UNIVERSIDADE DE PASSO FUNDO

Viviane Cantelli

RESISTÊNCIA À FADIGA DE ZIRCÔNIAS CIMENTADAS APÓS DIFERENTES TRATAMENTOS DE SUPERFÍCIE

Passo Fundo

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RESISTÊNCIA À FADIGA DE ZIRCÔNIAS CIMENTADAS APÓS DIFERENTES TRATAMENTOS DE SUPERFÍCIE

Tese apresentada ao Programa de Pós-Graduação em Odontologia da Faculdade de Odontologia da UPF, para obtenção do título de Doutor em Odontologia – Área de Concentração em Clínica Odontológica, sob orientação do prof. Dr. Alvaro Della Bona.

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Viviane Cantelli nasceu na cidade de Montauri, no Rio Grande do Sul, em outubro de 1994. Formou-se em Odontologia pela Faculdade Especializada na Área da Saúde do Rio Grande do Sul (FASURGS) em 2016. Em 2021 obteve o título de Mestre em Odontologia com área de concentração em Clínica Odontológica pela Universidade de Passo Fundo (UPF), apresentando a dissertação intitulada “Comportamento biomecânico de material para base de prótese total fabricada por impressão 3D”.

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LISTA DE ABREVIATURAS

CAD - *computer aided design* (desenho assistido por computador)
CAM – *computer aided manufacturing* (fabricação assistida por computador)
CP – corpo de prova
EDS - analise composicional por espectroscopia de raios X por energia dispersiva
Hz – Hertz
MEV - microscopia eletrônica de varredura
mm – milímetros
MPa – mega Pascal
N - Newton
s – segundos

RESUMO[1]

O presente estudo teve como objetivo (1) revisar, sistematicamente, a literatura com relação aos métodos de fadiga utilizados para avaliar zircônias translúcidas, correlacionando as falhas àquelas clinicamente reportadas e (2) avaliar a taxa de sobrevivência à fadiga de cerâmicas à base de zircônia unidas a cimento resinoso após diferentes tratamentos de superfície. Uma revisão sistemática foi conduzida sem restrição de tempo de publicação nas bases de dados PubMed e Scopus, seguindo o protocolo PRISMA-P e registrado no PROSPERO. Para o ensaio de fadiga,estruturas de zircônia (3Y-TZP e 5Y-PSZ) foram confeccionadas e cimentados sobre G10 após diferentes tratamentos de superfície (usinagem por CAD CAM, silicatização e jateamento com partículas de óxido de alumínio). O ensaio de fadiga foi realizado com uma carga de 120 N, aplicada à superfície cerâmica com um pistão esférico de metal de aço inoxidável (diâmetro 6 mm), com frequência de 3 Hz em água destilada a 37ºC. O teste foi interrompido após 10^4, 10^5, 5×10^5, 10^6 e $1.5\ x10^6$ ciclos e a presença ou ausência de falha foi observada utilizando transiluminação. Este estudo contribui para o entendimento dos métodos de avaliação de fadiga em zircônias translúcidas e sua correlação com falhas clínicas, além de fornecer informações sobre a influência dos tratamentos de superfície na resistência à fadiga.

Palavras-chave: Zircônia translúcida, Odontologia, Materiais dentários, Fadiga.

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ABSTRACT[2]

The present study aimed to (1) systematically review the literature regarding fatigue methods used to evaluate translucent zirconia, correlating failures with those clinically reported and (2) evaluate the fatigue survival rate of zirconia-based ceramics bonded to resin cement after different surface treatments. A systematic review was conducted with no limit on publication time using the PubMed and Scopus databases, following the PRISMA-P protocol and registered in PROSPERO. For the fatigue test, structures made of different types of zirconia (3Y-TZP and 5Y-PSZ) were manufactured and cemented on G10 after different surface treatments (CAD-CAM machining, silicatization and airborne particle abrasion using aluminum oxide particles). The fatigue test was carried out with a load of 120 N, applied to the ceramic surface with a spherical stainless steel metal piston (diameter 6 mm), and a frequency of 3 Hz in 37ºC distilled water. The test was momentarily stopped after 10^4, 10^5, 5×10^5, 10^6 and 1.5 $x10^6$ cycles to evaluate for failure using transillumination. This study contributes to the understanding of fatigue assessment methods in translucent zirconia and its correlation with clinical failures, in addition to providing information on the influence of surface treatments on the fatigue resistance.

Keywords: Translucent zirconia, Dentistry, Dental Materials, Fatigue.

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[2] Fatigue in translucent zirconia

INTRODUÇÃO

Um dos principais desafios da reabilitação oral é alcançar resultados satisfatórios relacionados a função e a estética. Para isso, é importante o entendimento das propriedades de cada material disponível, permitindo assim a seleção mais adequada para enfrentar cada desafio clínico específico. As primeiras zircônias parcialmente estabilizadas por ítria passaram por diferentes estratégias para resolver a deficiência relacionada as propriedades ópticas, buscando proporcionar maior translucidez e a possibilidade do uso em restaurações monolíticas (ZHANG *et al*., 2016; ZHANG E LAWN, 2018) e como resultado foi desenvolvido as zircônias translúcidas.

A utilização clínica da zircônia translúcida (GARDELL et al., 2021) representa um significativo avanço na odontologia restauradora, pois apresenta capacidade de proporcionar uma estética satisfatória aliada a boas propriedades mecânicas. É crucial compreender o comportamento mecânico e possuir conhecimento detalhado das propriedades para assegurar a durabilidade e eficácia clínica dos tratamentos cerâmicos. Isso inclui investigar os efeitos e alterações superficiais e estruturais resultantes dos tratamentos de superfície necessários para a cimentação adesiva de restaurações à base de zircônia monolítica, permitindo prever seu desempenho clínico.

Clinicamente, as restaurações dentárias precisam suportar os desafios da cavidade bucal, como variações de pH, umidade, mudanças de temperatura, tensões induzidas pela carga mastigatória e hábitos parafuncionais. Assim, as restaurações ficam suscetíveis à corrosão química e a mecanismos de fadiga, que podem reduzir a resistência do material e sua longevidade (Della Bona, 2009).

Para compreender os desafios intra-orais, diversos testes laboratoriais estão disponíveis para avaliar o comportamento mecânico de materiais, sendo os testes de fadiga essenciais para simular as condições do ambiente oral. Esses testes visam prever possíveis falhas por "fadiga" (WISKOTT *et al*., 1995; BARAN *et al*., 2001), como fraturas, desgaste ou deformações (BARAN *et al*., 2001), quando os materiais e estruturas são submetidos a cargas e estresse ao longo do tempo. Nesse sentido, esse estudo foi proposto para investigar os métodos de avaliação de fadiga em zircônias translúcidas e a correlação com falhas clínicas, além de fornecer informações sobre a influência dos tratamentos de superfície na resistência à fadiga das restaurações.

PROPOSIÇÃO

Esse estudo foi delineado para analisar o comportamento a fadiga de zircônia translúcida tendo como objetivos:

- Avaliar, de forma sistemática, a literatura com relação aos métodos de fadiga utilizados, associando as falhas resultantes com as reportadas clinicamente, testando a hipótese de que independentemente do método de fadiga utilizado, as falhas resultantes dos ensaios de fadiga são semelhantes as reportadas clinicamente.
- Avaliar a taxa de sobrevivência à fadiga de cerâmicas a base de zircônia (3Y-TZP e 5Y-PSZ) unidas ao cimento resinoso após diferentes tratamentos de superfície (usinagem por CAD CAM, silicatização e jateamento com partículas de óxido de alumínio), testando as hipóteses de que (1) os tratamentos de superfície resultam em taxa de sobrevivência à fadiga semelhante para zircônias monolíticas (3Y-TZP e 5Y-PSZ) unidas ao cimento resinoso e que (2) a 3Y-TZP apresenta maior taxa de sobrevivência à fadiga do que a 5Y-PSZ quando aderidas a um cimento resinoso.

ARTIGO I
FATIGUE METHODS FOR EVALUATING TRANSLUCENT DENTAL ZIRCONIA [3]

Abstract

Objective. To investigate fatigue methods for the evaluation of translucent zirconia and to associate *in vitro* failures with clinically reported ones.

Data. Studies published in English that used fatigue tests on dental translucent zirconia.

Sources. Two databases (MEDLINE/PubMed and Scopus) were electronically searched without any restriction on year of publication.

Study selection. A total of 4555 studies were identified. After removal of duplicates (78) and irrelevant articles (4316) that did

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not meet the inclusion criteria, 161 articles were considered eligible based on their titles and abstracts. These articles were fully read, leading to the inclusion of 41 studies in the review**.**

Results. The most widely used fatigue method for evaluation of translucent zirconia was step-stress (18 articles), followed by staircase (seven articles), and step-wise (two articles). Most studies had been conducted in a wet environment with the use of a stainless steel piston to apply load to cemented structures on a dentin-like substrate. Most fracture analyses indicated the fracture originated on the cementation or contact surface where the load was applied. Moreover, studies that utilized anatomical structures (dental crowns) reported fractures starting at the cervical margin of the crowns.

Conclusion. Most studies used the step-stress method. Only three studies reported failures similar to those found in clinical trials that used translucent zirconia restorations.

Clinical Significance. The study findings can assist on correlating clinical failures to the ones observed in vitro.

Keywords: Translucent zirconia, Dentistry, Dental Materials, Fatigue.

1. Introduction

Dental ceramics have been widely used in dental restorations, requiring constant research and development for the qualification of materials and fabrication methods. *In vitro* studies using structures that mimic serviceable conditions, such as fatigue tests, are crucial to show trends in mechanical behavior and structural longevity [1–9].

Zirconia-based ceramics, or simply zirconias, are broadly used in the health sector, including dentistry, owing to their high biocompatibility and mechanical properties [1, 10]. In addition to zirconium dioxide (ZrO_2), they contain a stabilizing agent, often yttrium oxide (Y_2O_3), which provides them with distinct mechanical properties when compared to other dental ceramics [1]. Consequently, their fatigue behavior differs from that of other dental ceramics [5].

The first partially yttria-stabilized zirconias (3Y-TZP) underwent different strategies to correct deficiencies associated with optical properties, with the aim of enhancing translucency and allowing for their use in monolithic restorations [11, 12]. Translucent zirconias were then developed, showing advantages such as reduced tooth preparation, smaller wear on the antagonist when compared to initially marketed zirconias, simpler procedures than those required for the fabrication of multilayered restorations on opaque zirconia structures, elimination of defects from ceramic veneers, and shorter fabrication time, while maintaining biocompatibility and good mechanical properties [10]. Evidently, this innovation urged researchers to investigate the various aspects of this new material, but the mechanical behavior in service is of utmost importance [5, 13–15]. The assessment of materials and structures under fatigue plays a key role in predicting their clinical behavior [5, 15, 3, 4. 6]

Numerous fatigue methods have been employed to assess translucent zirconias, but no critical analysis has been performed to verify the application of such methods in clinical practice. This study systematically reviewed papers in which fatigue methods were used to assess translucent zirconias, describing important results and providing information on the parameters used so as to evaluate fatigue methods, correlating *in vitro* failures with clinical ones and testing the hypothesis that, regardless of the fatigue method, those failures resulting from fatigue tests would be similar to those reported in clinical practice.

2. Materials and Methods

This review was conducted systematically to answer the following research questions: “What are the most widely used fatigue methods and parameters to evaluate translucent zirconia?” “Are *in vitro* fatigue failures similar to clinically reported failures?”

2.1. Protocol and registry

The protocol for this study followed the PRISMA-P statement for systematic reviews [16] and was registered in PROSPERO under ID CRD42024504747 on February 3, 2024.

2.2. Eligibility criteria

This review included studies published in English that carried out fatigue tests on translucent zirconia for use or potential use in restorative dentistry. Studies on other materials or other types of zirconia, studies on materials and/or structures not applied to restorative dentistry, literature reviews, manufacturer’s reports, commentaries, and conference abstracts were excluded from this review.

2.3. Sources of information and search strategy

Two databases (MEDLINE/PubMed and Scopus) were electronically searched without any restriction on year of publication. The last search occurred on February 22, 2024. The search strategy was based on PubMed MeSH terms and adapted for each database.

2.4. Selection of sources of evidence

The search was initially performed using the Mendeley reference software. Two researchers (V.C. and M.T.M.) identified the articles independently by analyzing their titles and abstracts for relevance and eligibility criteria. The retrieved records were classified as “include,” “exclude,” or “uncertain.” Full-text articles from the “include” and “uncertain” records were selected for later eligibility screening by the same independent researchers. Discrepancies in the screening of titles/abstracts and full-text articles were resolved through discussion. In case of disagreement, a third reviewer (A.D.B.) was consulted for their opinion.

2.5. Data tabulation

Those articles that met the inclusion criteria were critically evaluated by two independent reviewers (V.C. and A.D.B.). After selection, the following information was collected from each article:

- Publication details: authors, year of publication, and journal.

- Characteristics of *in vitro* studies.

- Type of translucent zirconia assessed.
- Details about the fatigue method.

2.6. Synthesis of results

This review primarily examined the fatigue methods used to evaluate translucent zirconias in dentistry, correlating *in vitro* failures with clinical failures. Clinical studies on monolithic zirconia crowns showing structural failures indicated that the fracture had originated on the cervical margin of the crowns [17, 18].

This study presents a descriptive analysis, taking the study design into consideration, as well as the characteristics of the materials (translucent zirconias) and the failure method used (Table 1).

3. Results

Figure 1 shows the study flowchart. Initially, 4,555 potentially relevant articles were retrieved (PubMed: n = 3,027; Scopus: n = 1,528). After removal of duplicates (78) and irrelevant articles (4,316) that did not meet the inclusion criteria, 161 articles were considered eligible based on their titles and abstracts. These articles were fully read, leading to the inclusion of 41 studies in the review.

Different types of specimens were employed for fatigue analysis. Out of the 41 studies included in this review, 21 assessed anatomical specimens, such as conventional single-unit crowns [19–35], endocrowns [36, 37], and fixed bridges [38, 39]. The remaining studies (n= 20) utilized non-anatomical specimens, such as discs [40–53], bars [54–57, 15,], and square plates [58].

Figure I.1: Study flowchart according to the PRISMA statement.

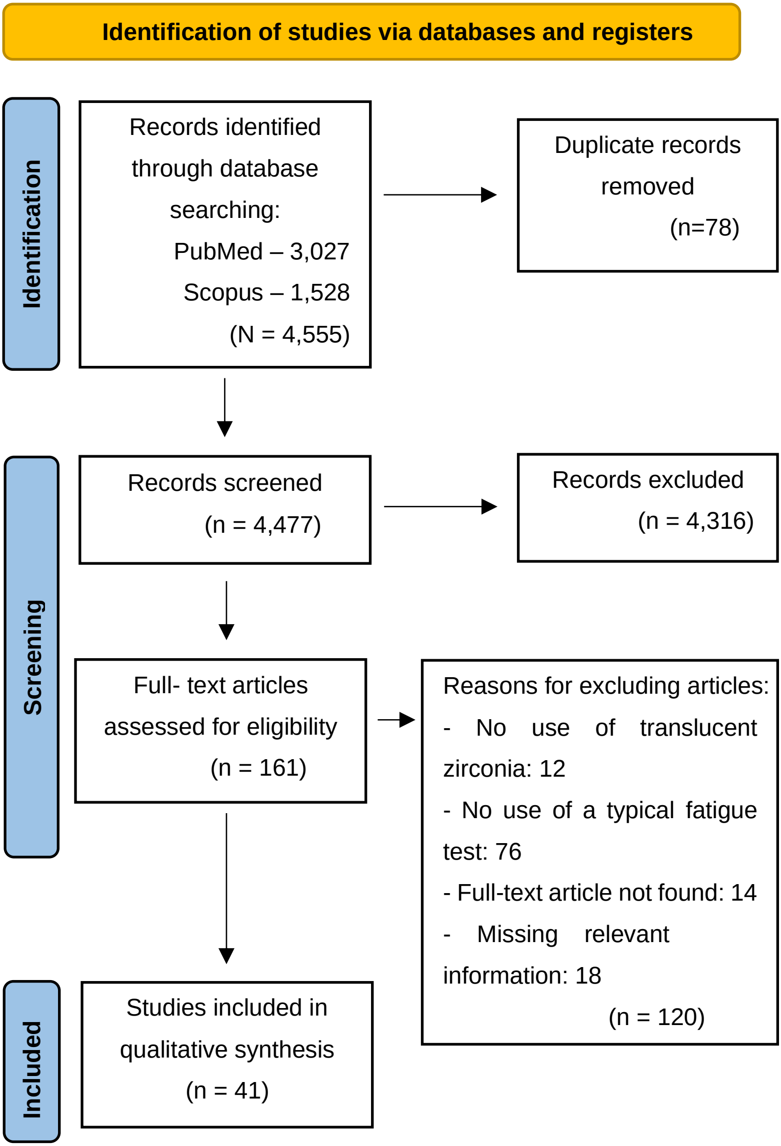

Table I.1: Studies in alphabetical order, reporting the fatigue method used to evaluate translucent dental zirconias.

Study	Objective	Translucent zirconia	Fatigue parameters
Adabo *et al.*, 2023 [19]	To assess the probability of survival of crowns made with a 3Y-TZP, a 5Y-TZP, and a lithium disilicate	5Y-TZP (Prettau Anterior, Zirkonzahn, Bruneck, Italy) 3Y-TZP (Prettau Zirkonzahn, Bruneck, Italy)	Step-stress fatigue method, at 10 Hz, using three load profiles: mild, moderate, and aggressive. Each profile started at 300 N and finished at 3,000 N. The mild profile increased the load (200 N) every 20,000 cycles up to 320,000 cycles; the moderate profile increased the load (250 N) every 15,000 cycles up to 195,000 cycles; and the aggressive profile increased the load (300 N) every 10,000 cycles up to 110,000 cycles.

Alves *et al.*, 2021 [20]	To evaluate and compare the fatigue performance of monolithic crowns manufactured from glass or polycrystalline CAD-CAM ceramic systems adhesively luted to a dentin analog.	Translucent yttrium fully stabilized polycrystalline zirconia (Trans YZ, Prettau Anterior).	Step-stress fatigue, in water, with a stainless steel sphere (Ø = 40 mm) loading at 20 Hz starting from 200 N for 5,000 cycles to adjust the testing assembly, followed by sequential increments with a step size of 100 N at each 10,000 cycles until failure.
Antón *et al.*, 2023 [53]	To examine the effect of high-speed sintering and the preshading of blanks (monochrome versus multilayer) on the fatigue behavior of 4Y-TZP ceramics.	4Y-TZP (Zolid DRS) 4Y-TZP (Zolid Gen-x) 4Y-TZP (Ceramill Zolid HT+PS)	Five different dynamic fatigue test conditions (P1-P5), at 10 Hz, in dry conditions. P1-P3 used step-stress protocols: P1- 50 N for 5,000 cycles; P2- 10 N for 1,000 cycles; and P3- 5% for 5,000 cycles. P4 used constant force (720 N). P5 used different constant load levels.

Antunes *et al.*, 2018 [21]	To evaluate the effect of different surface treatments on the marginal misfit and retentive strength between Y-TZP crowns and an epoxy resin.	Y-TZP ceramic (IPS e.max ® ZirCAD Ivoclar-Vivadent/ Schaan, Liechtenstein)	A total of 2×10^6 thermomechanical cycles were performed, with a stainless steel piston (Ø = 4 mm), at 4 Hz, with 100 N load. A total of 5,555 thermal cycles (5 °C – 55 °C, in a 60-s cycle, 30-s dwel time) per group.
Arcila *et al.*, 2021 [40]	To characterize the microstructure of three yttria partially stabilized zirconia ceramics and to compare their hardness, indentation fracture resistance (IFR), biaxial flexural strength (BFS), and fatigue flexural strength.	3Y-TZP (Vita YZ HT) 4Y-PSZ (Vita YZ ST) 5Y-PSZ (Vita YZ XT)	Step-stress fatigue test under distilled water, using a flat circular tungsten piston (Ø = 1.6 mm), at 20 Hz, with a minimum tension of 10 MPa and the maximum tension desired for each stage of the cycle. Initial stress (200 MPa) for 5,000 cycles, then additional incremental steps of 25 MPa for 10,000 cycles starting from 400 MPa until specimen fracture.

Auzani *et al.*, 2020 [41]	To evaluate the effect of shading procedures on the fatigue performance and optical properties of an yttria-stabilized tetragonal zirconia polycrystal ceramic (4Y-TZP)	4Y-TZP (IPS e.max ZirCAD MT BL, Ivoclar-Vivadent AG, Schaan, Liechtenstein)	Step-stress fatigue, in water, with a flat circular tungsten piston (Ø = 1.6 mm), at 20 Hz, with initial strength of 100 MPa for 5,000 cycles to accommodate piston/specimen, followed by additional incremental steps of 25 MPa for 10,000 cycles/steps starting from 200 MPa, until complete failure.

Barbosa-Júnior *et al.*, 2020 [22]	To inspect the mechanical fatigue behavior of an implant-supported restorative system using polyether ether ketone (PEEK) and yttria partially stabilized zirconia polycrystals (YZ) as materials for customized definitive implant-supported hybrid abutments, supporting two types of all-ceramic restorations: translucent zirconia (TZ) and lithium disilicate (LD) monolithic crowns	Highly translucent zirconia polycrystals (Prettau Anterior)	Step-stress fatigue method, the load was applied on the tooth palatal surface at a 45-degree angle using a stainless steel ball (Ø = 6mm), in water, at 10 Hz. Each profile started at 5,000 cycles on a relatively low load (200 N). Subsequently, 10,000 cycles were performed at progressive load levels (step-size) of 50 N starting at 250 N until failure detection.

Benalcazar Jalkh *et al.*, 2024 [23]	To evaluate the reliability and failure modes of ultrathin (0.5 mm) lithium disilicate, translucent and ultra-translucent zirconia crowns for posterior teeth restorations.	3Y-TZP (Zirconn Translucent, Vipi) 5Y-PSZ (Cercon XT, Dentsply Sirona)	Step-stress fatigue method, in water, by sliding a tungsten-carbide indenter (Ø = 0.7 mm) at a frequency of 2 Hz, using three different load profiles until failure or survival, ranging from 50 N up to 700 N.
Cadore-Rodrigues *et al.*, 2021 [42]	To evaluate the effect of distinct surface treatments on the fatigue behavior (biaxial flexural fatigue testing) and surface characteristics (topography and roughness) of a 5Y-PSZ ceramic.	5Y-PSZ (IPS e.max ZirCAD MT Multi, Ivoclar Vivadent, Schaan, Liechtenstein)	Step-stress fatigue test under water, at 20 Hz, starting with a load of 100 N for 5,000 cycles to adjust the sample/piston contact, then followed incrementing steps of 50 N for 10,000 cycles at each load step until failure.

Dal Piva *et al.*, 2021 [24]	To evaluate the effect of minimal tooth preparation on the mechanical behavior, reliability and translucency of posterior monolithic ceramic crowns.	High translucency zirconia HT (Vita Zahnfabrik, Bad Säckingen, Germany)	Step-stress fatigue test, in water, with stainless steel (Ø = 6 mm) piston at 10 Hz. Samples randomly distributed into three profiles: mild, moderate, and aggressive in the ratio of 3:2:1, according to the load increase and number of cycles (load profiles starting at 200 N and finished at 1,500 N) until failure.
De Carvalho *et al.*,2022 [43]	To evaluate the effect of four different finishing procedures on the fatigue strength of a fully stabilized zirconia (5Y-FSZ) material.	5Y-FSZ (Katana UTML, Kuraray Noritake)	Staircase fatigue method, at 20 Hz, for 10,000 cycles. The first specimen was tested at 60% of stress level in the monotonic test, with subsequent step sizes of 5%, either added or subtracted according to the survival or failure, respectively.

Demachkia *et al.*,2023 [36]	To evaluate the effect of the remaining tooth structure and different CAD/CAM materials on the fatigue performance and failure mode of endodontically treated premolars restored with endocrowns.	Ultra-translucent zirconia 5Y-PSZ (KATANA UTML)	Stepwise fatigue method, in distilled water, with a cylindrical stainless steel piston (Ø = 6 mm), at 20 Hz, with initial load of 200 N, and a constant increment of 100 N, with 10,000 cycles per step until fracture.
Elraggal *et al.*, 2023 [55]	To investigate the effect of acidic media, including beverages and gastric fluids on flexural strength and fatigue of CAD-CAM materials	High translucent zirconia (Ceramill Zolid HT+)	The fatigue was conducted using a total of 10^6 cycles, in distilled water, of 50 N loaded a sliding plate for 0.5 s and then leveled off for another 0.5 s so that the total duration of each fatigue cycle was 1 s.

Fraga *et al.*, 2016 [44]	To investigate the influence of the loading frequency on the zirconia fatigue strength.	(IPS e.max ZirCAD, Ivoclar Vivadent, Liechtenstein)	Staircase fatigue method was determined for 500,000 cycles, with a steel piston flat tip (Ø = 1.4 mm). Sinusoidal loading was applied with amplitude ranging from 10 MPa to the maximum tensile stress. Four different frequencies were tested: 2 Hz (control group), 10 Hz, 20 Hz, and 40 Hz.
Freitas *et al.*, 2023[a] [45]	To evaluate the surface roughness, translucency, fatigue failure load, and number of cycles for fatigue failure of an advanced lithium disilicate and three other ceramics for monolithic restorations	Y-PSZ (IPS e.max ZirCAD MT, Ivoclar)	Step-stress fatigue method using a cylindrical steel piston hemisphere (Ø = 40 mm), in water, at 20 Hz, loads starting at 200 N per 5,000 cycles were initially applied, then load increments of 50 N every 10,000 cycles until specimen failure.

Freitas *et al.*, 2023b [46]	To evaluate the surface properties and fatigue mechanical behavior of an advanced lithium disilicate ceramic in comparison to lithium disilicate and zirconia.	4Y-PSZ (IPS e.max ZirCAD)	Step-stress fatigue test in water, with a flat circular tungsten piston (Ø = 1.6 mm), at 20 Hz, for an initial stress of 100 MPa for 5,000 cycles, then 200 MPa for 10,000 cycles. Thereafter, increments of 25 MPa were added every 10,000 cycles until specimen failure (fracture).
Guilardi *et al.*, 2020 [58]	To evaluate the effect of surface roughness (polished vs. CAD/CAM milling roughness simulation) on the fatigue behavior of monolithic dental ceramic restorations manufacture d by CAD/CAM.	YZ (yttria-stabilized tetragonal zirconia polycrystal - VITA YZ T)	Stepwise fatigue method, in distilled water, using a cylindrical steel piston with flat tip (Ø = 1.6 mm), at 20 Hz, starting with a load of 20 N for 5,000 cycles, followed by constant load increases of 20 N per step (i.e. 40, 60, 80, 100 …) at a maximum of 10,000 cycles each step until fracture of the specimen.

Hassouneh *et al.*, 2020 [37]	To evaluate the post-fatigue load-to-failure and failure modes of endodontically treated premolar teeth restored with endocrowns fabricated from different CAD/CAM materials.	Monolithic translucent zirconia material, Initial® Zirconia Disks HT (GC Dental, Europe).	The test was carried out using dynamic fatigue method, in distilled water at 37 °C, with spherical tungsten carbide piston (Ø = 6 mm), at 5 Hz, for 600,000 cycles, with 10-50 N at 45° angle to the long axis of the tooth.
Holman *et al.*,2020 [56]	To evaluate the flexural strength (FS) degradation of newer zirconia materials compared to more traditional tetragonal zirconia materials.	3Y-TZP (Lava Plus, 3M ESPE); 4Y-PSZ (Katana STML, Kuraray); 5Y-PSZ (Katana STML, Kuraray; 5Y-PSZ (Lava Esthetic, 3M ESPE)	Staircase fatigue method, at 2 Hz, for 6,000 cycles. Initial load was 50% of the maximum FS of the material. Stress amplitude used 50% of the standard deviation of the maximum FS. Tests were conducted sequentially with applied load values increasing or decreasing by 20% according to the result (failure or survival) from the previous specimen.

Jerman *et al.*, 2021 [15]	To test the impact of three step-stress protocols on the fatigue behavior of two 3Y-TZP, one 4Y-TZP and one 5Y-TZP zirconia materials.	3Y-TZP - (T: translucent); 4Y-TZP - (ET: extra translucent); 5Y-TZP - (HT: high translucent). Materials are from Pritidenta (Germany)	Three step-stress fatigue protocols (P1-P3): P1- Load increased by 50 N every 5,000 cycles until fracture; P2- Load increased by 5% every 5,000 cycles until fracture; P3- Load increased by 10 N every 1,000 cycles until fracture.
Jurado *et al.*, 2023 [25]	To determine the fracture resistance of chairside CAD-CAM zirconia crowns for a mandibular first molar without occlusal rest and with four different rest seat designs	Y-PSZ (IPS e.max Zir-CAD MT for CEREC, Ivoclar Vivadent)	Cyclic loading was performed with the specimens immersed in room-temperature water, for a total of 200,000 load cycles, at 1 Hz with a load of 20 N.

Kashkari *et al.*, 2019 [26]	To compare the integrity of zirconia, lithium disilicate, and zirconia-reinforced lithium silicate CAD-CAM crowns after being subjected to cyclic loading and then subjected to static loading until fracture.	Prettau Zirconia, Zirkonzahn	The fatigue test was performed using steel ball (Ø = 5 mm), in distilled water at 37 ºC. Three specimens from each group were loaded for 10,000 cycles, and the other three specimens were loaded for 50,000 cycles, using 250 N maximum compressive load at 1,000 N/s loading rate and at 2 Hz.
Lawson *et al.*, 2019 [27]	To determine if surface treatment and cement selection affect the fracture load of traditional zirconia (3Y-PSZ), translucent zirconia (5Y-PSZ), or lithium disilicate crowns.	3Y-PSZ (Lava Plus; 3M ESPE, St. Paul, MN). 5Y-PSZ (Lava Esthetic; 3M ESPE)	The fatigue test was performed using a stainless steel ball (Ø = 8 mm), in water at 24 °C, at 1 Hz, with load of 100 N for 100,000 cycles.

Li *et al.*, 2023 [57]	To evaluate the fatigue behavior of zirconia specimens with microgrooved surfaces formed by femtosecond laser is reported	3Y-TZP blocks (Zenostar T, Wieland, Germany)	Staircase fatigue method. Loading was applied at 10 Hz for 10^6 cycles. The load increment was 5% of the peak value (60% of the fracture strength), increasing or decreasing according to the survival or failure of the previous sample. Eight pairs of specimens with opposite results (fracture or survival) were observed.
Li *et al.*, 2024 [28]	To evaluate the effect of low-temperature degradation on the fatigue performance of the novel "strength & shade gradient" multilayered zirconia restorations.	Multilayered zirconia (Meiying functional multilayered zirconia, UPCERA, China) 3YZir (ST, UPCERA, China) 5Y-Zir (TT, UPCERA, China)	Step-stress fatigue method, with tungsten steel ball indenter (Ø = 13 mm) in artificial saliva environment at 37 °C, at 10 Hz, with a minimum load of 6.3% and a maximum load of 11.3% for the first step, and 1×10^4 cycles for each step. For unfractured specimens, the maximum load of each step was increased by 5% increment until crown fracture.

Marchionatti *et al.*, 2019 [47]	To compare file-splitting multilayer (fused and cemented) with monolithic Y-TZP on the fatigue flexural strength and finite element analysis stresses.	Monolithic Y-TZP (IPS e.max ZirCAD - Ivoclar Vivadent)	Staircase fatigue method, in water, at 20 Hz, for 750,000 cycles. Sinusoidal loading started at 60% of the flexural strength. The test was conducted sequentially, increasing or decreasing the maximum applied stress by a fixed load increment (approximately 10% of the initial strength) according to whether the previous tested specimen survived or failed. If the specimen failed before reaching the 750,000 cycles, the stress level was decreased by one step size for the next specimen testing. In case of survival specimen, the stress level was increased by one step size for the next testing specimen.

Machry *et al.*, 2021 [48]	To evaluate the fatigue failure load, number of cycles until failure and survival probability of partially (PSZ) and fully-stabilized (FSZ) polycrystalline zirconia specimens with different thicknesses adhesively cemented onto foundations with distinct elastic moduli.	Zenostar T, Wieland Dental, Ivoclar Vivadent IPS e.Max ZirCAD MT Multi, Ivoclar Vivadent	Step-stress fatigue method using a hemi-spherical stainless steel piston (Ø = 40 mm), in water, at 20 Hz, for 10,000 cycles at each load step, starting with an initial load of 600 N and followed by progressive load levels of 100 N up to 2,800 N or until failure detection.
Marini *et al.*, 2023 [38]	To evaluate the fatigue behavior of strength-graded zirconia polycrystals used as monolithic three-unit implant-supported prosthesis.	Zirconia graded 3Y-TZP/5Y-TZP (IPS e.max® ZirCAD PRIME); Zirconia graded 4Y-TZP/5Y-TZP (IPS e.max® ZirCAD MT Multi)	Step-stress fatigue method, in water, with stainless steel piston rounded active portion (Ø = 6 mm), at 20 Hz, 20,000 cycles were performed at sequential increments of stress levels of 200 N (10% of the mean fracture strength) until failure occurred.

Nakamura *et al.*, 2018 [29]	To develop a clinically relevant load-to-failure test in combination with fatigue treatments involving thermal and mechanical cycling to evaluate the fracture resistance of monolithic zirconia crowns	Lava Plus Zirconia, 3M/ESPE	Mechanical cycling was performed in pure water at 37 ± 1 °C, using an inverse V-shaped two-plane steel indenter with an angle of 160°, at 14.5 Hz, loaded between 50 N and 300 N for 2.4×10^6 cycles.
Nishioka *et al.*, 2018 [49]	To evaluate the fatigue strength of different ceramic materials indicated for monolithic restorations.	High translucent Y-PSZ (YZ HT Vita Zahnfabrik)	Staircase fatigue method, in water for 100,000 cycles at 10 Hz. The first specimen was tested at 60% monotonic load-to-failure. The step size was 5% of the initial stress level, according to survival or failure of the previous specimen.

Packaeser *et al.*, 2023 [50]	To characterize the elastic modulus and Poisson's ratio of a resin cement with distinct viscosities, and to evaluate their impact on the static and fatigue strength of a translucent zirconia (4Y-PSZ) after air-abrasion surface treatment	4Y-PSZ IPS e.max ZirCAD MT, Ivoclar; Schaan, Liechtenstein)	A cyclic load was applied with a cylindrical tungsten piston (Ø = 1.6 mm), in water, at 20 Hz, generating an initial stress of 60 MPa up to 5,000 cycles to adjust the sample/piston contact, followed by increments of 25 MPa at each step of 10,000 cycles until failure.
Pereira *et al.*, 2019 [51]	To evaluate the fatigue behavior of monolithic translucent zirconia polycrystals (TZ) and lithium disilicate glass-ceramic (LD) bonded to different substrates	Translucent zirconia (Prettau Anterior, Zirkonzahn SRL)	Step-stress fatigue method using a hemispherical stainless steel piston (Ø = 6mm), in water, at 20 Hz, initial sinusoidal load ranging from a minimum of 10 N to the maximum load starting at 200 N for 5,000 cycles, followed by step size of 200 N until 2,800 N at a maximum of 10,000 cycles/step. Specimens were loaded until failure or to a maximum of 135,000 cycles at 2,800 N.

Preis *et al.*, 2017 [30]	To investigate the fatigue and fracture resistance of different CAD/CAM materials as implant- or tooth-supported molar crowns with respect to the clinical procedure (screwed/bonded restoration).	IPS e.max ZirCAD, Ivoclar-Vivadent	The fatigue was conducted with steatite balls (Ø = 12 mm), at a frequency of 1.6 Hz (simulated mouth opening: 2 mm), with a load of 50 N for 1.2×10^6 cycles. During mechanical loading specimens were thermally aged (5-55 ºC, 2-min cycle) for 2 × 3,000 cycles in distilled water.
Refaie *et al.*, 2023 [31]	to evaluate the effect of cyclic mechanical loading on the fracture resistance of 3D-printed zirconia crowns in comparison to milled zirconia crowns.	Zircônia IPS e.max ZirCAD LT (Ivoclar Vivadent, Nova York, EUA)	The dynamic mechanical loading was conducted with metal stylus with a 5-mm diameter spherical tip in a small basin connected by a temperature-controlled reservoir of distilled water 37 ± 2 °C, at 2 Hz, for 1.2 million cycles and applied load between 20 and 200 N.

Rosentritt *et al.*, 2020 [32]	Comparison of in-vitro fatigue and wear performance of 3Y-, 4Y-, 5Y-TZP and lithium disilicate ceramic, multilayer/monolayer 4Y-TZP and variation of wall thickness at 5Y-TZP	3Y-TZP-LA (DD Bio ZX^2) 4Y-TZP - multilayer and monolayer (DD cube ONE) 5Y-TZP (DD cubeX^2) - Dental Direkt GmbH Spenge, Germany.	The fatigue was conducted with a steatite sphere (Ø = 12 mm), with a load of 50 N for 1,200,000 cycles and 3,000 thermal cycles (5-55 ºC).
Skjold *et al.*,2020 [33]	To compare the fracture load after artificial short-term aging of monolithic, full-contour zirconia crowns with different amounts of yttria stabilization.	Super high translucent zirconia (5Y-Z - DD cube X^2) High translucent zirconia (3Y-Z - DD Bio ZX^2). - Dental Direkt, Spenge, Germany	The fatigue was conducted using a spherical stainless steel tip (Ø = 3 mm), in water at 37 °C, at 1 Hz, with load of 60-200 N, for 30,000 cycles

Soares *et al.*, 2021 [52]	To evaluate the fatigue behavior (load for failure, cycles for failure, and survival rates) and FEA analysis of different ceramic materials luted over different foundation substrates for implant-supported crowns.	TZ (IPS e.max ZirCAD MT Multi, Ivoclar Vivadent)	Step-stress fatigue method, in distilled water, with hemispherical stainless steel piston (Ø = 40 mm), at 20 Hz, with initial load of 200 N for 5,000 cycles, followed by steps of 10,000 cycles starting at 400 N up to 2,800 N or until failure, step size of 200 N.
Tomm *et al.*, 2023 [34]	To evaluate the fatigue resistance of monolithic zirconia and multilayer ceramic structures using the CAD-on technique in different thicknesses.	Zirconia crowns (Zenostar Zr Translucent, Ivoclar AG)	Step-stress fatigue method, with a stainless steel sphere (Ø = 40 mm), in water, at 20 Hz starting from 200 N for 5,000 cycles to adjust the testing assembly, followed by sequential increments with a step size of 200 N each 10,000 cycles until failure to a maximum load of 2,800 N.

Zacher *et al.*, 2020 [39]	To investigate the in vitro performance and fracture force of anterior implant-supported tooth-colored fixed dental prosthesis.	5Y-FSZ Pritidenta; 4Y-TZP Pritidenta	The fatigue was conducted with steatite balls (Ø = 6 mm), at a frequency of 1.6 Hz (simulated mouth opening: 2 mm), with a load of 50 N for 1.2×10^6 cycles. During mechanical loading specimens were thermally aged (5-55 ºC, 2-min cycle) for 2 × 3,000 cycles in distilled water.
Zimmermann *et al.*, 2020 [35]	To analyze the effect of CAD/CAM fabrication and sintering procedures on the fracture load of monolithic zirconia crowns with different material thicknesses	InCoris TZI C (Dentsply Sirona, York, USA)	Thermomechanical loading was conducted with 1.2 million cycles, 1.7 Hz, invariable occlusal load 49 ± 0.7 N, thermal cycling 5-55 ºC, dwell time 120 seconds, 12,000 cycles, water change time 10 s, human natural molar cusp antagonist.

Zucuni *et al.*, 2017 [53]	To evaluate the effects of different pre-sintering fabrication processing techniques of Y-TZP ceramic (CAD/CAM vs. in-lab), considering surface characteristics and mechanical performance outcomes.	IPS e.max ZirCAD for inLab MO	Typical staircase fatigue method, in water, with a flat circular tungsten piston (Ø = 1.6 mm), at 6 Hz, with 20,000 cycles, sinusoidal loading with amplitude ranging from a minimum of 10 MPa to the maximum force.

4. Discussion

Fatigue is often defined as degradation (wear, weakening) of a structural component subjected to mechanical factors, chemical or biological stress and, in most cases, a combination thereof. Therefore, fatigue in a structure increases over time, given that defects tend to grow with use or service and the relationship between defect size and structural longevity (reliability) becomes exponential. Thus, properties of the materials always degrade over time, and the fatigue parameter reflects how the material performs over time, thereby determining the longevity of a structure [1, 2, 59]. In dental ceramics, with the numerous challenges posed by the oral environment and susceptibility of the material to subcritical crack growth (SCG), ceramic structures are more prone to fatigue failures [59].

In our review, we found studies that perform what is known as sample aging prior to testing specimens with static loading. For instance, a load (e.g., 50 N) is applied by 1 million cycles either with or without thermal cycling before subjecting the specimen to monotonic loading until failure occurs. In such cases, some damage is assumed to accumulate, but it does not always mimic clinical damage.

This assumption of damage accumulation is often discussed in studies that use chewing simulation. Chewing simulators are typically employed to clinically mimic the masticatory process and cause relevant long-term cyclic fatigue outcomes using non-clinical specimens. However, the experimental setups in such approach have to be carefully adjusted to at least create relevant damage accumulation [60, 61]; otherwise, investigators will be wasting their time and leading readers into misinterpretations [2]. The present systematic review excluded studies that employed chewing simulators for sample aging instead of using well-established fatigue methods.

Cyclic fatigue testing has been utilized to assess fatigue resistance and time to failure of ceramic materials and structures, thus seeking to predict their clinical behavior [2]. These tests can be performed using conventional or accelerated methods.

Conventional fatigue tests are time-consuming and require a sizable amount of specimens [7]. As a result, accelerated fatigue tests have been used much more frequently, including the boundary [62], staircase [63], step-stress [64], and stepwise [36, 58] methods.

In the present review, the step-stress method was the most widely employed accelerated fatigue test to assess translucent zirconia [15, 19, 20, 22–24, 28, 34, 38, 40 – 42, 45, 46, 48, 51, 52, 54] ,followed by the staircase method [43, 44, 47, 49, 53, 56, 57] and by the stepwise method [36, 58]. Use of the boundary method for cyclic fatigue testing on translucent zirconia was not found in any of the reviewed articles.

Different types of specimens were used in the fatigue studies included in the present review, such as single-unit crowns [19–35], endocrowns [36, 37], fixed bridges [38, 39], discs [40 – 53], bars [15, 54–57], and square plates [58].

In laboratory mechanical tests, some variables are clinically important. For instance, failure and loads leading to failure are sensitive to water, to the type of luting material, and to bonding to infrastructure materials [65]. In this respect, there seems to be a consensus that cementation of the specimens interferes with the mechanical behavior of ceramics [66, 67]. In this review, most studies conducted the fatigue test on specimens cemented onto some structure [19–37, 39, 45, 48, 50–52]. Standardization of dentin substrates for the cementation of ceramic specimens for mechanical tests is probably the major challenge in this type of experiment. Our systematic review found only three studies that used human tooth dentin as substrate, and two of them utilized endocrowns as restorations [36, 37], whereas one employed crowns on molars [30]. The difficulty in standardizing dentin substrates has led to the search for dentin-like materials. Fiberglass-reinforced epoxy (G10) is the most widely used in laboratory studies [20, 21, 24, 26, 28, 31, 34, 45, 48, 51] to assess fatigue on cemented translucent zirconia. The bond strength of resin cement to wet and dry G10 was compared with the bond strength to dentin, indicating adequate compatibility between G10 and dentin, both in terms of adhesion and elastic behavior. Therefore, G10 has been recommended as dentin substitute in mechanical strength tests [65]. Other studies used infrastructures made from other resin composites [23, 25, 27, 29, 32, 33, 48, 50], methacrylates [35], epoxy resin with aluminum and polyoxymethylene copolymer [29], polyetheretherketone and yttria-stabilized zirconia [52], zirconia [51]; metal die [19], titanium [51], Ni-Cr metal alloy [48], and cementation on implant-abutment analog [22, 30, 39]. Considering the type of substrate, one should note that the higher the stiffness, the higher the load needed for fracture of the ceramic specimen [35]. A stiffer substrate enhances the load-bearing capacity under fatigue of the restoration assembly because it reduces stress concentration on the inner surface of ceramic restorations, thereby reducing the risk of restoration failure. On the other hand, a less rigid base concentrates more stress on the inner surface of ceramic restorations, increasing the likelihood of restoration failure due to radial cracks on this surface [52].

Another important variable that should be considered in fatigue testing is the loading piston. Pistons made from various materials and with different tip shapes have been employed to mimic clinical behavior. The diameter and shape (spherical or flat) of the active tip of the loading piston can induce different mechanical responses in the tested materials [68]. In the clinical setting, the human tooth should be the piston of choice in experiments evaluating the mechanical behavior of dental materials. However, teeth are difficult to obtain and standardize and they are prone to fracture during the test. Therefore, pistons made from different materials (e.g., stainless steel and tungsten), ceramics (e.g., zirconia-based, lithium disilicate-based, and steatite), and composites (e.g., G10) have been investigated. One study assessed different types of load application and recommended the use of a lithium disilicate-based ceramic piston for mechanical testing of ceramics [69].

The present review demonstrated that stainless steel, followed by tungsten and steatite, was the most widely used material in loading pistons. The active tip of stainless steel pistons most typically used is the one with a spherical shape and different diameters: 40 mm [20, 45, 48, 52], 8 mm [25, 27], 6 mm [22, 24, 36, 38, 51], 5 mm [26, 31] and 3 mm [33]. It was also employed in a cylindrical shape with a flat tip of 1.6 mm in diameter [47, 58], 1.4 mm in diameter [44], and in two inverted V-shaped planes [29]. As for tungsten pistons, the flat shape with 1.6 mm in diameter [40–42, 46, 53], the spherical one with 6 mm in diameter [37] and with 13 mm in diameter [28], and the cylindrical one with 1.6 mm in diameter [50] were most frequently used. Regarding steatite pistons, the spherical shape with 6 mm in diameter [39] or 12 mm in diameter [30] was the most widely used one. One study reported on the use of a natural human molar cusp antagonist, with load application to the central fissure [35].

Some information about the pistons, such as shape, material, and diameter, was missing in a few studies [21, 23, 32, 43, 55, 56], whereas no information about the pistons used in fatigue testing was provided by other studies [15, 19, 49, 54, 57].

The types of equipment for fatigue testing of translucent zirconia varied considerably across studies, and ElecrtoPlus E300 (Instron Corporation) was one of the most frequently used [20, 22, 24, 36–38, 40–44, 47, 48, 51–53, 57, 58]. Mechanical tests performed in water show lower strength of ceramic materials when compared to tests conducted under dry conditions, and this is due to the corrosion of ceramic by water molecules, leading to crack growth. This aspect is directly associated with clinical failures of ceramic restorations [1]. Most fatigue tests were performed in a wet environment, such as water [19–25, 27, 29, 30, 32–35, 38, 39, 41, 42, 44, 46–49, 51, 53], distilled water [26, 31, 36, 37, 40, 45, 50, 52, 55, 58] and artificial saliva [28]. Two studies conducted the tests under dry conditions [54, 56], and some studies did not provide any information about that [15, 43, 57].

Note that, even though the results of the fatigue tests do not fully reproduce the oral environment, they can show a trend in the clinical performance of structures in service. Clinical trials that could predict the performance of translucent zirconias and of the type of clinical failure in a more realistic fashion are still needed. Likewise, few clinical trials have reported on the performance and type of failure of any zirconia-based restoration (crowns and bridges) in any type of zirconia.

One study [70] evaluated the clinical performance of monolithic crowns made from translucent zirconia with occlusal reduction between 1.5 and 2.0 mm and found a survival rate of 93.3% at 3 years, with no crown fractures during the study.Another study [71] collected data over a 7.5-year period on 136,944 zirconia-based restorations, of which 93,848 were monolithic, with a fracture rate of 0.54% (n=77,411) for crowns and 1.95% (n=16.437) for bridges. A literature review [72] of nine articles assessed the clinical behavior of single-unit crowns made from monolithic zirconia. Only two articles reported failures associated with cracks or fractures in the restorations [17, 73], but the longest follow-up period had an average duration of 2.1 years, and only one restoration was reported as having been fractured, with no fractrographic analysis performed [73]. A study [17] involving 84 crowns, only one fracture occurred at the cervical margin of the crown after 16 months. Another study [18] also reported fractures in zirconia-based crowns in the cervical margin. These studies [17, 18] were the only ones to report on the use of fracture analysis. In the present review, only three *in vitro* studies reported fractures that were similar to clinical fractures [28, 29, 33]. However, the parameters and methodological variables of these studies are dissimilar, not allowing for any correlation between them. This could then suggest some tendency towards a certain type of fracture, regardless of the fatigue test used. Lack of information about fracture analysis in *in vivo* and *in vitro* studies significantly hinders any evaluation and comparison between the fatigue methods.

Studies that used the step-stress method for assessing crowns reported fractures initiating in the occlusal contact region [19, 28] and on the cementation surface [19, 20, 23, 34]. The study that employed the stepwise method [36] reported fracture originating on the occlusal surface, below the contact with the piston.

Some studies utilized fatigue methods as a means of aging. Consequently, most of them did not report any type of failure during the fatigue tests [21, 25–27, 30–32, 35, 37, 39]. Standardization of *in vitro* studies poses a challenge and calls into question the clinical validity of the data obtained [69]. Limitations of *in vitro* fatigue tests include the lack of certain serviceable conditions and of the oral environment, such as temperature variations and changes in pH, sliding movements, wear patterns, and other biological factors. Nevertheless, *in vitro* studies serve as a valuable alternative for pointing out trends in the behavior and clinical performance of recently developed dental materials [43].

5. Conclusions

Fatigue testing methods for the assessment of translucent zirconia structures vary considerably. Most studies assessed structural fatigue of materials using the step-stress method, but they showed a broad array of parameters utilized in fatigue testing, mainly regarding the load value, duration, and application mode. Notwithstanding the reported clinical failures in translucent zirconia structures, only three *in vitro* studies mentioned similar failures, and the hypothesis of the present study was then rejected. Owing to the small number of clinical trials, probably because of the recent introduction of these materials, correlating the failures observed in *in vitro* studies with clinical failures is not an easy task;

therefore, further studies reporting clinical failures with translucent zirconias are needed.

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Fatigue methods for evaluating translucent dental zirconia

--Manuscript Draft--

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Abstract:	Objective To investigate fatigue methods for the evaluation of translucent zirconia and to associate in vitro failures with clinically reported ones. Data Studies published in English that used fatigue tests on dental translucent zirconia. Sources Two databases (MEDLINE/PubMed and Scopus) were electronically searched without any restriction on year of publication. Study selection A total of 4555 studies were identified. After removal of duplicates (78) and irrelevant articles (4316) that did not meet the inclusion criteria, 161 articles were considered eligible based on their titles and abstracts. These articles were fully read, leading to the inclusion of 41 studies in the review. Results The most widely used fatigue method for evaluation of translucent zirconia was step-stress (18 articles), followed by staircase (seven articles), and step-wise (two articles). Most studies had been conducted in a wet environment with the use of a stainless steel piston to apply load to cemented structures on a dentin-like substrate. Most fracture analyses indicated the fracture originated on the cementation or contact surface where the load was applied. Moreover, studies that utilized anatomical structures (dental crowns) reported fractures starting at the cervical margin of the crowns. Conclusion Most studies used the step-stress method. Only three studies reported failures similar to those found in clinical trials that used translucent zirconia restorations. Clinical Significance The study findings can assist on correlating clinical failures to the ones observed in vitro.
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ARTIGO II

RESISTÊNCIA À FADIGA DE ZIRCÔNIAS CIMENTADAS APÓS DIFERENTES TRATAMENTOS DE SUPERFÍCIE[4]

Resumo

Objetivo. Avaliar a taxa de sobrevivência à fadiga de cerâmicas a base de zircônia (3Y-TZP e 5Y-PSZ) unidas ao cimento resinoso após diferentes tratamentos de superfície (usinagem por CAD CAM, silicatização e jateamento com partículas de óxido de alumínio).

Métodos. Corpos de prova em formado de disco foram obtidos a partir de blocos de zircônia monolítica 3Y-TZP (Vita YZ HT) e a 5Y-PSZ (Vita YZ XT). Os discos foram divididos aleatoriamente em seis grupos, de acordo com o material e tratamento de superfície: controle (3Y-None e 5Y-None), silicatização (3Y-Cojet e 5Y-Cojet) e jateamento com partículas de óxido de alumínio (3Y-

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Al_2O_3 e 5Y-Al_2O_3). Todos foram cimentados em um suporte de resina epóxi reforçada com fibra (G10). O ensaio de fadiga foi realizado utilizando uma carga de 120 N, com frequência de 3 Hz, sendo interrompido para avaliação após 10^4, 10^5, 5×10^5, 10^6 e 1.5 $x10^6$ ciclos e a presença ou ausência de falha foi registrada. Amostras representativas foram avaliadas em microscopia (MEV) e para composição química (EDS). Os dados de fadiga foram analisados usando Kaplan – Meier (log rank) e Holm–Sidak. Os modos de falha foram reportados quantitativa e qualitativamente. A relação entre os modos de falha foi analisada pelo Chi-square test.

Resultados. Não houve diferença significativa para os modos de falha ($p=0,30$) entre todos os grupos. Os tratamentos de superfície mostraram curvas de sobrevivência significativamente diferentes ($p < 0,001$) para a cerâmica 5Y, mas não houve diferença para a cerâmica 3Y ($p = 0,54$). Considerando o tipo de tratamento de superfície, apenas o jateamento com Cojet mostrou uma diferença significativa ($p < 0,001$) entre as cerâmicas 3Y e 5Y.

Conclusão. Exceto quando tratada com silicatização, A resistência a fadiga foi semelhante entre as cerâmicas 3Y-TZP e 5Y-PSZ, exceto quando usado silicatização, que prejudicou o desempenho a fadiga da 5Y-PSZ.

Palavras-chave: Zircônia translúcida, Odontologia, Materiais dentários, Fadiga.

1. Introdução

As cerâmicas a base zircônia utilizadas na odontologia apresentam propriedades mecânicas diferenciadas das demais cerâmicas odontológicas, apresentando na sua composição o dióxido de zircônio (ZrO_2) e um estabilizador, usualmente o óxido de ítrio (Y_2O_3). São amplamente empregadas na odontologia devido à sua excelente biocompatibilidade e propriedades mecânicas [1,2], sendo classificadas em três gerações. A primeira geração, constituída de zircônia tetragonal estabilizada com 3 mol% de ítria, apresenta ótimas propriedades mecânicas, porém é relativamente opaca, levando à necessidade de cobertura estética em restaurações totais [3,4]. As zircônias de segunda geração mantêm a resistência [5], reduzindo o teor de alumina para aumentar a translucidez [6], porém, ainda não atingem a estética desejada para dentes anteriores [7]. A terceira geração, com maior teor de zircônia na fase cúbica [6], oferece maior translucidez ao aumentar o conteúdo de ítria para 4 a 5 mol% [8]. Esses avanços visam produzir restaurações monolíticas estéticas [9, 10], reduzindo o preparo dentário, desgaste do antagonista e tempo de fabricação, mantendo a biocompatibilidade e excelentes propriedades mecânicas [2].

A zircônia translúcida, embora proporcione resultados estéticos satisfatórios, apresenta uma redução na resistência ao lascamento à medida que sua translucidez aumenta devido ao aumento da fase cúbica em relação à fase tetragonal, o que pode comprometer suas propriedades mecânicas [11]. Quanto à adesão, é importante considerar o tratamento de superfície para promover a união com diferentes substratos. Opções como jateamento com partículas de óxido de alumínio e silicatização mostram-se viáveis. O jateamento com alumina promove alterações topográficas que aumentam a área de superfície e a retenção micromecânica, melhorando a adesão na interface da cerâmica com o sistema de cimentação [12-14]. A união promovida pela silicatização ocorre por interação química entre a superfície cerâmica, coberta por sílica, e o material resinoso por meio de um agente bifuncional silano, e também por promover um padrão topográfico favorável, que permite uma união micromecânica ao adesivo. Essas técnicas são essenciais para melhorar a resistência de união e garantir a durabilidade das restaurações cerâmicas [1].

Um dos grandes desafios na reabilitação oral é proporcionar resultados satisfatórios relacionados à função e a estética do paciente. Para alcançar isso, é importante ter conhecimento das propriedades de cada material disponível e poder determinar a melhor escolha para cada desafio clínico. Diversos testes laboratoriais estão disponíveis para avaliar o comportamento mecânico de materiais, sendo os testes de fadiga essenciais para simular as condições do ambiente oral. Esses testes visam prever possíveis falhas por "fadiga" [15, 16], como fraturas, desgaste ou deformações [16], quando os materiais e estruturas são submetidos a cargas e estresse ao longo do tempo. A abordagem mais clinicamente relevante é o método cíclico, apesar de ser mais demorada, proporciona uma melhor compreensão da resposta do material às condições simuladas [17].

A utilização clínica de zircônia translúcida [18], pode apresentar um grande avanço na odontologia restauradora, pois oferece uma estética satisfatória e boas propriedades mecânicas. A compreensão de seu comportamento mecânico e conhecimento das propriedades é crucial para garantir sua durabilidade e eficácia clínica.

Com o atual conhecimento ainda permanecem questionamentos em relação aos efeitos e alterações superficiais e estruturais produzidos por tratamentos de superfície necessários à cimentação adesiva de restaurações à base de zircônia monolítica, para que se possa predizer o desempenho clínico. Assim, o objetivo do presente estudo é avaliar a taxa de sobrevivência à fadiga de cerâmicas a base de zircônia (3Y-TZP e 5Y-PSZ) unidas ao cimento resinoso após diferentes tratamentos de superfície (usinagem por CAD CAM, silicatização e jateamento com partículas de óxido de alumínio), testando as hipóteses de que (1) os tratamentos de superfície resultam em taxa de sobrevivência à fadiga semelhante para zircônias monolíticas (3Y-TZP e 5Y-PSZ) unidas ao cimento resinoso e que (2) a 3Y-TZP apresenta maior taxa de sobrevivência à fadiga do que a 5Y-PSZ quando aderidas a um cimento resinoso.

2. Materiais e Métodos

2.1 Preparação das amostras

Os corpos de prova foram obtidos a partir de blocos pré-sinterizados de zircônia monolítica estabilizada por ítria (Y-TZP) sendo a 3Y-TZP (Vita YZ HT, Vita Zahnfabrik, Alemanha) e a 5Y-PSZ (Vita YZ XT, Vita Zahnfabrik, Alemanha). Os blocos foram fresados para formar cilindros, e depois foram fatiados para produzir os CPs em forma de disco com dimensões finais, após a sinterização, de 1,2 ± 0,2 mm de espessura e 14 ± 2 mm de diâmetro (ISO 6872:2015).

Os CPs foram cimentados em um substrato análogo à dentina (G10- resina epóxi reforçada com fibra - NEMA G10, International Paper, Hampton, SC, USA) de 25 mm de diâmetro com 5 mm de espessura. Partículas de óxido de alumínio (125 µm; Mega ox, Germany) foram usadas para jatear (20 s) a superfície de união do G10, com uma pressão de 2 bar e distância de 10 mm. As superfícies jateadas foram limpas em banho sônico com água destilada (5 min) seguido de jatos de água/ar e secas com ar antes do processo adesivo.

Além do grupo controle, onde a superfície cerâmica foi mantida conforme usinagem em CAM, o preparo da superfície de união das estruturas cerâmicas dos outros dois grupos experimentais foi realizado com jateamento de partículas (pressão= 2 bar, distância= 10 mm) com as seguintes características:

SI- Silicatização, jateamento com partículas de óxido de alumínio cobertas por sílica (CoJet Sand, 3M ESPE, Seefeld, Alemanha) de toda superfície adesiva e limpeza da superfície com jatos de ar; e

PA- Jateamento com partículas de óxido de alumínio (D50 = 125 µm) de toda superfície adesiva e limpeza da superfície jateada em banho sônico com água destilada (5 min) seguido de jatos de água/ar e secas com ar antes do processo adesivo.

O sistema adesivo (Primers A e B, Ivoclar Vivadent, Schaan, Liechtenstein) foi aplicado na superfície jateada de G10 e a superfície de união das cerâmicas foi tratada com silano (Monobond S, Ivoclar Vivadent, Schann, Liechtenstein) por 3 min e seca com ar por 10 s. O cimento resinoso dual contendo monômero de fosfato (MDP) (Multilink N, Ivoclar Vivadent, Schaan, Liechtenstein) foi misturado e aplicado nas superfícies tratadas de G10 e cerâmica, que foram unidas e colocadas sob carga de 750 g por 1 min. O excesso de cimento foi removido e os corpos-de-prova foram fotoativados por 40 s.

2.2 Ensaio de fadiga

Uma carga de 120 N foi aplicada à superfície cerâmica com um pistão esférico de metal de aço inoxidável (diâmetro da ponta ativa: 6 mm) usando uma máquina de fadiga pneumática (BioPDI, Biocycle, São Carlos, Brasil) com frequência de 3 Hz em água destilada a 37ºC. O teste foi interrompido após 10^4, 10^5, 5×10^5, 10^6 e 1.5 $x10^6$ ciclos e a presença ou ausência de falha foi observada utilizando transiluminação [19].

Falhas do tipo trinca (crack), lascamento e fratura catastrófica foram consideradas e reportadas em todas as etapas do teste de fadiga. As trincas foram reportadas como radiais, originadas da interface cerâmica-cimento (entalhe) ou cônicas (Hertzian cracks) originadas na área de contato com o pistão.

2.3 Análise topográfica e composicional

Amostras representativas de cada tratamento de superfície foram montadas em suportes de alumínio e cobertas em carbono para avaliação topográfica em microscópio eletrônico de varredura (MEV- Vega LM3, Tescan, Republica Tcheca) e analise composicional por espectroscopia de raios X por energia dispersiva (EDS) (Xmax, Oxford, Republica Tcheca).

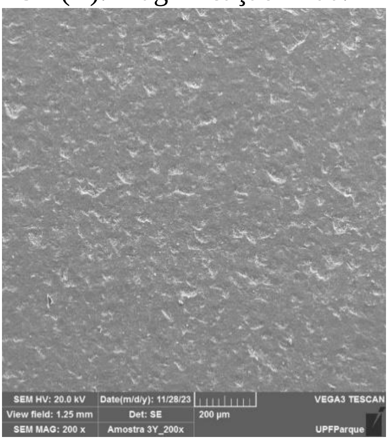
2.4 Análise estatística

Os dados de fadiga foram analisados usando Kaplan – Meier (log rank) e teste de sobrevivência Holm–Sidak (α= 0.05). Os modos de falha foram reportados quantitativa e qualitativamente. A relação entre os modos de falha foi analisada pelo Chi-square test (α = 0.05).

3. Resultados

As figuras 1 - 5 apresentam a topografia dos tratamentos de superfície, além da análise composicional das superfícies cerâmicas tratadas (Figuras 2-5).

Figure II.1: Imagens representativas da superfície dos grupos controle (usinagem CAD CAM) das zircônias 3Y-TZP (A) e 5Y-PSZ (B). Magnificação x200.



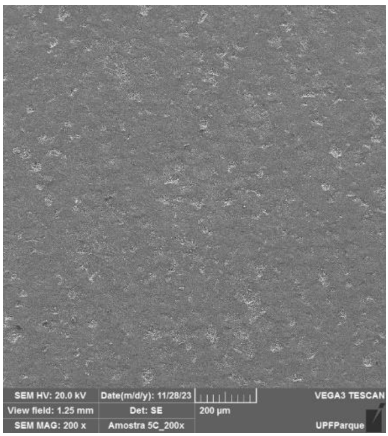


Figure II.2: Imagem representativa de SEM (x1000) da superfície da cerâmica 3Y-TZP após silicatização. Análise de EDS da superfície tratada confirmou a presença dos elementos químicos componentes da zircônia avaliada.

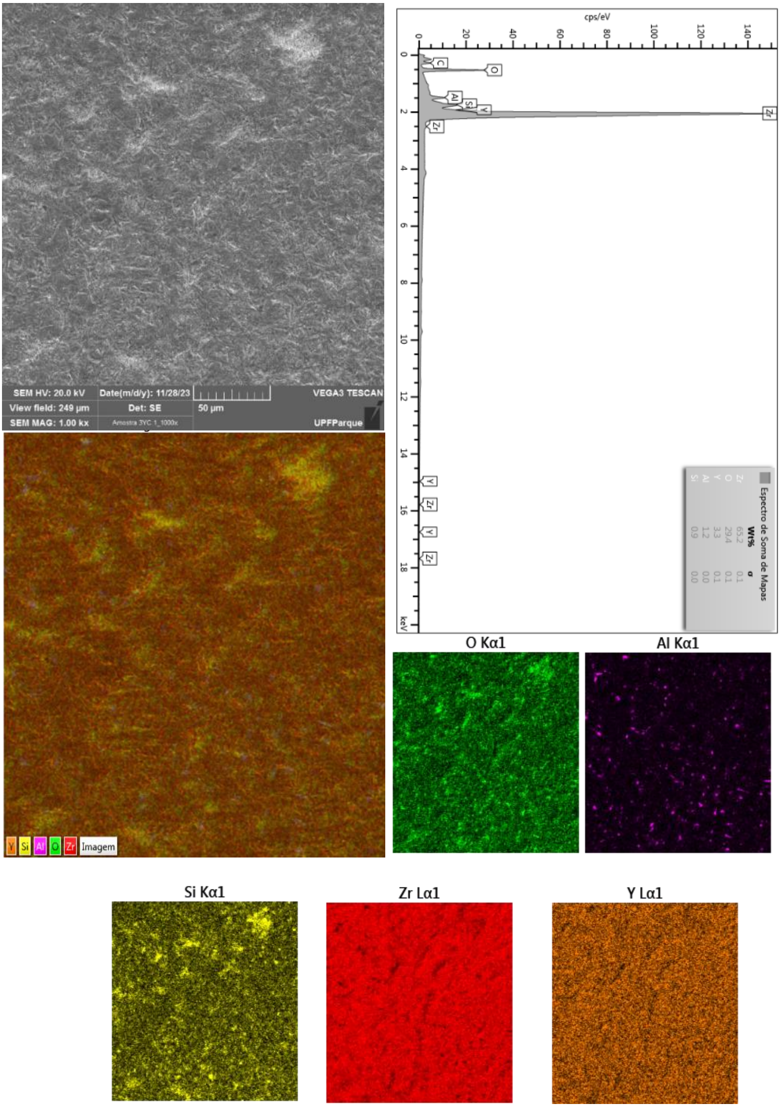


Figure II.3: Imagem representativas de SEM (x1000) da superfície da cerâmica 3Y TZP após jateamento com partículas de óxido de alumínio. Análise de EDS da superfície tratada confirmou a presença dos elementos químicos componentes da zircônia avaliada.

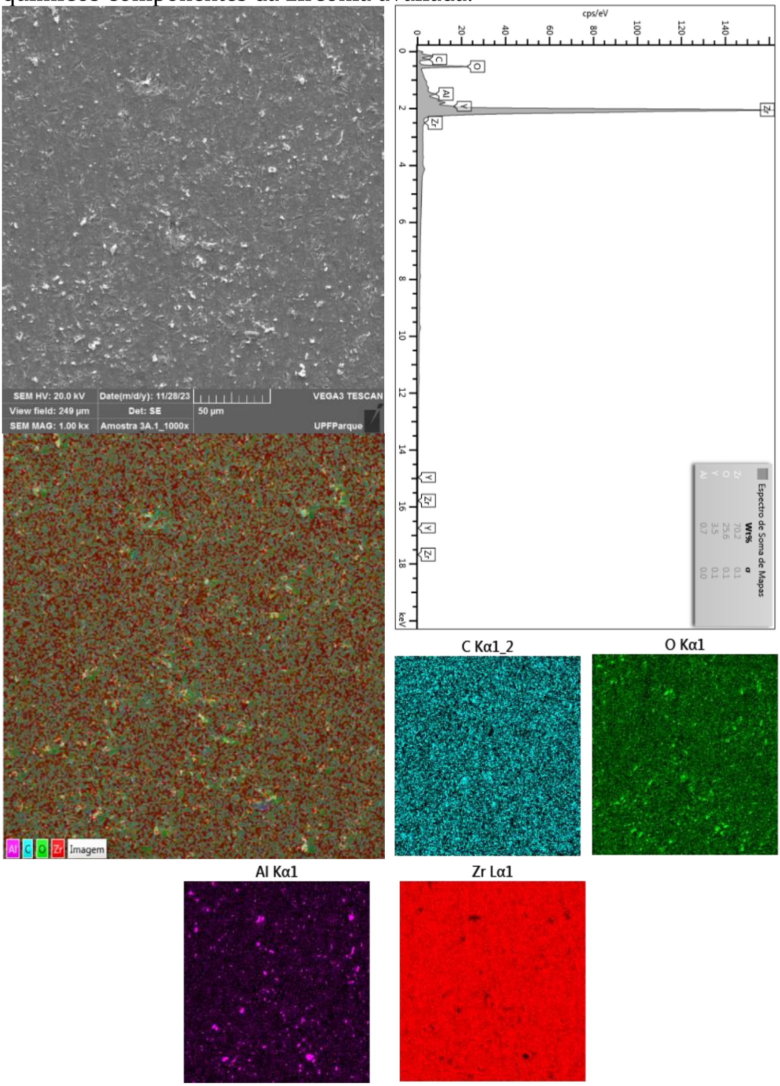


Figure II.4: Imagem representativas de SEM (x1000) da superfície da cerâmica 5Y PSZ após silicatização. Análise de EDS da superfície tratada confirmou a presença dos elementos químicos componentes da zircônia avaliada.

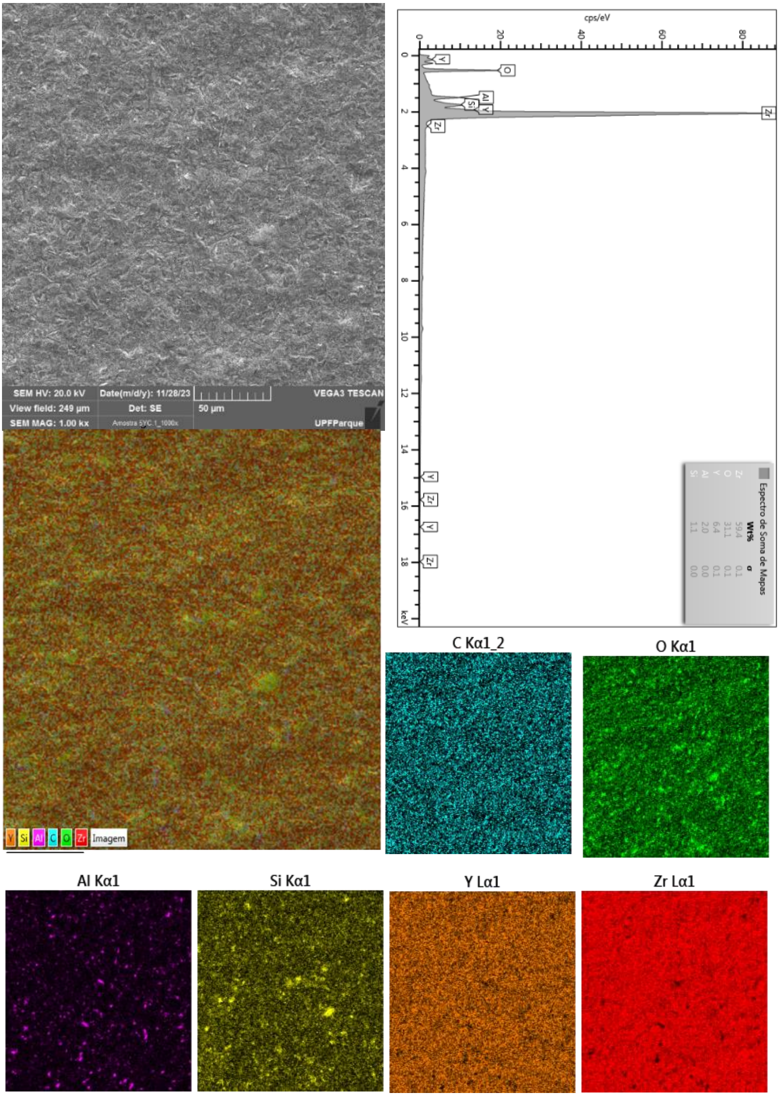
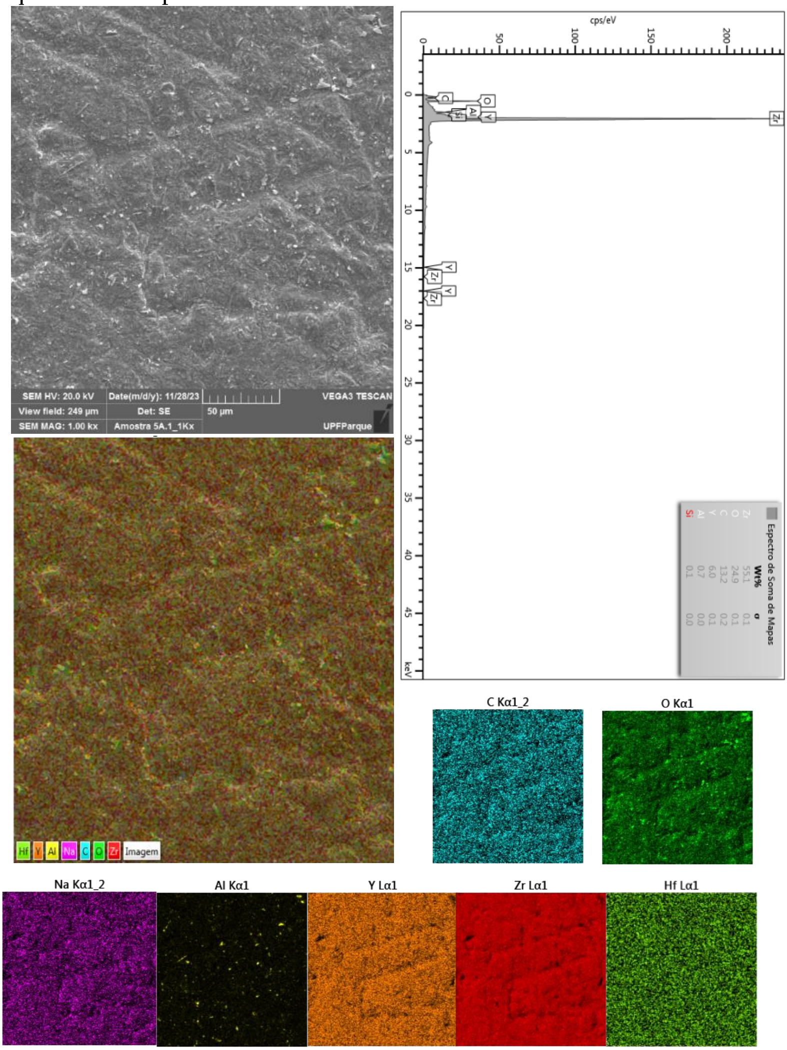


Figure II.5: Imagem representativas de SEM (x1000) da superfície da cerâmica 5Y PSZ após jateamento com partículas de óxido de alumínio. Análise de EDS da superfície tratada confirmou a presença dos elementos químicos componentes da zircônia avaliada.



O número e o tipo de falhas, e a taxa de sobrevivência em cada grupo experimental são apresentados na Tabela 1. A maioria dos CPs do grupo 5Y-Cojet (90%) não sobreviveu ao ensaio de fadiga (1,5 x 10^6 ciclos), enquanto que a maioria (60-75%) dos CPs dos demais grupos sobreviveu ao ensaio de fadiga.

O Chi-square test foi utilizado para determinar se a proporção de modos de falha era diferente para diferentes grupos. Não houve diferença significativa para os modos de falha (p=0,30) entre todos os grupos. Mais precisamente, não houve diferença quanto ao modo de falha entre as ceramicas 3Y e 5Y usando Cojet (p=0,39), particulas Al2O3 (p=0,15) ou sem tratamento (p=0,25). Além disso, os modos de falha não foram diferentes entre os tres grupos de 5Y (p=0,64) e entre os tres grupos de 3Y (p=0,25). A trinca foi o modo de falha mais predominante, com algumas falhas catastróficas e nenhum lascamento.

Tabela II.1: Falhas e sobrevivência para cada grupo experimental.

Grupos experimentais	Falhas				Taxa de Sobrevivência
	trinca	lascamento	catastrófica	total	
5Y-Cojet	16	0	2	18	10%
5Y-Al_2O_3	7	0	0	7	65%
3Y-Cojet	6	0	0	6	70%
3Y- Al_2O_3	6	0	2	8	60%
5Y-None	7	0	1	8	60%
3Y-None	3	0	2	5	75%

As Figuras 6 e 7 apresentam as curvas de sobrevivência de Kaplan-Meier para os grupos experimentais. Entre os grupos 5Y, os três tratamentos de superfície apresentam curvas de sobrevivência significativamente diferentes ($p < 0,001$). No entanto, entre os grupos 3Y, não há diferença significativa ($p = 0,54$). Considerando o tipo de tratamento de superfície, apenas o jateamento com Cojet mostrou uma diferença significativa ($p < 0,001$) entre as cerâmicas 3Y e 5Y, enquanto que o jateamento com Al2O3 (p=0,70) ou sem tratamento (p=0,38) nao mostraram diferenças entre as cerâmicas 3Y e 5Y.

Figura II.6: Curvas de sobrevivência versus número de ciclos para os grupos 5Y.

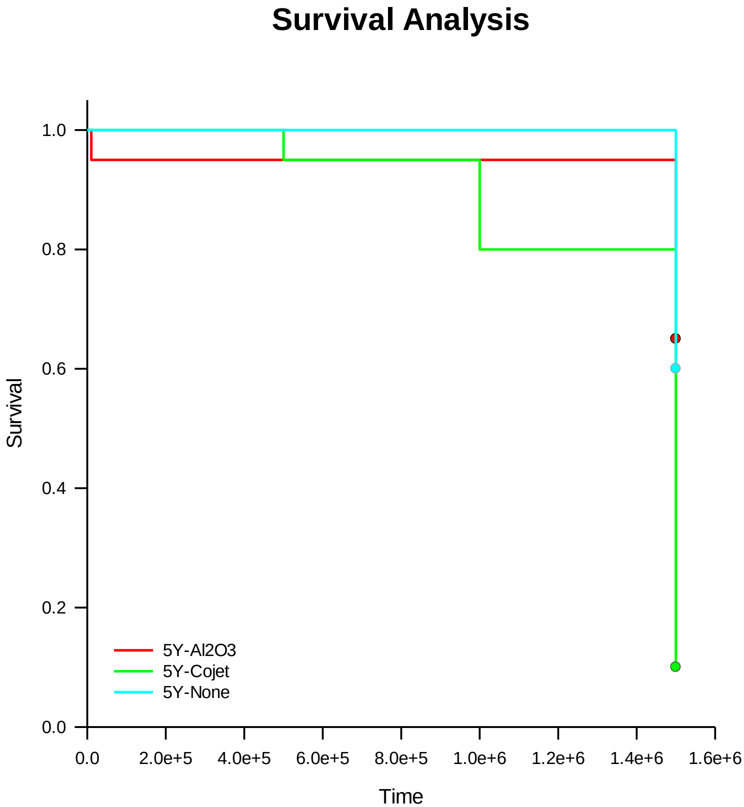
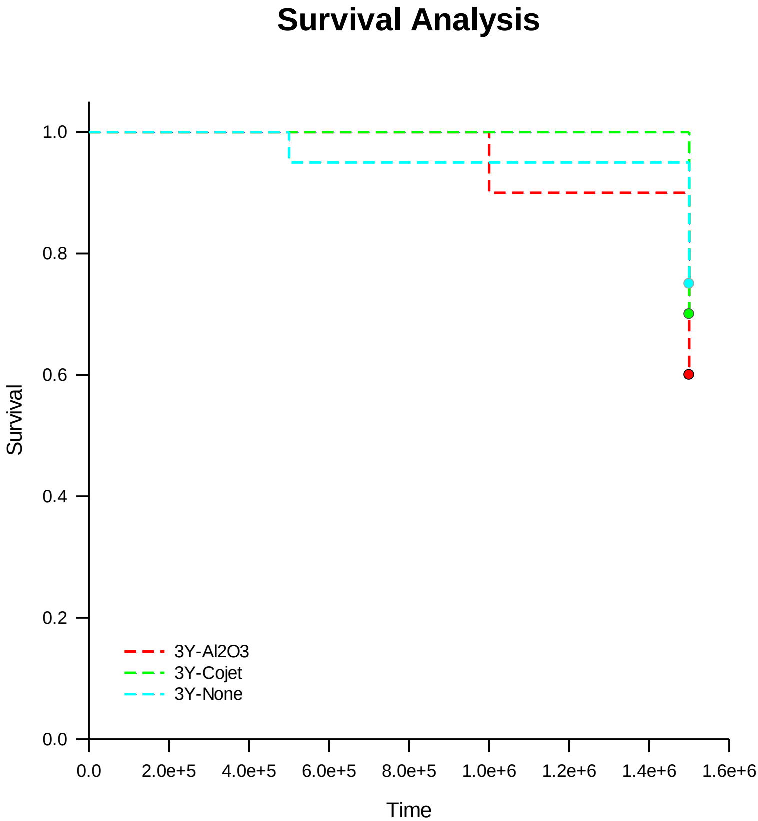


Figura II.7: Curvas de sobrevivência versus número de ciclos para os grupos 3Y.



4. Discussão

Existe uma ampla discussão sobre qual tipo de zircônia e tratamento de superfície proporcionam a melhor restauração, de acordo com o caso clinico, ao longo do tempo. Este estudo teve como objetivo avaliar o comportamento à fadiga de cerâmicas à base de zircônia 3Y-TZP (Vita YZ HT) e 5Y-PSZ (Vita YZ XT) aderidas ao cimento resinoso após diferentes tratamentos de superfície (jateamento com partículas de óxido de alumínio, silicatização e o controle da usinagem por CAM). As hipóteses testadas foram que (1) os tratamentos de superfície resultam em taxa de sobrevivência à fadiga semelhante para ambas as zircônias avaliadas (3Y-TZP e 5Y-PSZ), e que (2) a 3Y-TZP tem um desempenho superior à fadiga em comparação com a 5Y-PSZ. Os resultados mostraram que ambas as hipóteses foram parcialmente aceitas pois a 5Y-PSZ silicatizada apresentou uma taxa de sobrevivência significativamente inferior aos demais grupos experimentais. Contudo, os tratamentos de superfície utilizados não alteraram a taxa de sobrevivência da 3Y-TZP. Além disso, não foi possível determinar que o 3Y-TZP apresenta um desempenho superior em termos de resistência à fadiga em relação ao 5Y-PSZ, já que ambos apresentaram resultados estatisticamente semelhantes, exceto quando submetidos ao tratamento de silicatização.

O maior teor de ítria melhora a translucidez e a aparência dos materiais de zircônia dentária, porém essas alterações na composição do material também afetam as propriedades mecânicas, com uma maior concentração de ítria sendo associada a diminuição na resistência e na tolerância a danos de cerâmicas a base de zircônia estabilizadas por ítria (Y-Z) [20-22]. Estudos reportaram redução de resistência a fratura em coroas 5Y-Z comparadas a coroas de 3Y-Z [20, 22]. Em outro estudo a 5Y-TZP mostrou valores de fratura mais baixos do que 4Y- e 3Y-TZP, mas com desempenhos comparáveis de ciclagem térmica e carregamento mecânico [21]. No entanto, no presente estudo, a 5Y-PSZ mostrou comportamento de fadiga inferior a 3Y-TZP apenas quando usado silicatização.

O preparo da superfície com jateamento é realizado com objetivo de melhorar a adesão e o desempenho das restaurações de zircônia cimentadas. No entanto, neste estudo, não foram observadas diferenças significativas entre os grupos controle (sem tratamento de superfície) e os grupos com tratamento de jateamento com partículas de Al2O3 para estruturas de zircônia 3Y-TZP e 5Y-PSZ. Resultados semelhantes foram observados em outro estudo, onde a abrasão por partículas de alumina não afetou significativamente a carga de fratura das coroas de zircônia (5Y-Z) [22], e outro estudo indicou que esse tratamento de superfície aumentou a resistência à flexão biaxial estática e à fadiga de estruturas de zircônia (4Y-PSZ) [23]. Outro estudo mostrou que os tratamentos de superfície (jateamento com partículas de óxido de alumínio e silicatização) melhoraram a resistência de união à zircônia convencional e de alta translucidez [24]. Não obstante, outro estudo mostrou que jateamento com partículas de alumina reduziu a resistencia a fadiga da zircônia (5Y-PSZ), enquanto que a silicatização não prejudicouo o comportamento à fadiga do 5Y-PSZ [25]. Outro estudo mostrou que a silicatização (CoJet e SilJet) da superfície da zircônia (IPS e.max ZirCAD) resultou na maior durabilidade de união de um cimento resinoso antes e após o envelhecimento mecânico, quando comparado com grupo controle e grupo jateado com partículas de alumina [26]. Outro estudo [27] mostrou que a silicatização da zircônia (IPS e.max ® ZirCAD) apresentou os menores valores de resistência de união quando comparado com os demais grupos (glaze spray, glaze pó/líquido e primer de zircônia). Esses estudos justificam as hipoteses apresentadas para o presente estudo.

Os resultados inferiores do grupo 5Y-Cojet observados no presente estudo, se devem, provavelmente, à questionável estabilidade da ligação do jateamento com sílica ao longo do tempo, o que foi observado pela maioria (14 das 18 falhas) dos CPs de 5Y-Cojet falharam na última fase de fadiga. Um estudo mostrou que a silicatização não resultou em uma camada de sílica uniforme e estável para YPSZ [28].

Tem sido reportado uma variação significativa nos protocolos de jateamento, incluindo diferenças no tamanho das partículas e nos parâmetros operacionais, como pressão, distância e tempo de aplicação. Essa diversidade dificulta a comparação e associação com outros estudos.

Sugere-se que os métodos de jateamento por partículas não são o suficiente para promover a melhor adesão do cimento resinoso à cerâmicas a base de zircônia [14, 29], a adesão química adicional utilizando primers contendo monômeros funcionais, como o 10-metacriloxidecil di-hidrogenofosfato (10-MDP) é desejável [1, 14, 30, 31]. Primers à base de silano também têm sido utilizados para melhorar a união entre adesivos e superfícies silicatizadas [1]. Esse raciocínio foi utilizado na seleção dos materiais no presente estudo.

Com o intúito de simular ambiente clinico, as estruturas ceramicas foram cimentadas adesivamente a estruturas análogas a dentina (G10- em resina epóxi reforçada com fibra de vidro) e submetidas ao ensaio de fadiga em água a 37ºC [19]. Resutados de testes de adesão do cimento resinoso ao G10 em ambientes úmidos e secos, comparando com a adesão à dentina, mostraram uma boa compatibilidade, tanto em termos de adesão quanto de comportamento elástico [32].

As imagens em MEV representativas da superfície das cerâmicas após cada tratamento de superfície, mostrou que ambos os protocolos de jateamento promoveram uma alteração superficial mais proeminente em comparação com o grupo controle (usinado por CAM). Análise de EDS da superfície tratada confirmou a presença dos elementos químicos componentes da zircônia e do preparo da superfície. Na figura 5, o mapeamento químico por EDS mostrou o elemento Hf (háfnio), isso pode ser explicado pela grande similaridade química com zircônio, o que se torna difícil a separação durante a analise [33].

Este estudo apresenta algumas limitações, como o uso de um pistão de aplicação de carga arredondado de aço inoxidável, ao invés de utilizar pistões de esmalte ou outros materiais restauradores antagonistas [34, 35]. A opção por um pistão metálico de ponta esférica foi devido à necessidade de suportar a elevada carga utilizada no ensaio, e pela similaridade de comportamento, comparado a outros pistões, em testes de fadiga [35]. A ausência de deslizamento durante a aplicação da carga, que ocorre clinicamente, também pode ser considerado uma limitação do estudo [36]. A aplicação de cargas multiaxiais pode influenciar a resposta dos materiais aos estímulos, alterando os resultados e padrão de falha. Apesar disso, a dificuldade de fornecer uma simulação completa do ambiente oral é uma limitação nos estudos in-vitro, sugerindo-se estudos clínicos para confirmar os resultados in vitro.

5. Conclusões

Considerando as limitações do presente estudo, podemos concluir que:

- A silicatização prejudicou o desempenho a fadiga da zircônia 5Y-PSZ quando comparado ao controle e ao jateamento com partículas de alumina. Contudo, esse efeito negativo da silicatização não foi observado na 3Y-TZP.

- Exceto quando tratada com silicatização, a resistência a fadiga da 5Y-PSZ foi semelhante a 3Y-TZP.

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CONSIDERAÇÕES FINAIS

Com base nos resultados dessa tese podemos concluir que:

– Existe grande variabilidade nas metodologias de testes de fadiga utilizados para avaliar estruturas de zircônia translucida. Considerando as falhas clínicas em estruturas de zircônia translúcida reportadas, apenas três estudos in vitro apresentaram falhas semelhantes. Como existem poucos estudos clínicos, é difícil associar as falhas ocorridas nos estudos in vitro com falhas clinicas, mostrando a necessidade de mais estudos que reportem falhas clínicas com zircônias translúcidas.

– O tratamento de superfície com silicatização prejudicou o desempenho a fadiga da zircônia 5Y-PSZ quando comparado ao controle e ao jateamento com partículas de alumina. Contudo, esse efeito negativo da silicatização não foi observado na 3Y-TZP. Exceto quando tratada com silicatização, a resistência a fadiga da 5Y-PSZ foi semelhante a 3Y-TZP.

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