



DEBORAH PACHECO LAMEIRA

**FRACTURE STRENGTH OF MONOLITHIC AND BI-LAYER
ZIRCONIA-BASED CROWNS.**

**RESISTÊNCIA À FRATURA DE COROAS MONOLÍTICAS E
BICAMADA A BASE DE ZIRCÔNIA.**

Piracicaba
2014



**UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA**

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Thesis presented to the Piracicaba School of Dentistry of the University of Campinas in partial fulfillment of the requirements for the PhD degree in Dental Clinic, concentration area of Dental Prosthesis.

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Orientador: Prof. Dr. Wilkens Aurélio Buarque e Silva

Este exemplar corresponde à versão final da tese defendida pelo aluna Deborah Pacheco Lameira, orientada pelo Prof. Dr. Wilkens Aurélio Buarque e Silva.

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Alexandre Brait Landulpho

Fernanda Paixão

Mario Alexandre Coelho Sinhoreti

Filipe Polesi Branco

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Prof. Dr. WILKENS AURELIO BUARQUE E SILVA

Prof. Dr. ALEXANDRE BRAIT LANDULPHO

Profa. Dra. FERNANDA PAIXÃO

Prof. Dr. MARIO ALEXANDRE COELHO SINHORETI

Prof. Dr. FILIPE POLESÍ BRANCO

ABSTRACT

The purpose of this study was to evaluate the fracture strength and failure mode of zirconia crowns in monolithic or bi-layer configuration after artificial aging. Thirty-two bovine incisors received crown preparation and were scanned for the manufacturing of Y-TZP crowns using CAD/CAM technique. Crowns were fabricated according to the following groups (n=10/group): PM polished monolithic zirconia crowns of 1.5mm uniform thickness; GM glazed monolithic zirconia crowns of 1.5mm uniform thickness; BL bi-layer crowns with 0.8mm zirconia core and 0.7mm of veneered porcelain. All crowns were cemented on each respective tooth with resin cement (RelyX Unicem-2, 3M-ESPE). After the cementation, specimens were submitted to artificial aging in a chewing simulator (2.5 million cycles/ 3Hz frequency/8kg) in artificial saliva at 37°C, then the samples were submitted to fracture strength test (Instron) at a crosshead speed of 0.5mm/min. Two remaining crowns referring to PM and GM were submitted to Electron probe microanalysis (EPMA), to measure the yttrium levels after aging. One-way ANOVA and Tukey's test ($p=0.05$) were employed for analysis of the results. The fracture mode was evaluated with Scanning Electron Microscope (SEM). Monolithic zirconia crowns presented similar fracture strength results (PM= $3476.2N \pm 791.7$; GM= $3561.5N \pm 991.6$), which were higher ($p=0.02$) than the results for the bi-layer crowns (BL= $2060.4N \pm 810.6$). There was no significant difference in the yttrium content among worn occlusal surfaces (PM, GM) and undamaged surface in PM. Thus, the fracture strength of monolithic zirconia crowns was higher than bi-layer configuration.

Key Words: Ceramics, Crowns, Fatigue.

RESUMO

O objetivo deste estudo foi avaliar a resistência à fratura de coroas monolíticas e bicamada a base de zircônia, após envelhecimento artificial. Para este estudo, foram utilizados 32 incisivos bovinos onde foram feitos preparos de coroa total. Estes foram escaneados para a confecção de coroas a base de a zircônia tetragonal parcialmente estabilizada por ítrio (*yttrium partially stabilized tetragonal zirconia polycrystalline* Y-TZP), por um sistema CAD/CAM (computer-aided design and computer-aided manufacturing). As coroas foram fabricadas de acordo com os grupos experimentais (n=10/grupo): Coroa monolítica de zircônia polida PM (1.5mm de espessura); Coroa monolítica de zircônia com aplicação de glaze GM (1.5mm de espessura); Coroa bicamada com *coping* de zircônia BL (0.8mm de espessura) com cerâmica de cobertura (0.7mm de espessura). As coroas foram cimentadas nos respectivos preparos com cimento resinoso (RelyX Unicem-2, 3M-ESPE). Após a cimentação, as amostras foram submetidas ao envelhecimento artificial em uma simuladora de mastigação (2,5 milhões de ciclos/3Hz de frequência/8kg) em saliva artificial a 37°C, em seguida foi efetuado o teste de resistência à fratura em máquina Instron, com velocidade de 0,5 mm/min. Duas coroas remanescentes referente a PM e GM foram submetidas a análise estrutural *Electron probe microanalysis* (EPMA), para medir o nível de ítrio após o envelhecimento. Os resultados foram submetidos a Análise de Variância e teste de Tukey ($p=0.05$). O modo de fratura foi avaliado por meio de Microscopia Eletrônica de Varredura (MEV). As coroas de zircônia monolíticas apresentaram resultados semelhantes de resistência à fratura (PM = $3476,2N \pm 791,7$; GM = $3561,5N \pm 991,6$), que foram maiores ($p=0,02$) do que os resultados para as coroas bicamada (BL = $2060,4N \pm 810,6$). Não houve diferença significativa na quantidade de ítrio entre as superfícies oclusais desgastadas (PM, GM), e na superfície não desgastada de PM. Destes modo, a resistência à fratura de coroas monolíticas foi maior do que de coroas na configuração bicamada.

Palavras-chave: Cerâmicas, Coroas Dentárias, Fadiga.

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INTRODUÇÃO

O aumento da exigência estética em odontologia proporcionou o desenvolvimento de materiais cerâmicos tais como a zircônia, que apresenta maior resistência às forças mastigatórias aliada a elevadas propriedades estéticas.

Diferente das cerâmicas convencionais, que possuem vidro em sua composição, a zircônia tetragonal estabilizada por ítrio (yttrium partially stabilized tetragonal zirconia polycrystalline Y-TZP) é um material cerâmico com propriedades mecânicas superiores as demais cerâmicas odontológicas, dentre elas a resistência à tensão flexural que oscila entre 900 e 1400 Mpa (Denry & Kelly, 2008).

A zircônia apresenta três formas cristalográficas: a monoclinica em temperatura ambiente até 1170°C, a tetragonal de 1170°C até 2370°C, e a cúbica em temperaturas acima de 2370°C. Durante o resfriamento ocorre uma expansão de volume de 3% a 5% dos cristais associada à transformação da fase tetragonal para a monoclinica. Entretanto vários óxidos estabilizadores como, cálcio (CaO), magnésio (MgO), ítrio (Y₂O₃) ou céria (CeO₂), podem ser adicionados à zircônia para estabilizar essa transformação de fase em temperatura ambiente (Andreiulo *et al.*, 2011; Flinn *et al.*, 2012).

A concentração do agente estabilizador ou dopante tem um papel determinante no desempenho do material sob fadiga. Quando adicionada uma grande quantidade (8-12% em peso) de dopante, uma fase cúbica totalmente estabilizada pode ser produzida, o que inviabiliza a transformação da fase tetragonal para a monoclinica, resultando num pior desempenho do material. No entanto, ao adicionar quantidades menores (3-5%), é produzida zircônia tetragonal parcialmente estabilizada que corresponde a um estado metaestável, ou seja, diferente da forma que se encontraria em equilíbrio termodinâmico (Andreiulo; *et al.*, 2011).

A zircônia Y-TZP é estável em temperatura ambiente, porém, sob tensão, esta fase pode sofrer alteração para a fase monoclinica, com um aumento subsequente de cerca de 4 a 5% em volume. Este mecanismo, conhecido como “tenacificação por transformação” é o principal responsável pelas superiores propriedades mecânicas da zircônia (Andreiuolo; *et al.*, 2011; Denry & Kelly, 2008; Fabbri *et al.*, 2013).

Entretanto, devido sua natureza metaestável, os materiais a base de zircônia são suscetíveis a uma transformação de fase desfavorável em baixas temperaturas, esse fenômeno é conhecido como degradação em baixas temperaturas (low temperature degradation LTD). O envelhecimento ocorre por meio de uma lenta transformação de fase tetragonal para monoclinica nos grãos mais superficiais em contato com a água, ou fluido corporal. Isto gera rugosidade superficial e formação de microtrincas, abrindo possibilidade para água penetrar nas camadas subsuperficiais e gerar trincas maiores, causando consequente perda de resistência mecânica (Chevalier, 2006; Lughì & Sergo, 2010).

Esse fenômeno pode variar entre os materiais devido suas características microestruturas como, tamanho e orientação dos grãos, concentração do estabilizante e concentração de tensões residuais inerentes ao processo de confecção (Chevalier *et al.*, 2011; Flinn *et al.*, 2012; Fabbri *et al.*, 2013).

O impacto da LTD foi enfatizado desde 2001 (Haraguchi *et al.*) quando centenas próteses femorais falharam em um curto período de tempo. A falha foi associada ao envelhecimento acelerado e causou preocupação na comunidade biomédica, mesmo tendo ocorrido em um número limitado e em uma marca específica do material. Porém, a fratura dos implantes femorais estava associada à presença de porosidade no material o que permitiu a penetração de água através da sua estrutura e consequente degradação, o que promoveu a tensão

necessária à fratura. As fraturas foram relatadas apenas em implantes de Y-TZP que continham algum tipo de porosidade (Flinn *et al.*, 2012; Sanon *et al.*, 2013).

A falha das próteses femorais foi vista com preocupação na odontologia pelo uso de restaurações totalmente em zircônia sem cerâmica de cobertura. Descritas na literatura como monolíticas, as restaurações totalmente em zircônia tiveram sua inserção na clínica odontológica devido ao grande número de falhas na cerâmica de cobertura.

Alguns estudos prévios demonstraram que a taxa de sucesso clínico de restaurações a base de zircônia poderia variar entre 79 e 100% durante cinco anos (Ortorp *et al.*, 2012; Sorrentino *et al.*, 2012; Vigolo & Mutinelli, 2012), e que o tipo de falha mais frequente foi a fratura ou lascamento da cerâmica de cobertura.

Beuer *et al.* em 2012 demonstraram em seu estudo *in vitro* que coroas totalmente em zircônia apresentam maior capacidade de suportar cargas do que coroas formadas por *coping* de zircônia e cerâmica de cobertura, confeccionadas pela técnica de estratificação com pó e líquido.

O alto índice de falhas na cerâmica de cobertura em coroas a base de zircônia, demonstrou que a resistência dos sistemas cerâmicos livres de metal não depende somente do material de sua infraestrutura, mas também do material de cobertura. Onde um sistema bicamada pode apresentar uma infraestrutura de Y-TZP com alta resistência flexural (900-1400 MPa) e uma cobertura frágil de porcelana feldspática (30 a 97 MPa de resistência flexural) (Shijo *et al.*, 2009).

Alguns autores relataram que coroas a base de zircônia na configuração bicamada apresentam algumas desvantagens como: processo de confecção em várias etapas; maior suscetibilidade ao lascamento; baixa adesão da cerâmica de cobertura ao *coping*; tensão residual que pode ser desenvolvida durante o processo de estratificação (Stawarczyk *et al.*, 2011).

Uma opção para a resolução deste tipo de problema seria a confecção de coroas monolíticas de zircônia, podendo aumentar a estabilidade biomecânica deste material e expandir sua indicação clínica. Porém, seu comportamento e envelhecimento no meio oral ainda não foi completamente esclarecido (Beuer *et al.*, 2012).

Deste modo com o objetivo de contribuir com o esclarecimento destas questões, esta pesquisa *in vitro* visou comparar a resistência a fratura e o modo de fratura de coroas monolíticas a base de zircônia Y-TZP e coroas bicamada (coping de zircônia Y-TZP e cerâmica de cobertura). E avaliar a estabilidade química de coroas monolíticas a base de zircônia após o envelhecimento artificial.

CAPITULO 1

Fracture strength of monolithic and bi-layer zirconia-based crowns.

Deborah Pacheco Lameira^{1,2} , Frederico Andrade e Silva¹, Wilkens Aurelio Buarque e Silva¹, Grace M. De Souza² .

¹Prosthodontics and Periodontology Department, Piracicaba Dental School - UNICAMP, Piracicaba, SP, Brazil.

² Department of Clinical Sciences, Faculty of Dentistry, University of Toronto, Toronto, ON, Canada.

Corresponding author:

Grace M De Souza

Assistant Professor/ Clinical Sciences Department /Faculty of Dentistry/University of Toronto

124 Edward Street Room 352E. Toronto, ON, Canada. M5G 1G6

FAX (416) 979-4936

Grace.DeSouza@dentistry.utoronto.ca

Statement of the problem: The application of monolithic zirconia instead of bi-layer veneered restorations is controversial due to the aging characteristics of zirconia. **Purpose:** To evaluate fracture strength of yttria-tetragonal zirconia polycrystalline (Y-TZP) crowns in monolithic and bi-layer configuration after artificial aging. **Methods:** Thirty-two bovine incisors received crown preparation and were scanned for the manufacturing of Y-TZP crowns using CAD/CAM (LAVA 3M ESPE) technique. Crowns were fabricated according to the following groups (n=10): Polished monolithic zirconia crowns (PM) of 1.5mm uniform thickness; Glazed monolithic zirconia crowns (GM) of 1.5mm uniform thickness; bi-layer crown (BL) 0.8mm zirconia coping with 0.7mm porcelain veneer. After all crowns were cemented on each respective tooth with resin cement (RelyX Unicem 2, 3M ESPE), specimens were submitted to artificial aging in a chewing simulator (2.5 million cycles/3Hz frequency/80N) in artificial saliva at 37°C. Aged samples were submitted to fracture strength test (Instron) at a crosshead speed of 0.5mm/min and the load at failure was recorded. Two remaining crowns referring to G1 and G2 were submitted to a chemical composition analysis to measure the yttrium level after aging. One-way ANOVA and the Tukey test ($p=.05$) were employed for analysis of the results. The fracture mode was evaluated with Scanning Electron Microscope. **Results:** Monolithic zirconia crowns presented similar fracture strength (PM= $3476.2N \pm 791.7$; GM= $3561.5N \pm 991.6$), which were higher than bi-layer crowns (BL= $2060.4N \pm 810.6$). There was no difference in the yttrium content among the three surfaces evaluated in the monolithic crowns. **Conclusions:** Monolithic zirconia crowns present higher fracture strength than bi-layer veneered zirconia after 2.5 cycles in artificial aging. Surface finishing does not affect the fracture strength of monolithic zirconia crowns.

Clinical implications: The results of this study suggest that monolithic zirconia crowns, irrespective of the surface finishing, can support masticatory loads better than bi-layer zirconia based-crowns.

Keywords: High crystalline content zirconia, Fracture, Fatigue, Stability

Introduction

The increase of esthetics' demand has led to the development of metal-free restorations without metallic components.¹ Dental ceramics present numerous favorable characteristics including biocompatibility and excellent potential to simulate the optical characteristics of natural teeth.^{2,3} However, clinical trials recording survival rates for all-ceramic posterior crowns and fixed dental prostheses (FDPs) indicate the vulnerability of those systems to various failure modes.^{4,5} Therefore, ceramic materials with superior mechanical properties were developed in an attempt to improve the fracture toughness of all-ceramic prostheses and, consequently, to enhance the longevity of all-ceramic restorations. Yttrium partially stabilized tetragonal zirconia (Y-TZP) is a ceramic material with mechanical properties superior to the those of other dental ceramics, presenting flexural strength of approximately 900 to 1400 MPa.⁶ Therefore, Y-TZP allows the manufacturing of fixed partial prostheses (FPPs) in areas of high masticatory loads.⁷

However, the strength of an all-ceramic crown depends not only on the core but also on the veneer material, whereby a bi-layer system with a strong and tough Y-TZP core veneered with esthetic but brittle porcelain tends to fail prematurely. Moreover, such bilayer systems have several major drawbacks including the multistep fabrication process, low toughness of the veneer material, and weak bonding between veneer layer and coping.⁸ Therefore, for porcelain-veneered zirconia prostheses, bulk fracture of the zirconia framework appears to be quite uncommon, and the most commonly reported complication is chipping or cracking limited to the porcelain veneer.⁹⁻¹¹

The way to circumvent all these drawbacks is to replace the veneer/core bilayer with a monolithic restorative ceramic.⁸ The fracture resistance of monolithic lithium disilicate while submerged in a wet environment appears promising and prompts second-phase testing to evaluate the potential of various thicknesses

appropriate for posterior single-tooth applications.¹² Fabricating mono-block restorations from pure zirconia could increase the mechanical stability and expand the range of indications of those prostheses. However, its wear behavior and chemical stability have not yet been fully clarified. Zirconia has a monoclinic crystal structure from room temperature to 1170°C, tetragonal from 1170°C to 2370°C, and cubic at temperatures above 2370°C. Upon cooling, a volume expansion of 3% to 5% is associated with the transition of the tetragonal to monoclinic phase. However, several different oxides such as calcium (CaO), magnesium (MgO), yttrium (Y₂O₃) or ceria (CeO₂) can be added to zirconia to stabilize the tetragonal and stronger phase at room temperatures.^{13,14} The concentration of the stabilizer plays a decisive role in the performance of this material under fatigue and the addition of 3-5% of stabilizer results in partially stabilized tetragonal zirconia.

The tetragonal zirconia is then stable at room temperature, however under stress, it may undergo phase transformation to the monoclinic phase, with a subsequent increase of about 4 to 5% of local volume. This mechanism, known as "transformation toughening", is primarily responsible for the superior mechanical properties of zirconia.^{6,15} However, due to its metastable nature, zirconia-based materials are susceptible to unfavorable phase transformation at room temperature, and this phenomenon is known as "low temperature degradation" (LTD). Aging occurs through an uncontrolled slow transformation of superficial grains from tetragonal-to-monoclinic phase in contact with water. This creates surface roughness and formation of micro-cracks, creating possibilities for water penetration causing further phase transformation and consequent loss of mechanical strength.¹⁶⁻¹⁸

Previous studies have reported that the aging process would cause yttrium loss and decrease the tetragonal phase stability of zirconia-based restorations, leading to tetragonal-to-monoclinic transformation. It was hypothesized that this mechanism occurs as a result of the reaction between water (H₂O) and yttrium (Y₂O₃) to form yttrium hydroxide (Y(OH)₃), which depletes the stabilizing oxide

sufficiently to make the tetragonal phase unstable and, therefore, cause transformation to the monoclinic phase.^{14,19} Apart from the aging controversy, the application of full-contour zirconia restorations is currently discussed as an alternative to bi-layer veneered restorations, based on the fact that clinical failures are observed mainly in the veneer layer.²⁰ In spite of reducing the possibility of early fracture by eliminating the weak phase (veneer layer) from the restorative complex, phase transformation is a reason of concern, since the direct contact with saliva under masticatory loads may aggravate the water penetration and crack propagation.

Therefore, the purpose of this study was to compare the fracture strength and failure mode of two Y-TZP monolithic systems, either polished or glazed, and bi-layer veneered Y-TZP crowns. The content of yttrium of the monolithic crowns after artificial aging was also investigated. The null hypothesis was that the crown design, monolithic or bi-layer, had no effect on fracture strength of zirconia crowns after artificial aging.

Material and Methods

Thirty two healthy bovine incisors were used in this study, and a standardized crown preparation was performed in a lathe machine (MAGNUM-CUT FEL-2680 GZJ, Sao Paulo, Brazil) with the following dimensions: 4.2 mm diameter occlusal base, 6.0 mm diameter cervical base and 7.0 mm axial height (Fig. 1). The taper was established as 8 degrees for all axial walls and the cervical finish line was rounded shoulder. The tooth inner angles were rounded with fine grain diamond burs (KG Sorensen, São Paulo, Brazil).

Specimens were randomly divided in three groups (n=10) according to the crown fabrication technique: PM group: monolithic zirconia polished crowns (1.5 mm thickness); GM group: monolithic zirconia glazed crowns (1.5 mm thickness);

BL group: zirconia copings with hand-layered porcelain veneering (0.8 mm core and 0.7mm porcelain thickness). Two additional crowns, referring to PM and GM groups, were submitted to an Electron Probe Microanalysis (EPMA) to quantify the yttrium content after aging.

For fabrication of the non-anatomical crowns, all preparations were scanned by a non-contact optical 3D-scanning device (Lava Scan system scanner, 3M ESPE, Seefeld, Germany). All zirconia crowns and copings were designed by the same technician with Lava Scan Design System. Then, zirconia blocks (Lava Plus for monolithic crowns, and Lava Frame for by-layer crowns) were milled by using the Lava CNC 500 Milling Machine (3M ESPE, Seefeld, Germany). After the milling procedure, all copings and crowns were removed from the CAM-machine and were sintered in a furnace (Lava Furnace 200) for approximately 11 hours. The fully sintered crowns referring to PM were finished and polished with diamond wheels and bristle brushes (Brasseler Dental instruments). The crowns referring to GM received glaze firing after the sinterization. A silicone impression was taken from one finalized specimen of PM in order to duplicate its 1.5 mm thickness to control the final thickness of the veneered crowns. Copings referring to BL were veneered using the powder build-up technique with Lava Ceram veneer ceramic (3M ESPE, Seefeld, Germany). The thickness of veneered porcelain and the contour of the final crown were verified by measuring the crown at different locations with a digital caliper, and the firing cycle was controlled by an experienced dental technician, to ensure standardized crowns.

The crowns were cleaned for 10 min in an ultrasonic bath (Branson Ultrasonic Cleaner 3510 E-DTH; Branson, Nanbury, USA), and 10 samples of each group were cemented on their respective prepared tooth with a self-adhesive phosphate-based luting resin (RelyX Unicem 2 Automix, 3M ESPE). A static load of 5kg^{10,21} was applied for 7 minutes following the cementation procedure according to the manufacturer's instructions. The crowns for chemical analysis were cemented using temporary cement (RelyX Temp NE, 3M ESPE). After luting,

specimens were stored in distilled water at 37°C for 24 hours, and submitted to an aging procedure: 2,500,000 cycles, 80N, at 37°C under artificial saliva bath²². Loading was applied with a vertical displacement of 0.2mm and horizontal (occlusal) displacement of 0.5mm in a chewing simulator CS-4 (SD Mechatronik, Feldkirchen-Westerham, Germany). As a substitute for human enamel, hydroxyapatite steatite balls (3mm diameter) were used as antagonists and were replaced for each sample.²³

Aged specimens were loaded in a Universal Testing Machine (Instron Model 8501) under deionized water bath at room temperature, with a 5 mm diameter ball indenter (stainless steel) at a crosshead speed of 0.5 mm/min. The maximum fracture load was measured by applying compressive load to the occlusal surface until the crown failed. Catastrophic failure was defined as exhibition of visible cracks, sudden load drops and acoustic events of chipping or fracture.

The crowns were optically examined after fracture testing, and failure modes were divided into: total core fracture, chipping of the veneer, or fracture at core/veneer interface. One representative specimen from each group was mounted on stubs with carbon adhesive tape and colloidal silver paint. Then, specimens were sputter coated with gold-palladium and observed under Scanning Electron Microscopy (SEM)>.

The two remaining samples referring to PM and GM were used for quantification of yttrium content. The yttrium level was measured in 10 points starting from the worn occlusal surface (occlusal dimple) up to the most inner point of the coping of PM and GM, and in a surface away from the occlusal load in PM undamaged. Compositional analyses were performed by using Electron probe microanalysis (EPMA) on an electron microprobe (Camera SX-50/51 DCI 1300 DLL) with 40 degrees take-off angle, and beam energy of 15 keV.

Statistical analysis was carried out using SPSS 19.0 for Windows (SPSS

Inc., Chicago, IL, USA). Mean values and standard deviations (SD) were calculated and analyzed by means of one-way analysis of variance (ANOVA) and Tukey test with 95% confidence levels for both, fracture strength and Yttrium content.

Results

All restorations survived the artificial aging in the chewing simulator. The fracture strength of monolithic zirconia crowns presented similar results (PM= 3492.5N $\pm$ 748.2; GM= 3344.7N $\pm$ 1159.4), which were significantly ($p=.02$) higher than the results for the bi-layer crowns (BL= 2051.8N $\pm$ 764.7) (Table 3).

The failure pattern observed in PM and GM, showed total core fracture (Fig. 2). All the specimens from group BL showed fracture at core/veneer interface without infrastructure failure.

Fractographic analysis of PM and GM indicates that the direction of the crack propagation occurs from the occlusal surface to the center of the restoration, based on failure patterns, hackles and lines that are perpendicular to the crack origin (fig. 3). In BL fractographic analysis shows that the critical flaw is located in the middle of the surface damaged inside the veneer layer (fig. 4).

The Electron probe microanalysis indicates statistically ($p=.935$) similar concentration of yttrium among the surfaces: PM worn occlusal, GM worn occlusal and PM undamaged (Table 4).

Table 1. Two-way ANOVA test results of fracture strength effect.

Source	Sum of Squares	df	Mean Square	F	P
Between Groups	12563505.80	2	6281752.900	7.571	.002
Within Groups	22401284.20	27	829677.193		
Total	34964790.00	29			

Table 2. Two-way ANOVA test results of Yttrium content effect.

Source	Sum of Squares	df	Mean Square	F	P
Between Groups	.001	2	.000	.067	.935
Within Groups	.157	27	.006		
Total	.158	29			

Table 3. Mean of fracture strength (N).

Experimental group	Fracture strength (N)	Std. Deviation
Polished monolithic (PM)	3492.5	748.21
Glazed monolithic (GM)	3344.7	1159.45
Bi-layered veneered (BL)	2051.8*	764.76

* Statistically difference among experimental groups ($p=.02$).

Table 4. Mean of Yttrium levels (wt%) in the monolithic crowns (G1 and G2).

Experimental group	Yttrium content (%)	Std. Deviation
PM worn occlusal	2.0785 ^a *	0.9361
GM worn occlusal	2.0822 ^a	0.6728
PM undamaged	2.0700 ^a	0.6443

* Similar letters indicating statistically similar results among all groups ($p=.935$).

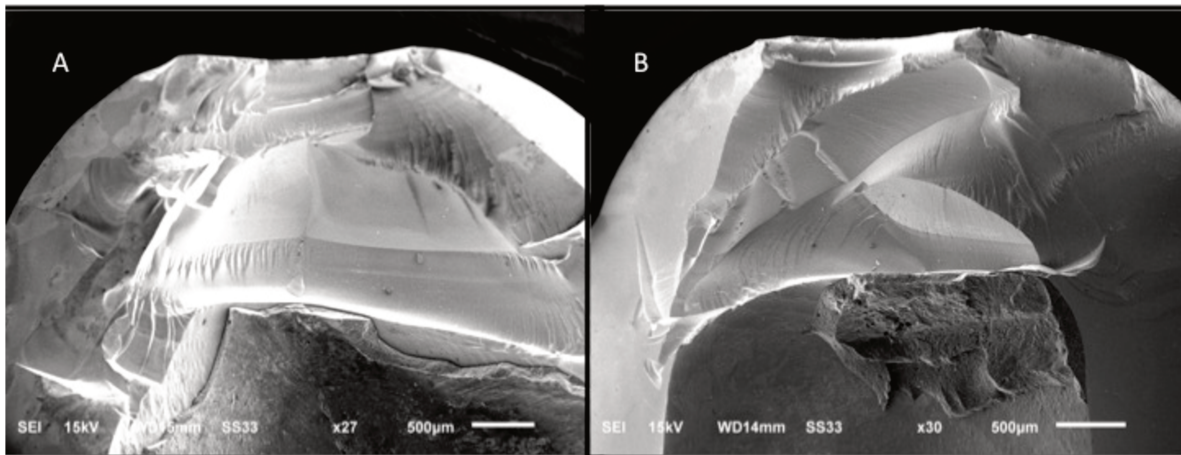


Figure 1. Overview of scanning electron micrographs of PM- polished monolithic crown (A – x27) and GM- glazed monolithic crown (B – x30) fractured specimens.

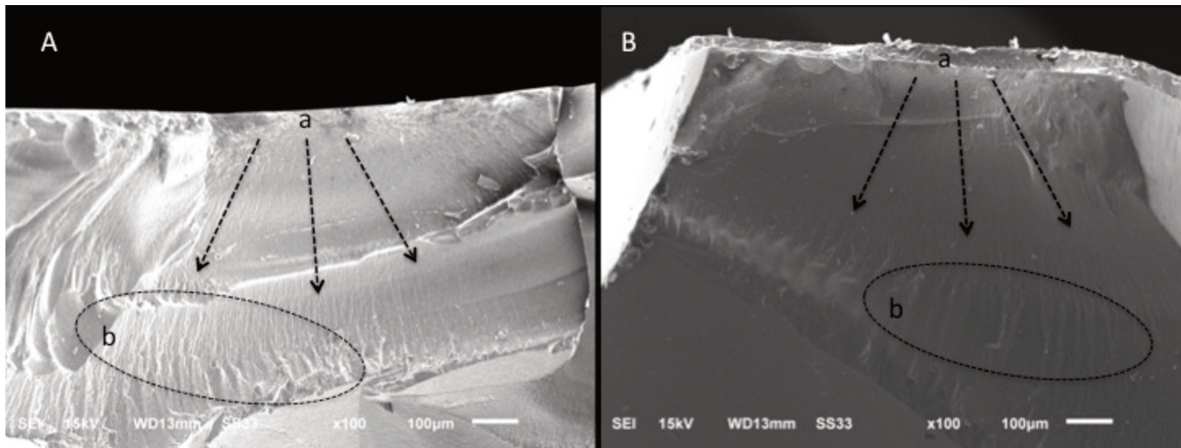


Figure 2. SEM micrographs of PM- polished monolithic (A) and GM- glazed monolithic (B) fractured specimens, indicating a similar fracture mechanism between them, whereby the crack propagation (arrows) starts at the occlusal surface (a), and hackles and lines (b) perpendicular to crack origin may be observed.

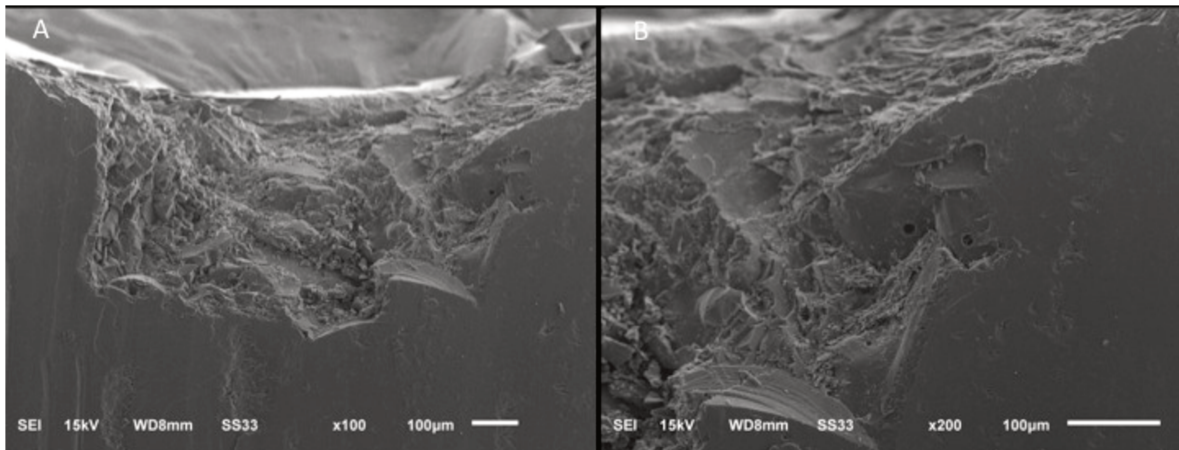


Figure 3. SEM micrograph of ceramic fracture surface showing a critical flaw (crack) in BL- bi-layer veneered fractured crown. Note the chipping at the occlusal surface (A), and voids inside the veneering layer (B).

Discussion

The application of artificial aging before the fracture strength test aimed to simulate the effect of the oral environment on zirconia-based crowns by associating cyclic loading, an antagonist tooth and artificial saliva. All these in vitro simulations were intended to represent expected clinical in vivo changes, which might result in the undesired phenomenon of low temperature degradation (LTD). 2.5 million mechanical cycles were selected to simulate 5 years aging in the oral environment, considering that an average adult would perform around 500,000 loading cycles/year^{24,25}. However, there is a large variation between number of cycles and the vertical loading applied in aging studies in the literature, with in vitro studies reporting the application of 5,000 to 400,000 cycles.^{23,26–30} Indeed, several studies performed 1,200,000 cycles with 5kg of vertical load.^{20,31–33}

All crowns survived the artificial aging in the chewing simulator. This result indicates a stable performance of zirconia-based crowns under a constant load of 8kg (78N) during 5 years. Previous studies that evaluated the clinical performance

of zirconia-based restorations, demonstrated a survival rate of 79-100% after 5 years, ³⁴⁻³⁶ with the most frequent clinical problem being the fracture of the veneering ceramic. Those results may be explained by the uneven masticatory loads presented in vivo, which also varies according to the type of food to be triturated by the posterior teeth. Moreover, other variables are present in the mouth, such as pH and temperature variations, and these effects on the fracture strength and chemical stability of all-ceramic crowns is not well known.

The null hypothesis, that the Y-TZP crown design, monolithic or bi-layer has no effect on fracture strength, was rejected. Therefore, the present study showed higher fracture strength of monolithic zirconia crowns in comparison to the bi-layer configuration.

Previous studies analyzing the fracture strength of all-ceramic monolithic crowns indicate a superior performance for the monolithic design. Monolithic lithium disilicate restorations and hand-layer veneered Y-TZP core evidenced that the highest fatigue load-to-failure values were presented by monolithic crowns, and the lowest for veneered Y-TZP crowns. ³⁷ Even though the monolithic system was made of lithium disilicate, better results were obtained when compared to bi-layer Y-TZP. According to the authors, the enhanced performance of monolithic crowns may be explained by the elimination of the interface between core and veneer, which is believed to be the weak link in bi-layer systems.

Another in vitro study evaluated the load-bearing capacity of four different zirconia based crowns, including: zirconia core with powder build-up porcelain (veneering technique), with CAD/CAM generated veneering (sintering technique), glazed full-contour zirconia, and polished full-contour zirconia. The results showed that veneered zirconia substructures had significantly less load-bearing capacity compared to all the other groups. ³⁸

Nevertheless, it is important to consider that all groups evaluated showed higher fracture load (PM= 3492.5N; GM= 3344.7N; BL= 2051.8N) than maximum

chewing forces reported in the literature. Previous studies mentioned that the average maximum biting force of healthy and young adults is approximately 700 N.^{39,40} Therefore, the results indicate that the fracture load of all groups tested in this study may withstand the clinical applications without restrictions. However, clinical reports of failed bi-layer zirconia-based restorations due to chipping or cracking are commonly reported in the literature.^{9–11}

In the present study, the groups referring to monolithic crowns, polished and glazed, showed a total core fracture pattern. This result was expected, since PM has only one material layer and GM has a thin glaze layer which leads to a bulk structural fracture. On the other hand, all the bi-layer crowns showed fracture at core/veneer interface. Failure mode at the veneer layer has been reported for bi-layers crowns, most commonly in powder build-up technique than in the sintering or pressed veneering technique.^{38,41} The powder build-up technique was used in the present study, and it is more sensitive and subject to variability due to the individual building and firing procedures. The process may result in the addition of impurities and porosities, which maximizes the risk of crack propagation (figure 3). Therefore, the technique and the low mechanical properties of the veneer material may be the reason for this mode of failure, as well as for the lower fracture strength presented by samples in BL group, since the inner coping was still intact after the mechanical testing. In contrast, there are some researches reporting complete failure (core/veneer) of all Lava CAD/CAM crowns⁴² and total coping fracture.⁴¹

There is no consensus about the mechanism to explain the origin of the LTD, but three mechanisms are proposed in the literature. The first is that water (H_2O) reacts with yttrium (Y_2O_3) to form yttrium hydroxide ($Y(OH)_3$), which depletes the stabilizing oxide sufficiently to allow transformation of the monoclinic phase. The second mechanism is water attack of the Zr–O bond, leading to stress accumulation due to movement of $-OH$ into the crystal structure. This motion generates lattice defects that act as nucleating agents for subsequent

transformation from the tetragonal-to-monoclinic phase. And the third theory is that, O_2^- (not OH^-) from water dissociation fills oxygen vacancies.¹⁴

The content of yttrium after aging was evaluated in previous studies. An in vitro study reported the loss of yttrium (from 6.76 wt% to 4.83 wt%) when Vita In-Ceram YZ was aged in boiled water for 7 days, which supports the first LTD's origin mechanism.⁴³ However, another study with the same experimental method reported no difference in the yttrium content after aging, even showing increase in monoclinic phase fractions (from 2 to 21%).¹⁴ This apparently contradictory result may be due to the different compositions of zirconia used.

In the current study there was no difference in the yttrium content among occlusal worn surfaces and undamaged surfaces. Thus, this result can support the hypothesis that the chemical composition of monolithic crowns was not affected by the occlusal loading.

The results of this study demonstrated that monolithic zirconia-based crowns might have reliable fracture strength after 5 years of occlusal loading. Indeed, the fabrication of monolithic zirconia restorations might allow for extended clinical application, reducing a major drawback, which is fracture of veneering ceramic. However, future researches concerning about if temperature or pH variations can influence in the fracture strength and chemical stability of monolithic zirconia crowns after artificial aging, should be conducted. And in vivo studies should be performed to evaluate the clinical behavior of monolithic zirconia restorations.

Conclusion

According to the results of this study, Y-TZP monolithic crowns (polished and glazed) present higher fracture strength than bi-layered veneered Y-TZP crowns. There was no evidence of yttrium depletion after 2.5 million cycles in artificial aging.

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CONCLUSÃO

De acordo com os resultados do presente estudo, as coroas monolíticas a base de zircônia apresentaram maior resistência à fratura do que as coroas de configuração bicamada, com infraestrutura em zircônia e cerâmica de cobertura.

As superfícies oclusais desgastadas das coroas monolíticas de zircônia não apresentaram perda de ítrio após 2,5 milhões de ciclos de envelhecimento artificial.

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* De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors. Abreviatura dos periódicos em conformidade com o Medline.

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APÊNDICE 1 Ilustrações de Materiais e Métodos.

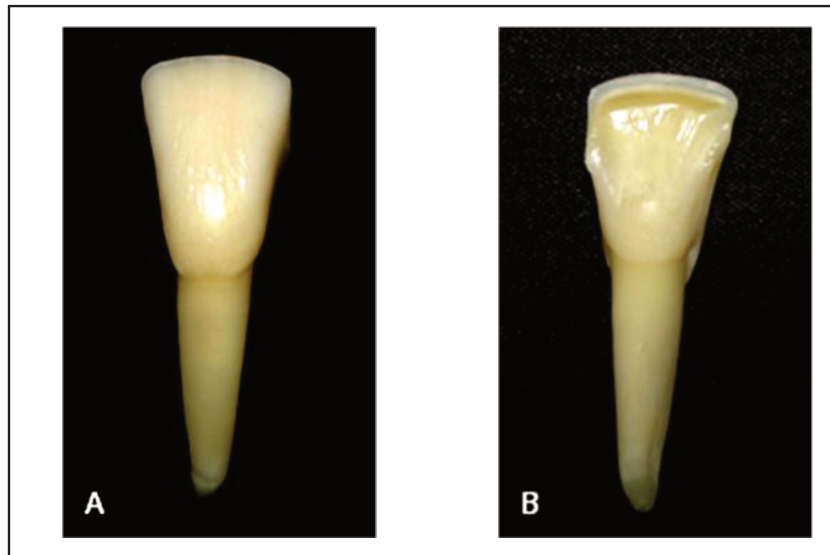


Figura 1 - Dente bovino: A) Face vestibular e B) face lingual.

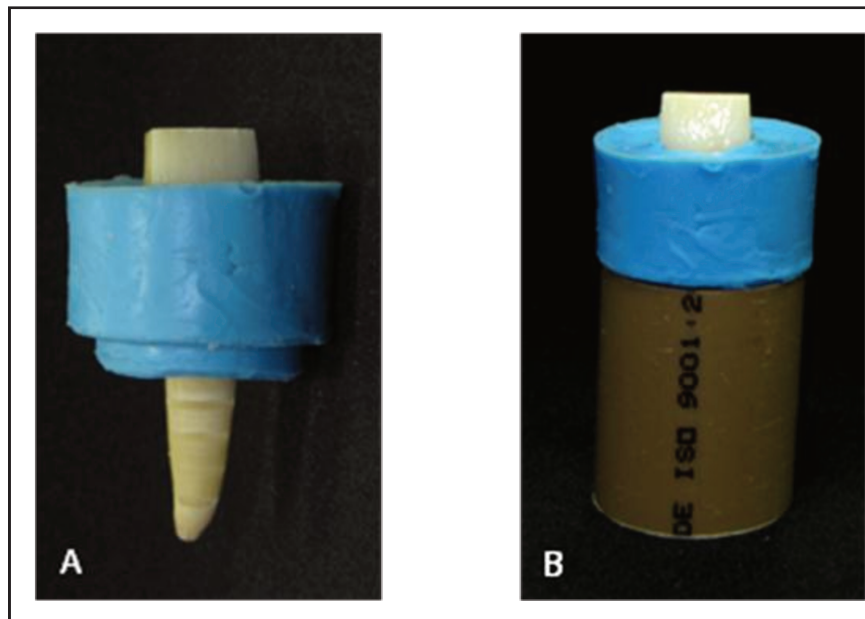


Figura 2 - A) Dente posicionado na matriz de silicone, B) conjunto dente/matriz posicionado para inclusão sobre o tubo de PVC.

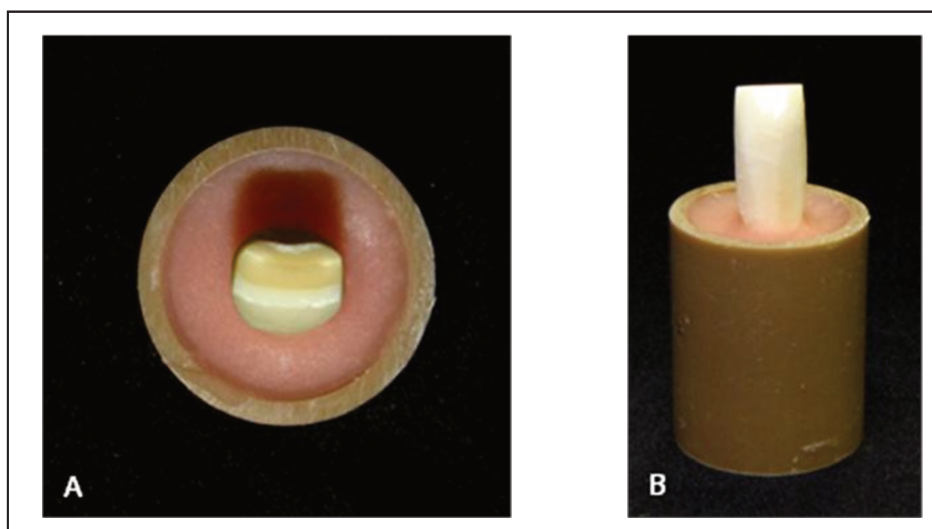


Figura 3 - Dente incluído : A) vista superior e B) vista frontal.

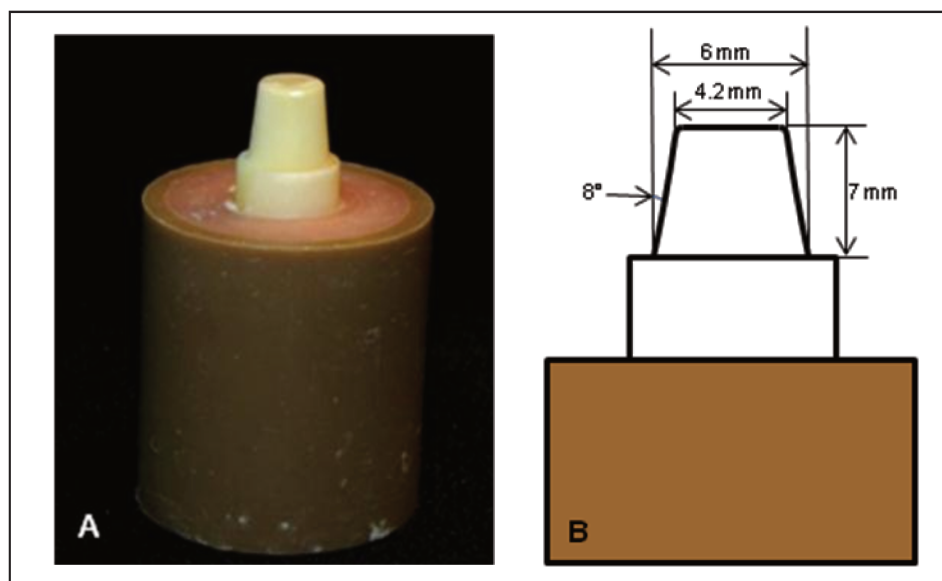


Figura 4 - A) Dente preparado e B) desenho esquemático do preparo.

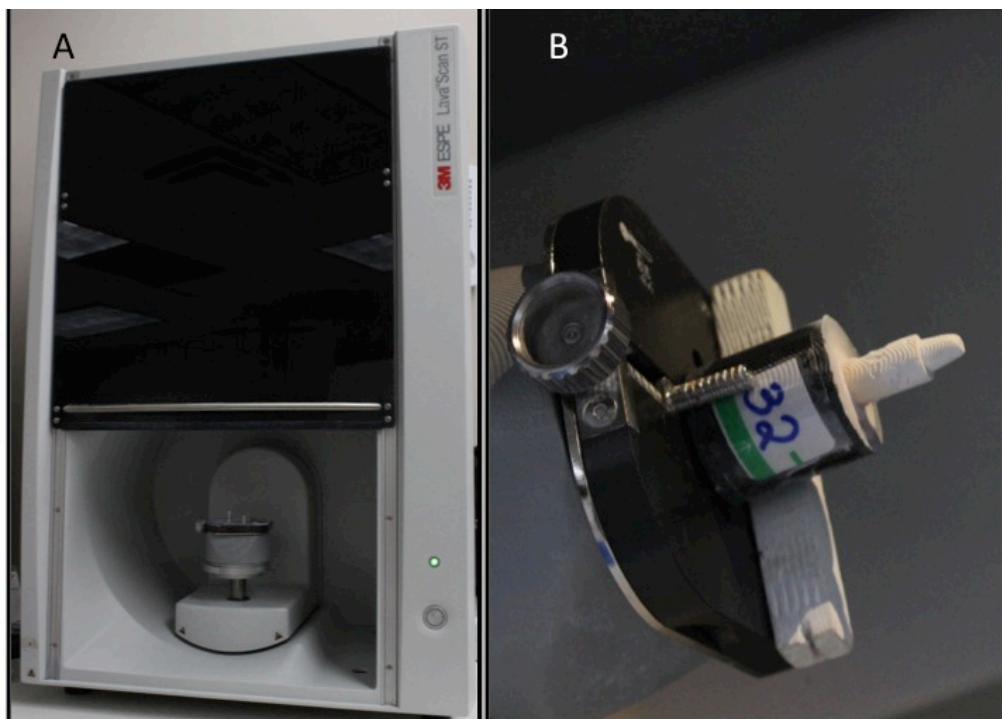


Figura 5 – A) Scanner ótico 3D e B) preparo sendo escaneado.

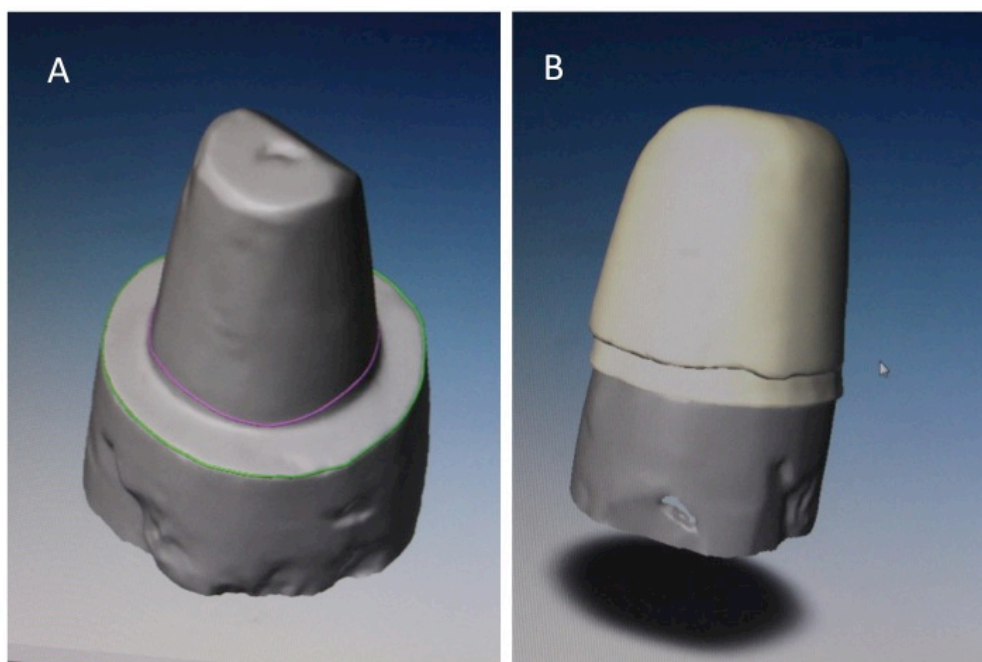


Figura 6 – A) Imagem do preparo escaneado e B) desenho da coroa.

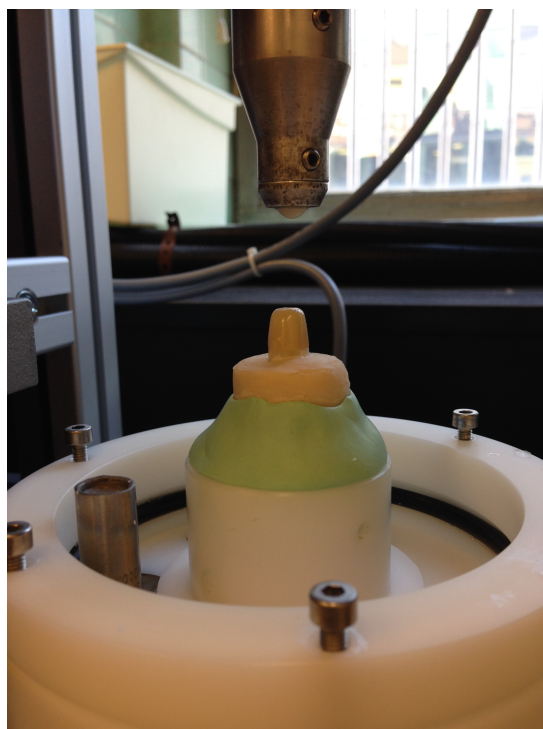


Figura 7 – Amostra posicionada na maquina simuladora de mastigação.

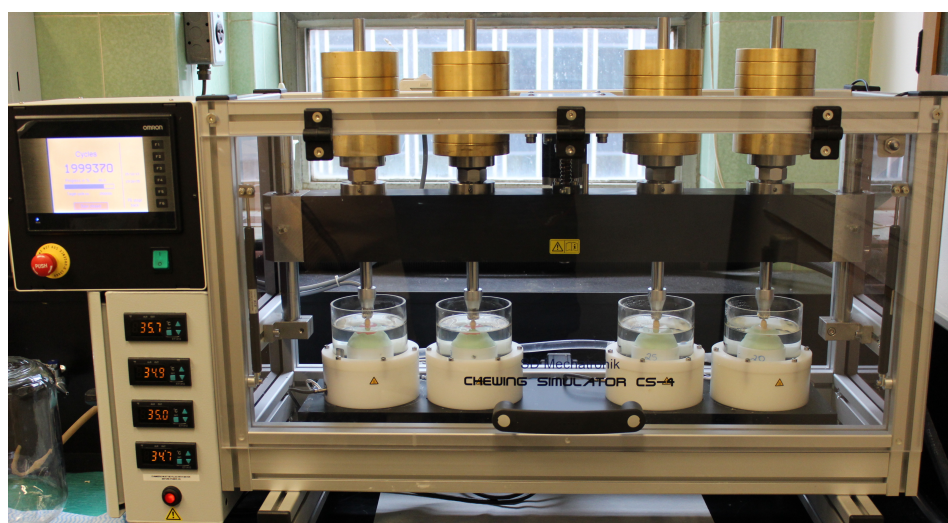


Figura 8 – Simuladora de mastigação durante o envelhecimento artificial.

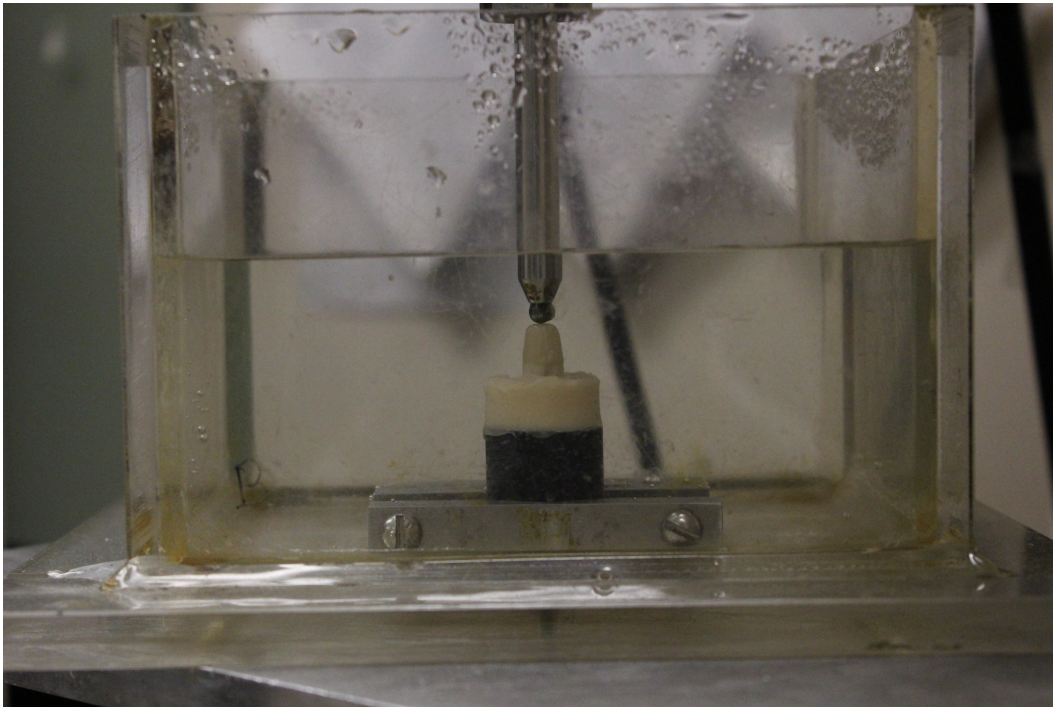


Figura 9 - Amostra posicionada durante o teste e resistência a fratura.

APÊNDICE 2 - Propriedades do materiais avaliados ².

Propriedades	Lava™ Plus
Resistencia flexural	> 1100 MPa
Modulo de elasticidade	210 GPa
Coeficiente de expansão térmica (25 °C – 500 °C) (ISO 6872)	10,5 ± 0,2 1/K
Dureza Vickers	> 1200
Media de tamanho dos grãos	0,4 µm
Densidade (ISO 13356)	6,08 g/cm ³

Propriedades	Lava™ Frame Ceram
Resistencia flexural (ISO 6872)	1272 MPa
Resistencia flexural (ISO 6872)	1625 MPa
Modulo de elasticidade	210 GPa
Dureza Vickers	> 1250
Media de tamanho dos grãos	0,5 µm
Densidade	6,08 g/cm ³

² Dados fornecidos pela empresa 3M ESPE
http://solutions.3m.com/wps/portal/3M/en_US/3M-ESPE-NA/dental-professionals/products/espe-catalog/~/_Lava-Plus-High-Translucency-Zirconia?N=5144617+3294798097&rt=rud

Propriedades	Lava™ Ceram Veneer Ceramic
Resistencia flexural (ISO 6872)	85 MPa
Modulo de elasticidade	80 GPa
Coeficiente de expansão térmica (25 °C – 500 °C) (ISO 6872)	10×10^{-6} 25-500°C
Dureza Vickers	530
Media de tamanho dos grãos	25 µm
Densidade	2,5 g/cm ³

ANEXO 1

Folha _____
Processo _____
Rubrica _____



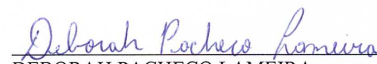
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Faculdade de Odontologia de Piracicaba

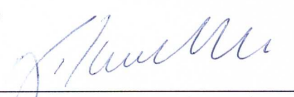


DECLARAÇÃO

As cópias de artigos de minha autoria ou de minha co-autoria, já publicados ou submetidos para publicação em revistas científicas ou anais de congressos sujeitos a arbitragem, que constam da minha Tese de Doutorado intitulada "RESISTÊNCIA A FRATURA DE COROAS MONOLITICAS E BICAMADA A BASE DE ZIRCONIA.", não infringem os dispositivos da Lei nº 9.610/98, nem o direito autoral de qualquer editora.

Piracicaba, 21 de Maio de 2014.


DEBORAH PACHECO LAMEIRA
RG: 3789411
Autor(a)


WILKENS AURELIO BUARQUE E SILVA
RG: 16.342.307
Orientador(a)

ANEXO 2

Submissions Being Processed for Author Grace de Souza, DDS, MSc, PhD

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 Action 	Manuscript Number 	Title 	Initial Date Submitted 	Status Date 	Current Status 
Action Links		Fracture strength of aged monolithic and bi-layer zirconia-based crowns.	May 13, 2014	May 13, 2014	Submitted to Journal

Page: 1 of 1 (1 total submissions)

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