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Taiane Secretti Missau

EFEITO DO CONDICIONAMENTO DE INLAYS CERÂMICAS COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO NA CARGA DE FRATURA SOB FADIGA DE PRÉ-MOLARES RESTAURADOS

Santa Maria, RS
2016

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

Orientadora:Prof[a] Dr[a].Marília Pivetta Rippe
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Aprovado em 26 de julho de 2016:

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Santa Maria,
RS 2016

DEDICATÓRIA

Aos meus pais, Janir e Anilto, pelo amor incondicional, apoio e suporte para que eu seguisse em frente.

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RESUMO

EFEITO DO CONDICIONAMENTO DE INLAYS CERÂMICAS COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO NA CARGA DE FRATURA SOB FADIGA DE PRÉ-MOLARES RESTAURADOS

AUTORA: Taiane Missau
ORIENTADORA: Marília Pivetta Rippe
CO-ORIENTADOR: Luiz Felipe Valandro

Este estudo avaliou o efeito do condicionamento de inlays de cerâmica feldspátic com diferentes concentrações de ácido fluorídrico na carga de fratura sob fadiga de pré-molares. Sessenta pré-molares superiores foram embutidos em cilindros de PVC com resina acrílica, 2 mm abaixo da junção cemento-esmalte e preparados com caixas oclusais (2 mm) e proximais (4mm) padronizadas através de uma máquina adaptada para preparo. Os dentes foram divididos de forma randomizada em três grupos (n = 20): HF1; HF5 e HF10 (condicionamento com ácido fluorídrico durante 60 s nas concentrações de: 1%, 5% e 10% respectivamente). Os dentes foram moldados, os troquéis obtidos escaneados e as restaurações fresadas através do sistema CAD/CAM a partir de blocos pré-fabricados de cerâmica feldspática (VitaBlocks Mark II). A superfície dentária foi tratada com ácido fosfórico (38%) seguida da aplicação do sistema adesivo (Single Bond). A superfície interna das inlays foi condicionada de acordo com os respectivos grupos, seguido da aplicação do agente de união silano. A cimentação das inlays foi realizada com cimento resinoso (RelyX ARC). A carga de falha sob fadiga foi determinada utilizando o método da escada (*staircase*) a uma freqüência de 10 Hz e 10^5 ciclos, cuja carga inicial foi de 585,5 N, aplicada somente sobre as vertentes das cúspides, através de um cilindro de 8mm de diâmetro acoplado à máquina de teste (Instron Electropuls E3000). Os dentes fraturados foram analisados sob estereomicroscoscópio para classificação da falha. Análise estatística dos dados foi feita com ANOVA 1-fator. Não houve diferença estatística dos valores médios na carga de fratura sob fadiga entre os grupos testados: HF1-448,5N (±79,09); HF5-360,75 N (±55,4); HF10-409,5 N (±121,1). Considerando o modo de fratura, houve uma predominância de fraturas de interface (50%), seguida de fratura de cúspide (34,6%). Conclui-se que as concentrações de ácido fluorídrico utilizadas (1, 5 e 10%) não influenciam na carga de fratura sob fadiga de inlays de cerâmica feldspática cimentadas em pré molares.

Palavras-chave: Ácido fluorídrico. Cerâmica Feldspática. Inlay. Carga de falha sob fadiga.

ABSTRACT

EFFECT OF ETCHING CERAMIC INLAYS WITH DIFFERENT HYDROFLUORIC ACID CONCENTRATION ON THE FATIGUE FAILURE LOAD OF PRE-MOLARS RESTORED

AUTORA: Taiane Missau
ORIENTADORA: Marília Pivetta Rippe
CO-ORIENTADOR: Luiz Felipe Valandro

The aim of this study was to evaluate the effect of different concentrations of hydrofluoric acid on the fatigue failure load of premolars restored with feldspathic ceramic inlays. Sixty upper premolars were embedded in PVC cylinders with acrylic resin up to 2 mm below the cement- enamel junction and prepared using an adapted device for that purpose. Teeth were randomly divided into three groups (n=20): HF1, HF5, and HF10, (conditioning with hydrofluoric acid for 60 s at concentrations of 1%, 5%, and 10%, respectively). Preparations were scanned using the CAD/CAM system with prefabricated blocks of feldspathic ceramic. The internal surfaces of the inlays were treated according to the groups, followed by application of silane coupling agent, and RelyX ARC resin cement was applied for cementation. The fatigue failure load was determined using the staircase method (10 Hz and 10^5 cycles in each step). The initial load (585.5 N) was applied on the slopes of the cusps through a cylinder attached to the test machine (InstronElectroPuls E3000). The fractured teeth were analyzed under a stereomicroscope for failure analysis. Statistical analysis was performed using one-way ANOVA. There was no statistical difference among the fatigue failure load of the tested groups: HF1 (448.5 N ±79.1), HF5 (360.7 N ±55.4), and HF10 (409.5 N ±121.1). Regarding the fracture mode, there was a predominance of interface fracture (50%), followed by cusp fracture (34.6%). It can be concluded that hydrofluoric acid concentrations used (1%, 5%, and 10%) did not influence the fatigue failure load of feldspathic ceramic inlays cemented on premolars.

Keywords: Hydrofluoric acid. Feldspathic ceramic. Inlay. Fatigue failure load

SUMÁRIO

1. INTRODUÇÃO GERAL

Atualmente é nítida a procura dos pacientes por restaurações estéticas inclusive na região posterior (DIETSCHI et al., 1990). Preparos dentais parciais mais conservadores são considerados menos retentivos e mais adesivo-dependentes (BOTTINO et al., 2009), sendo essa abordagem restauradora possível devido a evolução dos sistemas adesivos, dos cimentos resinosos e das cerâmicas odontológicas. Sobretudo, o sucesso clínico desse tipo de restauração depende substancialmente da excelente resistência de união entre o cimento resinoso à cerâmica e à estrutura dental. (HAYASHI et al., 2000; FRADEANI et al., 2002)

Em pré-molares e molares a confecção de inlays como abordagem restauradora é uma opção conservadora, já que representa um preparo minimamente invasivo e preserva a estrutura dentária (FEITOSA et al., 2013). Nesse caso, a perda de esmalte e dentina devido ao preparo diminui a rigidez do dente, e consequentemente leva a um aumento na deformação de cúspide sob tensão oclusal (GONZALEZ-LOPEZ et al., 2006), contudo, restaurações indiretas podem diminuir a quantidade de deflexão da cúspide (LEE et al., 2007). Alguns fatores afetam esta deformação, tais como o tamanho e forma da cavidade, e o módulo de Young do material restaurador (LEE et al., 2004). Como opção para esse tipo de restauração temos a cerâmica feldspática, cujas restaurações feitas desse material têm apresentado elevada taxa de sobrevivência e mostrado resistência à compressão e ao desgaste (AMARAL et al., 2011; LEITE et al., 2005).

Esse material é geralmente utilizado para a confecção de restaurações inlays, onlays e laminados e sua constituição apresenta conteúdo de quartzo, feldspato e alguns óxidos de metal que formam uma fase vítrea de estrutura amorfa (CARVALHO et al., 2011; KELLY et al., 2008), o que permite simular as propriedades ópticas da estrutura dental. Esta cerâmica pode ser utilizada em forma de blocos pré-fabricados para o sistema CAD/CAM (Computer-aided Design/Computer-aided Manufacture), os quais possuem grãos refinados que geram uma cerâmica livre de poros, produzindo restaurações mais duráveis, e relativamente mais resistentes à fratura com boa capacidade de polimento e alta resistência ao desgaste (KURBAD A & REICHEL et al., 2006; HEYMANN et al., 1996).

Essas cerâmicas são classificadas como ácido sensíveis, já que possuem sílica em sua constituição. Assim o procedimento de tratamento de superfície recomendado é o condicionamento da face de cimentação com ácido fluorídrico. Este método promove mudanças topográficas e permite o embricamento mecânico desta cerâmica com o cimento resinoso (SAAVEDRA et al., 2009), promovendo uma boa resistência de união que pode ser potencializada pelo agente silano, o qual proporciona a união química.

Contudo esta resistência adesiva pode ser ameaçada pelas tensões cíclicas da mastigação, que em uma determinada magnitude podem levar a falha da restauração por fadiga. Este tipo de falha é definida como o crescimento de fissuras microscópicas em áreas de concentração de tensão com cargas contínuas fazendo com que essas fendas se fundam em uma fissura crescente que enfraquece a restauração, ocorrendo a falha. No que se refere à fadiga nas estruturas dentárias, a maioria das falhas mecânicas são atribuídas a um processo que encontra o seu fim catastrófico só depois de muitos anos de serviço (WISKOT et al., 1995).

Nesse sentido, embora o protocolo de adesão mencionado esteja bem estabelecido na literatura, reiteramos que ainda não existe relato na literatura quanto à concentração ideal deste ácido a ser utilizado referente a carga de fratura sob fadiga de pré-molares restaurados com inlays cerâmicas. Assim, o objetivo do presente estudo é avaliar o efeito do condicionamento com diferentes concentrações de ácido fluorídrico na carga de fratura sob fadiga de pré-molares restaurados com *inlay* de cerâmica feldspática.

1 EFEITO DO CONDICIONAMENTO DE INLAYS CERÂMICAS COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO NA CARGA DE FRATURA SOB FADIGA DE PRÉ-MOLARES RESTAURADOS

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EFFECT OF ETCHING CERAMIC INLAYS WITH DIFFERENT HYDROFLUORIC ACID CONCENTRATION ON THE FATIGUE FAILURE LOAD OF PRE-MOLARS RESTORED

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Running title: Hydrofluoric acid on the fatigue failure load of feldspathic inlays.

EFFECT OF ETCHING CERAMIC INLAYS WITH DIFFERENT HYDROFLUORIC ACID CONCENTRATION ON THE FATIGUE FAILURE LOAD OF PRE-MOLARS RESTORED

CLINICAL RELEVANCE STATEMENT

Premolars restored with feldspathic ceramic inlays (milled by CAD-CAM system) presented the same fatigue failure load, when different acid concentrations (1%, 5%, and 10%) were used to treat the inner surface of restorations before cementation.

ABSTRACT

The aim of this study was to evaluate the effect of different concentrations of hydrofluoric acid on the fatigue failure load of premolars restored with feldspathic ceramic inlays. Sixty upper premolars were embedded in PVC cylinders with acrylic resin up to 2 mm below the cement- enamel junction and prepared using an adapted device for that purpose. Teeth were randomly divided into three groups (n=20): HF1, HF5, and HF10 (conditioning with hydrofluoric acid for 60 s at concentrations of 1%, 5%, and 10%, respectively). Preparations were scanned and the restorations were machined by CAD/CAM system from blocks of feldspathic ceramic. The internal surfaces of the inlays were etched, the silane coupling agent applied and RelyX ARC resin cement was used for cementation. The fatigue failure load was determined using the staircase method (10 Hz and 10^5 cycles in each step). The initial load (585.5 N) was applied on the slopes of the cusps through a cylinder attached to the test machine (Instron Electro Puls E3000). The fractured teeth were analyzed under a stereomicroscope for failure analysis. Statistical analysis was performed using one-way ANOVA. There was no statistical difference among the fatigue failure load of the tested groups: HF1 (448.5 N ±79.1), HF5 (360.7 N ±55.4), and HF10 (409.5 N ±121.1). Regarding the fracture mode, there was a predominance of interfacial fracture (50%), followed by cusp fracture (34.6%). It can be concluded that hydrofluoric acid concentrations used (1%, 5%, and 10%) did not influence the fatigue failure load of feldspathic ceramic inlays cemented on premolars.

Keywords: Feldspathic ceramic, Inlay, Fatigue failure load, Hydrofluoric acid.

INTRODUCTION

Nowadays, esthetic and minimally invasive restorations are made possible through adhesive dentistry. Despite the fact that inlays are a conservative restoration option when compared with partial dentures (FPDs),[1] their preparation leads to enamel and dentin loss, which decrease resistance mainly in premolars, since it increases the cusp deflection under occlusal tension.[2] Moreover, Costa *et al*.[3] showed that the cavity size is a significant factor as it influences the stress distribution and fracture strength of premolars.

Another factor that can influence the strength of teeth restored with inlays is the type of restoration. According to Lee *et al*.,[4] indirect restorations decrease the cusp deflection due to the absence of polymerization shrinkage of the restorative material in the oral environment. On the other hand, shrinkage in direct restorations results in a large degree of cusp deflection, creating micro-cracks in the tooth structure. Thus, feldspathic ceramic is widely used for indirect restorations such as inlays, onlays, veneers, and covering ceramics of FPD. Furthermore, this ceramic has wear strength and high survival rates and with stands high values of compression.[5,6]

The clinical success of ceramic inlay restorations is based on a durable bond strength between the resin cement, the ceramic, and the tooth structure,[7,8] since the expulsive preparation for such restorations has very low mechanical friction between the preparation wall and the inner surface of the restoration. Thus, the higher the bond strength between the tooth and the restoration, the lower the cusp deflection.[3] Moreover, feldspathic ceramics are classified as acid-sensitive due to the presence of silica on their constitution.[9,10,11,12] Therefore, the recommended surface treatment is the conditioning of the cementing surface with hydrofluoric acid, which promotes topographical changes and allows mechanical interlocking of this ceramic with resin cement.[13]

However, the ceramic surface topography depends not only on its composition, but also on the acid concentration and etching time.[14] Venturini *et al*.[15] reported that 3%, 5%, and 10% hydrofluoric acid promoted a higher and a more stable bond strength of resin to feldspathic ceramic after long-term aging than 1% hydrofluoric acid. These results showed that resin adhesion to this type of ceramic seems to be dependent on micro-topographical changes. Higher acid concentrations produce more intense surface changes, leading to greater mechanical interlocking.[16,17] However, when the same concentrations were tested in relation to flexural strength, Venturini *et al*.[18] showed that hydrofluoric acid weakens the feldspathic ceramic, regardless of the concentration, when compared with no etching treatment.

Consequently, there is a modification of the "defects" present in the ceramic surface and sub- surface, which could impact on the mechanical behavior of these restorations when exposed to cyclic intermittent loading. Besides, the quality of the adhesive interface may have an impact on bond strength values, leading to a higher or lower cusp deflection.[3] While hydrofluoric acid etching of the feldspathic ceramic promotes appropriate bond strength values,[17,19] Addison *et al*.[20] commented that this process may weaken the ceramic surface depending on the acid concentration. Therefore, it is extremely important to find the hydrofluoric acid concentration that, simultaneously, enhances adhesion to these ceramics and does not promote their weakening. Moreover, this possible weakening associated with the cyclic loading of mastication may lead to fatigue failure of the restoration that occurs when the final loading cycle exceeds the mechanical capacity of the material or restored tooth.[21]

The cyclic loading is especially important when posterior teeth are considered, mainly the upper premolars, which suffer more vertical fractures with possible loss of teeth.[22] The vertical fractures in upper premolars are related to their complex anatomy and location, in an area of high masticatory load.[3] Additionally, some researchers believe that failures in restorations start at the cementing surface and appear to be involved with tensile stress between the ceramic and the luting agent.[23,24]

Therefore, concerning a test set-up that simulates the real restorative scenario with ceramic inlays under cyclic mechanical loading, there is no study indicating the optimal concentration of hydrofluoric acid to promote stable adhesion without weakening the restoration. Moreover, the higher the bond strength, the lower the cusp deflection.[25] Costa *et al*.[3] reported that etching procedure has a significant impact on the adhesive interface, and it can influence the fatigue failure load of tooth restored with ceramic inlays.

Thus, the objective of this study was to evaluate the effect of different concentrations of hydrofluoric acid on the fatigue failure load of premolars restored with inlays of feldspathic ceramics milled with the CAD-CAM system and to evaluate the mode of failure of the restored premolars. The null hypothesis tested was that different acid concentrations would not influence the fatigue failure load of teeth restored with ceramic inlays.

MATERIALS AND METHODS

Experimental Design

This present research was approved by the Research Ethics Committee of Federal University of Santa Maria (UFSM) (N^o. 1.178.683).

Sixty extracted human premolars were provided by the Human Teeth Bank of the UFSM and stored in water, at environmental temperature (25^{o}C about). Teeth were selected following the inclusion criteria of absence of visible cracks or caries cavities under visual examination. The specimens were randomly divided into three groups (n=20) (Table 1). After teeth randomization (www.randomizer.org), buccolingual and mesiodistal measurements of each tooth were performed with a digital caliper (Starrett 727, StarrettIndustria e ComercioLtda, Itu, Brazil) to verify the homogeneity of teeth size in each group through the Levene test, which showed randomization was done satisfactorily.

Teeth were embedded in a plastic cylinder (h=14mm, Ø=25 mm) containing chemically cured acrylic resin (Dencrilay, Dencril, Caieiras, Brazil) up to 2 mm below the cemento-enamel junction, with the occlusal surface parallel to the horizontal plane.

Tooth Preparation

Standardized cavity preparations (inlay type) were performed in teeth, using a conical trunk diamond bur with rounded angles (KG Sorensen 3131, Barueri, SP, Brazil). Burs were mounted on a high-speed hand piece fixed to a modified optical microscope (Figure 1).

First, a mesio-occlusal-distal (MOD) cavity was prepared to a depth of 2 mm under cool water. Then, the proximal boxes were made 2 mm deeper in relation to the previously prepared pulpal wall, with the same diamond bur. Preparations had the following dimensions: occlusal box depth, 2 mm; proximal box depth, 4 mm; occlusal isthmus similar to the bur diameter, and rounded internal line angles. Each diamond bur was used for the preparation of five teeth. Afterwards, all preparations were polished with diamond burs with the same shape and lower grit (extra fine, KG Sorensen 3131FF; Barueri, SP, Brazil) (Figure 2).

Production/milling of inlays

Cavities were impressed using addition silicone and a one-step impression (EliteHD, Zhermack, BadiaPolesine, Italy, Batch #122842). Impressions were poured using type IV die stone (Durone IV, Dentsply, Petrópolis, Rio de Janeiro,Brazil). All inlays were made in the CEREC inLab milling machine (MC XL model, Sirona Dental Systems, Benshein, Hessen, Germany). Then, casts were sprayed with scanning powder (Optispray CEREC, Sirona, Benshein, Hessen, Germany) and optically captured by scanning (inEos Blue, Sirona, Benshein, Hessen, Germany). The image was sent to software, which formed a three- dimensional virtual model. The cement space in the software of the CAD/CAM system was pre-established and standardized at 90 µm. After inlays design, restorations were milled from feldspathic ceramic blocks (Vita Mark II for Cerec/inLab, 2M2C / I12, and 2M3C / I12 Vita Zahnfabrik, Bad Säckingen, Germany).

Ceramic Surface Treatment and Cementation

The internal surfaces of the inlays were treated with different concentrations of hydrofluoric acid: 1%, 5%, and 10% (FGM, Joinville, Brazil). The protocol was the same for all groups: etching time of 60 s, rinsing with air–dry spray for 30 s, and air-drying for 30 s. Then, a silane coupling agent (ESPE-Sil, 3M ESPE, Neuss, Germany) was applied and allowed to dry for 5 min, as recommended by the manufacturer. Teeth surfaces were conditioned with 37% phosphoric acid (Atacktec CAITHEC, São José dos Pinhais, Brazil) for 20 s, followed by washing and drying. The adhesive system (Single Bond, 3M ESPE, Sumaré, Brazil) was applied to the surfaces for 20 s, air-sprayed, and then photoactivated (Radii Cal, SDI, Bayswater, Australia) for 20s. The resin cement (RelyX ARC, 3M ESPE, Sumaré, Brazil) was mixed, following the manufacturer's instructions, and applied to the inner surface of the ceramic inlay. The restoration was then positioned on the preparation, and a load of 750 g was applied over the occlusal inlay surface for 1 min. The excess resin cement was removed, and photoactivation (Radii-cal, SDI Limited, Bayswater, VIC, Australia) was performed for 20 s in each surface. After cementation, the specimens were stored in distilled water at 37°C for, at least, seven days before conducting the staircase load experiment.

Fatigue Failure Load

First, a monotonic compression test was performed with two teeth in a universal testing machine (DL-2000, EMIC, Pines, PR, Brazil), with the same piston used in the fatigue test. Thus, the initial load of the fatigue test was 60% of the mean of the monotonic failure loads (585.5 N).[26]

The fatigue test was conducted under water, according to the staircase method,[26] in an Instron testing machine (InstronElectroPuls E3000, Instron Corporation, Norwood, MA, United States). The sample was placed on a metal platform at an angle of 90°, in which a cylinder piston (Ø=8mm) applied a load only on the cusp surfaces, without contact with the restoration. An occlusal marker (Carbon Paper Film Parkell Accu-S017, New York, USA; 21 µm thick) was used to ensure that the piston did not touch the inlays. The fatigue failure load was determined for 10^5 cycles at a frequency of 10 Hz. The load of the successive steps was increased or decreased according to the survival or failure of the previous specimen. This increase or decrease corresponded to 10% of the pre-established initial load (58.5N). All steps were performed in water at room temperature. The fatigue failure load (σ) and standard deviation (SD) were determined using Equations 1 and 2, respectively, according to Collins: [26]

$$\sigma = \sigma_{X0} + d\left(\frac{\sum i n_i}{\sum n_i} \pm 0.5\right) \quad \text{(Eq. 1)}$$

$$\text{E } DP = 1.62d\left(\frac{\sum n_i \sum i^2 n_i - (\sum i n_i)^2}{(\sum n_i)^2} + 0.029\right) \quad \text{(Eq. 2)}$$

Where *X0* is the lower load considered in the analysis, and *d* is the fixed increment. In order to determine the fatigue failure load, analyses were based on data of less frequent events (survival or failure). In equation 2, the negative sign was used if the less frequent event was failure, and the positive sign was used when survival was the less frequent event. The lowest load level considered was designated i=0, i=1 the next, and so forth, and *ni* was the number of failures or survival in the given load level.

Failure Analysis

After visual examination, fractures were classified on a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) according to the following criteria: Cusp failure (Cusp), small fractures and/or cracks in the tooth structure; Restoration failure (Rest), fractures and/or cracks most evident in the restoration, and Interface failure (Interf), fracture and/or crack at the interface with propagation through the restoration.

Topographical analysis under scanning electron microscopy of the ceramic conditioned surfaces

Six milled restorations of feldspathic ceramic were conditioned with 1%, 5%, or 10% hydrofluoric acid as the tested inlays were conditioned. Two specimens (milled) of each group were subjected to morphological analysis in scanning electron microscopy (SEM). For the analysis by SEM (MEV-FEG, Inspect F50, FEI, Tokyo, Japan), the specimens were properly metalized to be analyzed under different magnifications (500x, 1000x, and 2000x).

Statistical Analysis

Fatigue failure load values were analyzed using one-way ANOVA test ($p < 0.05$).

RESULTS

One-way ANOVA revealed no significant differences among the tested groups (p=0.14) (Table 2). Survival was the event less frequent for HF5 and HF10 groups, and failure was the event less frequent for HF1 group (Figure 3). Most failures had their origin at the interface between teeth and restoration (Tables 3 and 4). An irreparable fracture, 3 mm below the cement-enamel junction, was observed only in the HF10 group.

The micrographs of the ceramic surfaces were noticeably different according to the acid concentration regarding the untreated ceramic surface (Figure 5).

DISCUSSION

The present study found that the fatigue failure load of premolars restored with feldspathic ceramic inlays was not influenced by different concentrations of hydrofluoric acid (1%, 5%, and 10%), and, therefore, the null hypothesis was accepted.

This could be explained by the use of silane coupling agent, which was applied in all specimens and enhanced the bond strength through chemical bonding between the resin cement and the ceramic material.[9,10,14] Moreover, it is known that the use of resin cement can increase the fracture resistance by means of blunting the defects of ceramic restorations.[27,28] These two factors could justify the similar fatigue failure load among the tested groups.

Cusp deflection and adequate bonding are factors related to resistance to fracture.[3] The type of cement used and the quality of the bonding interface between tooth and restoration may impact the displacement values: the higher the adhesive strength, the lower the cusp deflection.[3] In terms of adhesion, hydrofluoric acid etching is indispensable to promote effective bond strength between resin cements and silica-based ceramic surface.[29] The acid etching changes the ceramic surface topography, increasing the roughness, and creates micromechanical interlocking when combined with a resin cement.[30,31] This step is followed by the application of a silane coupling agent, which promotes chemical adhesion.[9,32]

On the other hand, some studies have suggested that different hydrofluoric acid concentrations and etching times can weaken ceramic surface.[14, 33,34] However, according to Venturini *et al*.[18], who tested the same acid concentrations as those in this study, reported no statistical difference in flexural strength among the acid concentrations and showed a reduction in strength after etching only when compared with untreated ceramic. They suggested that the pores promoted after hydrofluoric acid etching could act as source of crack initiation.

Although the fracture origin of all inlays occurred at the interface tooth/inlay in the occlusal face, the fracture itself occurred in different parts of the teeth among the groups. There was a predominance of interface failure (50%), followed by cuspal failure (34.6%). More than 50% of the failures were at the tooth/inlay interface and were found in the HF5 group, which presented failures with crack propagation along the restoration interface (77.7%) (Table 3). These results might indicate that etching with 5% hydrofluoric acid created topographic defects that the resin cement could not fill properly due to its viscosity, creating discontinuity areas in the material. These areas may, then, concentrate tensile stress responsible for the crack initiation and propagation that leads to failure. The cuspal failure can be assigned to the device set-up used for the fatigue testing, which produced stress on cusp surface[23] through the compression load, given that the piston was near to cusp tips.[1] This is consistent with a previous report[3], which indicated, using finite element analyses, that fractures started on the occlusal surface (at the load point) and propagated in a cervical direction.

SEM images clearly revealed the progressive effect of different hydrofluoric acid concentrations on the ceramic microstructure compared with untreated ceramic surface (Figure 5 A-C), showing greater dissolution of the glassy matrix and the presence of pores in the surface after etching. These pores could act as sources of crack initiation. However, although the SEM micrographs demonstrated more intense alterations in the ceramic surface topography as a result of higher acid concentrations, no significant difference in the fatigue failure load of inlays was noted for the tested acid concentrations.

One of the statistical designs used in fatigue studies with dental ceramics is the staircase method. Therefore, the staircase method was chosen due to its viability and low variability.[28] For this, at least 15 to 30 specimens, after the beginning of the up-and-down pattern (point where the first reversal occurs), are required to adequately perform the test.[26] Thereby, the current research followed protocols to approximate to the clinical failures of ceramic inlays with cracks starting from the cementation surface.[23] Besides, the load was applied on the cusp and not on the interface as the aim was to verify if the bond between teeth and ceramic inlay would influence the cusp deflection, and, according to the failure analysis, it seems it has been confirmed.

A monotonic test with small-diameter ball indenters creates a stress state resulting primarily in surface damage that is not seen as part of clinical failure.[23] Our study used restored premolars submitted to a cyclic loading resembling the clinical situation. This type of load occurs in the mouth and can be simulated in laboratories with controlled parameters such load, total number of the cycles, and frequency.[37] *In vitro* studies have inherent limitations, and some clinical conditions cannot be simulated. This *in vitro* study does not fully simulate the forces in the oral environment, where teeth are subjected to various angle forces. Thus, it is difficult to draw direct correlations to the clinical performance if lateral forces were not simulated. Another limitation the achievement of standardization regarding functional age of teeth, morphologic variations of the pulp, and abnormalities in dentin composition before extraction.[38,39] Therefore, the results of this study require careful interpretation. Further studies should be performed with additional investigations with other groups of teeth, using other methodologies (such as stepwise), and another ceramic material.

CONCLUSION

The different hydrofluoric acid concentrations used, 1%, 5%, and 10%, do not affect the fatigue failure load of the premolars restored with feldsphatic ceramic inlays.

ACKNOWLEDGMENTS

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TABLES

Table 1. Experimental design.

Group	Ceramic Surface Treatment
HF 1	Etching with 1% hydrofluoric acid*
HF 5	Etching with 5% hydrofluoric acid*
HF 10	Etching with 10% hydrofluoric acid**

* Condac Porcelana, FGM, Santa Catarina, Brazil. ** Experimentally formulated by FGM.

Table 2. Fatigue failure load (σ) and standard deviation (SD) of the specimens tested by the staircase method.

Group	Fatigue failure load (± SD)
HF 1	488.5 (±79.1)
HF 5	360.75 (±55.4)
HF 10	409.5 (±121.2)

Table 3. Failure mode distribution in each experimental group.

	Failure Mode			
Group	**Cusp**	**Rest**	**Interf**	**Total**
HF 1	3 (50%)	1 (16.66%)	2 (33.33%)	6 (100%)
HF 5	1 (11.11%)	1 (11.11%)	7 (77.77%)	9 (100%)
HF 10	5 (45.45%)	2 (18.18%)	4 (36.36%)	11 (100%)
Total	9 (34.61%)	4 (15.38%)	13 (50%)	26 (100%)

Cusp: small cracks and/or fractures in tooth structure; Rest: fractures and/or cracks in restoration; Interf: cracks and fracture with the origin at the interface.

FIGURES

Figure 1- Adapted device to make standardized preparations.

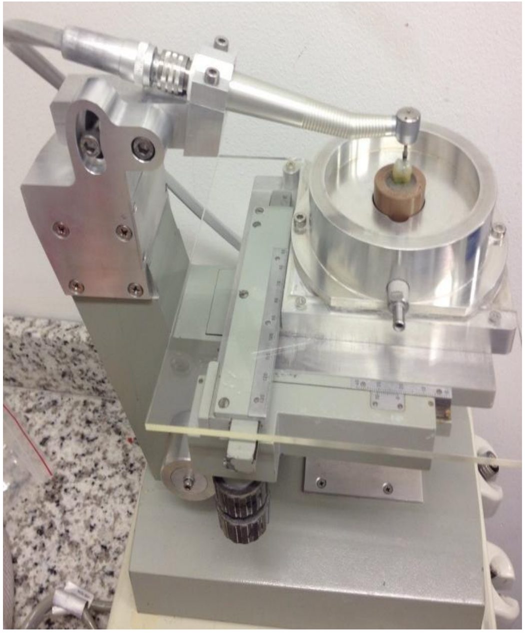
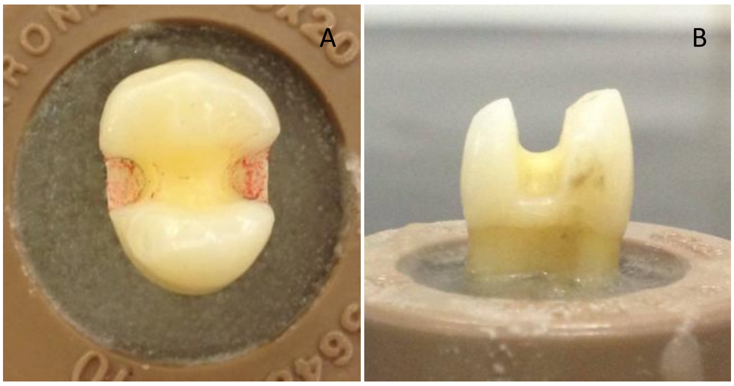


Figure 2- Prepared teeth for inlay.



A) Occlusal view; B) Mesial view.

Figure 3-Fatigue failure load events for each experimental group. The square hollow refers to the test beginning.

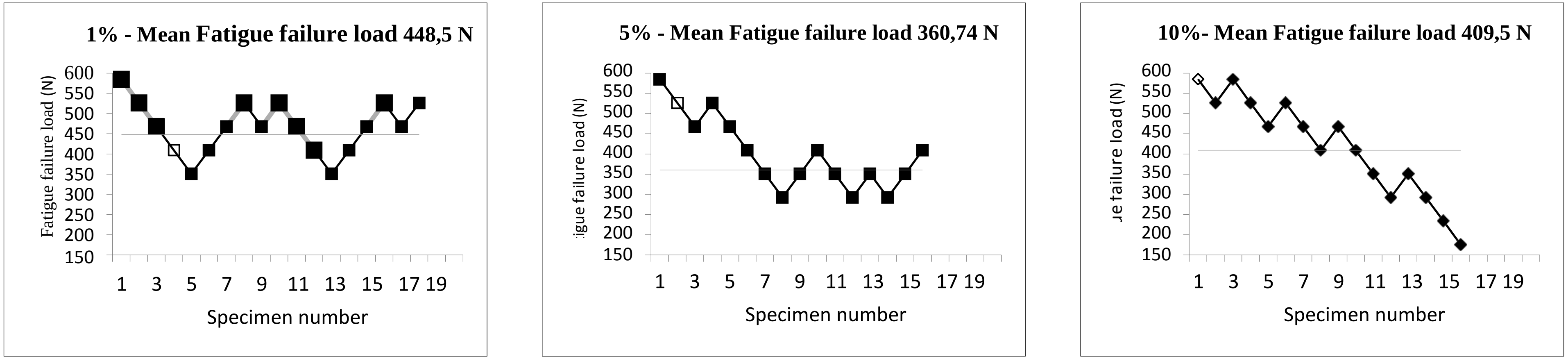
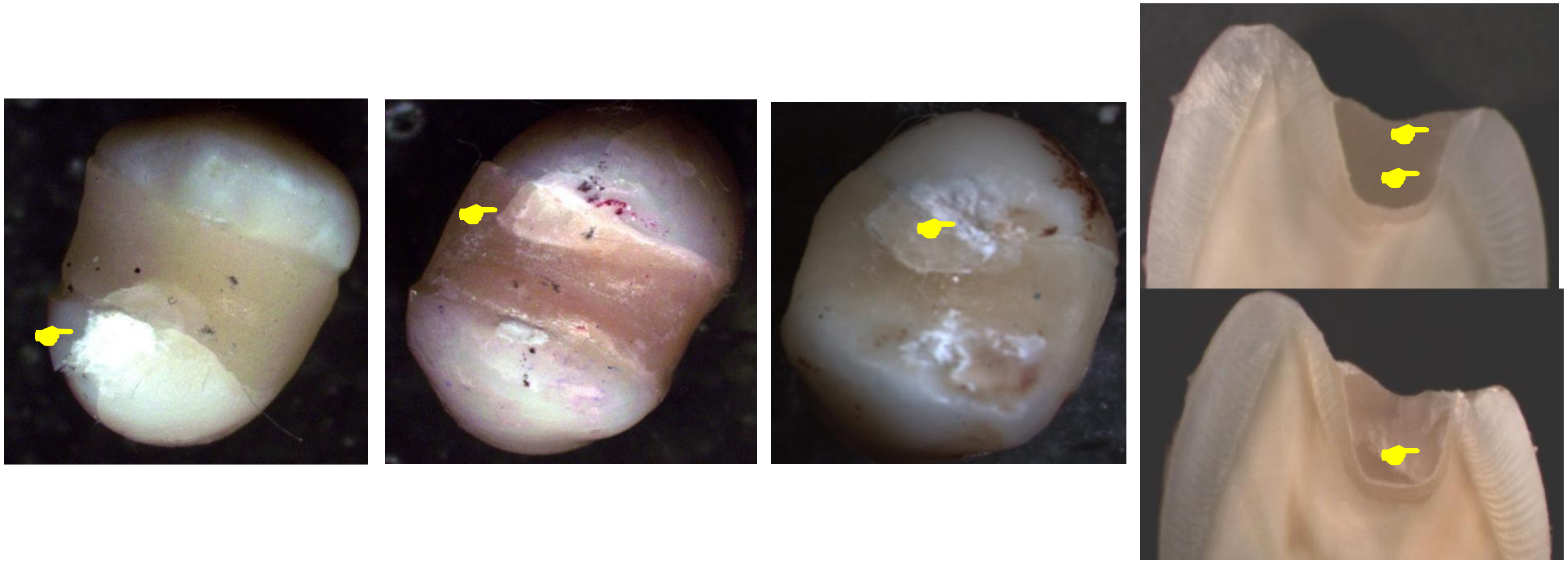


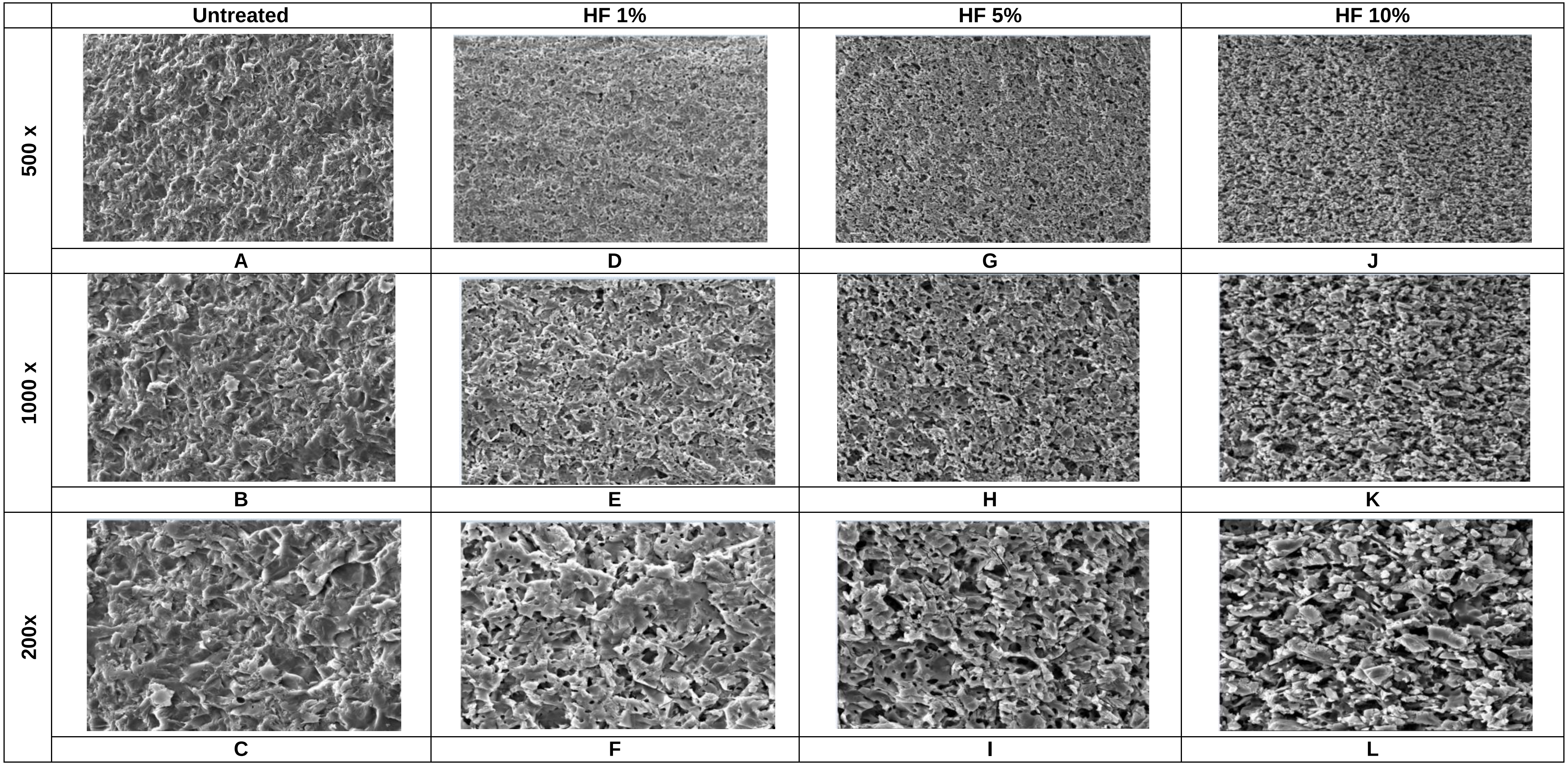
Figure 4- Representative images of the failure modes obtained with stereomicroscope.



A. Cusp Failure **B. Rest failure** **C. Interf failure** **D. Cross-section Interf. failure**

**A- Cusp failure: small cracks and/or fractures in the tooth structure; B- Restoration failure: fractures and/or cracks in the restoration; C- Interface failure: cracks and fractures with origin at the interface. D- Cross-section of a restored tooth with interface failure; the indicators show that the origin of the failure seems to start at the interface tooth/restoration.*

Figure 5- Representative micrographs of ceramic surface after different acid etching treatments compared with untreated surface. From top to bottom: 500x, 1000x, 2000x magnifications. A-C) untreated surface; (D-F) treated with HF 1%;(G-I) treated with HF 5%; (J-L) treated with HF 10%.The surface patterns of the etched surfaces were noticeably different. Higher hydrofluoric acid concentrations promoted deeper and more evident craters and pits, while slight topographic changes were created for the HF1 group.



3 CONCLUSÃO

A confecção de inlays como abordagem restauradora em pré-molares é uma ótima opção já que nosso estudo mostrou altos valores de carga de falha sob fadiga de pré-molares restaurados com inlays cerâmicas. Concomitante a isso, a utilização de diferentes concentrações de ácido fluorídrico não afetam a carga de falha sob fadiga de inlays de cerâmica feldspática cimentadas em pré-molares de acordo com o presente estudo.

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ANEXO A- NORMAS PARA PUBLICAÇÃO NO PERIÓDICO OPERATIVE DENTISTRY

INSTRUCTIONS TO AUTHORS

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        - identifying information such as:
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     - affiliation (e.g. Department of Dental Materials, School of Dentistry, University of Michigan)

  3. **MENTION OF COMMERCIAL PRODUCTS/EQUIPMENT** must include:
     - full name of product
     - full name of manufacturer
     - city, state and/or country of manufacturer

  4. **MANUSCRIPTS AND TABLES** must be provided as Word files. Please limit size of tables to no more than one US letter sized page. (8 ½ ” x 11”)

  5. **ILLUSTRATIONS, GRAPHS AND FIGURES** must be provided as TIFF or JPEG files with the following parameters

  - line art (and tables that are submitted as a graphic) must be sized at approximately 5” x 7” and have a resolution of 1200 dpi.
  - gray scale/black & white figures must have a minimum size of 3.5” x 5”, and a maximum size of 5” x 7” and a minimum resolution of 300 dpi and a maximum of 400 dpi.
  - color figures must have a minimum size of 2.5” x 3.5”, and a maximum size of 3.5” x 5” and a minimum resolution of 300 dpi and a maximum of 400 dpi.
  - color photographs must be sized at approximately 3.5” x 5” and have a resolution of 300 dpi.

- **OTHER MANUSCRIPT TYPES**

  1. **CLINICAL TECHNIQUE/CASE STUDY MANUSCRIPTS** must include:
     - a running (short) title
     - purpose
     - description of technique
     - list of materials used
     - potential problems
     - summary of advantages and disadvantages
     - references (see below)

  2. **LITERATURE AND BOOK REVIEW MANUSCRIPTS** must include:
     - a running (short) title
     - a clinical relevance statement based on the conclusions of the review
     - conclusions based on the literature review…without this, the review is just an exercise
     - references (see below)

- **FOR REFERENCES**

  REFERENCES must be numbered (superscripted numbers) consecutively as they appear in the text and, where applicable, they should appear after punctuation.

  The reference list should be arranged in numeric sequence at the end of the manuscript and should include:
  1. Author(s) last name(s) and initial (ALL AUTHORS must be listed) followed by the date of publication in parentheses.
  2. Full article title.
  3. Full journal name in italics (no abbreviations), volume and issue numbers and first and last page numbers complete (i.e. 163-168 NOT attenuated 163-68).
  4. Abstracts should be avoided when possible but, if used, must include the above plus the abstract number and page number.
  5. Book chapters must include chapter title, book title in italics, editors’ names (if appropriate), name of publisher and publishing address.
  6. Websites may be used as references, but must include the date (day, month and year) accessed for the information.

7. Papers in the course of publication should only be entered in the references if they have been accepted for publication by a journal and then given in the standard manner with "In press" following the journal name.
8. **DO NOT** include unpublished data or personal communications in the reference list. Cite such references parenthetically in the text and include a date.

EXAMPLES OF REFERENCE STYLE

- Journal article: two authors
  Evans DB & Neme AM (1999) Shear bond strength of composite resin and amalgam adhesive systems to dentin *American Journal of Dentistry* **12(1)** 19-25.

- Journal article: multiple authors
  Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin *Critical Review of Oral and Biological Medicine* **8(3)** 306-335.

- Journal article: special issue/supplement
  Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **(Supplement 6)** 119-144.

- Abstract:
  Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **82(Special Issue B)** Abstract #0051 p B-19.

- Corporate publication:
  ISO-Standards (1997) ISO 4287 Geometrical Product Specifications Surface texture: Profile method – Terms, definitions and surface texture parameters *Geneve: International Organization for Standardization* **1st edition** 1-25.

- Book: single author
  Mount GJ (1990) *An Atlas of Glass-ionomer Cements* Martin Duntz Ltd, London.

- Book: two authors
  Nakabayashi N & Pashley DH (1998) *Hybridization of Dental Hard Tissues* Quintessence Publishing, Tokyo.

- Book: chapter
  Hilton TJ (1996) Direct posterior composite restorations In: Schwarts RS, Summitt JB, Robbins JW (eds) *Fundamentals of Operative Dentistry* Quintessence, Chicago 207-228.

- Website: single author
  Carlson L (2003) Web site evolution; Retrieved online July 23, 2003 from: http://www.d.umn.edu/~lcarlson/cms/evolution.html

- Website: corporate publication
  National Association of Social Workers (2000) NASW Practice research survey 2000.

NASW Practice Research Network, 1. 3. Retrieved online September 8, 2003 from:http://www.socialworkers.org/naswprn/default

ANEXO B – PARECER DO COMITÊ DE ÉTICA E PESQUISA DA UFSM– ARTIGO 1.

UNIVERSIDADE FEDERAL DE SANTA MARIA/ PRÓ-REITORIA DE PÓS-GRADUAÇÃO E

PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Efeito de diferentes concentrações de ácido fluorídrico na resistência à fadiga de inlays de cerâmica feldspática

Pesquisador: MARÍLIA PIVETTA RIPPE

Área Temática:

Versão: 3

CAAE: 46057515.2.0000.5346

Instituição Proponente: Universidade Federal de Santa Maria/ Pró-Reitoria de Pós-Graduação e

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 1.178.683

Data da Relatoria: 11/08/2015

Apresentação do Projeto:

O projeto tem o título "Efeito de diferentes concentrações de ácido fluorídrico na resistência à fadiga de inlays de cerâmica feldspática" e está vinculado ao Programa de Mestrado em Ciências Odontológicas da UFSM.

Objetivo da Pesquisa:

Avaliar o efeito de diferentes concentrações de ácido fluorídrico na resistênda à fadiga entre dentina e inlay de cerâmica feldspática cimentadas em pré-molares.

Avaliação dos Riscos e Benefícios:

Riscos: sem riscos, pois os dentes serão desinfetados previamente ao início da pesquisa propriamente dita. Os dentes serão obtidos através da doação do Banco de Dentes Permanentes Humanos da UFSM.

Benefícios: poderá ser definida uma concentração de ácido mais adequada para o condicionamento da cerâmica feldspática que seja eficiente na resistência a fadiga de inlays e ao mesmo tempo seja menos tóxica ao cirurgião-dentista.

Os riscos e benefícios estão bem claros.

Comentários e Considerações sobre a Pesquisa:

.

Considerações sobre os Termos de apresentação obrigatória:

Foram apresentados o Termo de Confidencialidade a Autorização Institucional e a Declaração do Banco de Dentes.

Recomendações:

Veja no site do CEP - http://w3.ufsm.br/nucleodecomites/index.php/cep - na aba "orientações gerais", modelos e orientações para apresentação dos documentos. Acompanhe as orientações disponíveis, evite pendências e agilize a tramitação do seu projeto.

Conclusões ou Pendências e Lista de Inadequações:

.

Situação do Parecer:

Aprovado

Necessita Apreciação da CONEP:

Não

Considerações Finais a critério do CEP:

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Bairro: Camobi **CEP:** 97.105-970
UF: RS **Município:** SANTA MARIA
Telefone: (55)3220-9362 **E-mail:** cep.ufsm@gmail.com