS, 2003).

Após a definição da geometria de teste e do método de fadiga, o delineamento dos estudos ainda engloba a definição dos parâmetros de teste a serem empregados. Para isso, deve ser levado em consideração a microestrutura do material, o mecanismo de propagação de trincas e como cada um desses parâmetros influenciará na indução à fadiga cíclica.

O material utilizado para a confecção do aplicador de carga (pistão), formato e dimensões da ponta ativa são fatores que podem influenciar o modo de falha e distribuição de tensões durante o carregamento (KELLY, 1999). No entanto, este parâmetro não foi relatado pela maioria dos estudos. É importante observar que, materiais com alto módulo de elasticidade tendem a induzir maior pressão de contato e, conseqüentemente, maior concentração de tensões, podendo causar danos de contato na superfície do material, que não são observadas com frequência nas falhas clínicas (KELLY et al., 2010). Neste sentido, uma análise fractográfica criteriosa é fundamental para determinar o padrão de falha gerado.

Por outro lado, parâmetros como ambiente de teste úmido e análise fractográfica foram adotados pela maioria dos estudos, demonstrando o cuidado dos autores da aproximação com o cenário clínico. Sabe-se que a presença de umidade pode influenciar os fenômenos de fadiga por meio de um processo de degradação química (KELLY et al., 2017), o que reforça a necessidade de um ambiente úmido durante o teste. Adicionalmente, a realização de uma análise fractográfica é essencial para caracterizar as falhas observadas, servindo para elucidar a origem e causas da falha, as condições de carregamento e a direção de propagação da trinca (SCHERRER et al., 2017). Dependendo da geometria de teste, diferentes padrões fractográficos podem ser encontrados. Neste sentido, espera-se que as trincas em corpos de prova testados em configurações de ensaio de flexão comecem de defeitos na superfície de tração (parte inferior) em direção à superfície de compressão (parte superior), onde uma curvatura de compressão é encontrada (SCHERRER et al., 2017). Já nas geometrias de teste cimentadas que simulam um cenário mais próximo ao das restaurações clínicas, modos de falha devem mimetizar aqueles que são relatados clinicamente. Dentre eles, trincas iniciando na superfície da interface de cimentação oposta à aplicação de carga (KELLY et al., 2010) e na região da margem da coroa, justaposta ao término do preparo da restauração (ØILO et al., 2014). Defeitos introduzidos pelo processamento em ambos os cenários seriam responsáveis por desencadear concentrações de tensões durante o carregamento mecânico que induziriam a coalescência desses defeitos e subsequentemente propagação catastrófica de trincas (ZHANG; SAILER; LAWN, 2013). Outra região responsável pela origem das falhas é a superfície oclusal (SAILER et al., 2009). Nesta região, as falhas partiriam de defeitos introduzidos por procedimentos de usinagem CAD/CAM e/ou ajuste oclusal, que fazem parte dos processos de fabricação e adaptação/ajuste das restaurações (DELLA BONA; KELLY, 2008; TAUFER; DELLA BONA, 2019).

Em relação a frequência de carregamento, frequências próximas a 2 Hz foram as mais adotadas pelos estudos, o que influencia diretamente no tempo necessário para realização dos ensaios de fadiga cíclica. Neste contexto, alguns estudos têm avaliado o uso que frequências de carregamento mais altas (10, 20 e 40 Hz) como alternativa para acelerar os ensaios de fadiga em materiais cerâmicos como zircônia, leucita, alumina infiltrada por vidro e fluorapatita (FRAGA et al., 2016, 2020; JOSHI et al., 2014; KELLY et al., 2010). No entanto, a degradação mecânica ocasionada pelo atrito entre as paredes da trinca durante o carregamento cíclico, devido à mudança na tensão concentrada na

ponta da trinca de tração para compressão (JIAN; ZHIHAO; XIAOTIAN, 1993), pode ser influenciada pela frequência de carregamento adotada pelo ensaio de fadiga cíclica, visto que o tempo disponível para abertura e fechamento da trinca pode variar de acordo com a frequência adotada. Além disso, as diferentes microestruturas dos materiais cerâmicos irão influenciar em suas propriedades e nos mecanismos de propagação de trincas (CHEVALIER; GREMILLARD; DEVILLE, 2007; KELLY et al., 2017; KRUZIC et al., 2018).

Assim, o segundo estudo da presente dissertação teve como proposta avaliar se é possível acelerar os ensaios de fadiga aumentando a frequência de carregamento sem influenciar o comportamento mecânico à fadiga de restaurações de cerâmica infiltrada por polímeros (PICN) e de dissilicato de lítio (LD). Os resultados demonstraram que uma frequência de carregamento de 20 Hz é uma alternativa viável para acelerar os ensaios de fadiga sem afetar o comportamento mecânico e as características fractográficas em ambas as cerâmicas estudadas. Assim, foi possível reduzir o tempo necessário (aproximadamente 90%) para os testes de fadiga usando uma frequência de 20 Hz em vez de 2 Hz. Por exemplo, o tempo consumido para testar um corpo de prova de PICN foi reduzido de 13h50min a 2 Hz para 1h20min a 20 Hz, enquanto para o LD o tempo foi reduzido de 11h a 2 Hz para 1h12min a 20 Hz. Portanto, embora seja observado uma diminuição no tempo de contato do pistão com os materiais ensaiados na frequência de 20 Hz, essa diminuição não teve influência no comportamento mecânico à fadiga dos materiais testados.

As limitações dos estudos da presente dissertação incluem principalmente a grande heterogeneidade entre os estudos incluídos em relação aos materiais considerados, os formatos dos espécimes, características de teste, parâmetros e métodos adotados. No entanto, embora permita uma discussão e orientação para estudos futuros, a ausência de estudos primários comparando a influência de tais fatores na aquisição de dados de fadiga dificulta a indicação de um ou outro parâmetro. Além disso, os resultados do estudo *in vitro* são limitados à avaliação de restaurações de cerâmica PICN e LD e, portanto, uma avaliação prévia é necessária para indicar altas frequências de carregamento para outros materiais restauradores.

5 CONSIDERAÇÕES FINAIS

Com base nas investigações científicas apresentadas nos artigos, pode-se concluir que:

- Configurações de teste de flexão, carregamento axial e ensaios de fadiga acelerada são os mais empregados para induzir fadiga cíclica em espécimes não anatômicos de materiais restauradores dentários. No entanto, a definição da geometria de teste, direção de carregamento e do método de fadiga deve estar relacionada ao objetivo do estudo.

- Frequências de carregamento abaixo de 2 Hz são as mais utilizadas. Apesar disso, frequências de carregamento mais elevadas (ou seja, 20 Hz) são uma alternativa para acelerar os ensaios de fadiga cíclica de materiais como a cerâmica Y-TZP, cerâmica infiltrada por polímeros e cerâmica de dissilicato de lítio.

- Pistões confeccionados com diferentes materiais e formatos/dimensões da ponta ativa são descritos na literatura. Porém, seu uso não deve alterar o modo de falha descrito para a geometria de teste adotada após execução de uma cuidadosa análise fractográfica.

- Um ambiente de teste úmido foi descrito na maioria dos estudos, visto que é uma ferramenta importante para simular a degradação hidrolítica dos materiais observada em cenários clínicos.

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ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *MATERIALS SCIENCE AND ENGINEERING C: MATERIALS FOR BIOLOGICAL APPLICATIONS*

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