UNIVERSIDADE FEDERAL DE SANTA MARIA
CENTRO DE CIÊNCIAS DA SAÚDE
PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS ODONTOLÓGICAS

Andressa Borin Venturini

EFEITO DA CONCENTRAÇÃO DE ÁCIDO FLUORÍDRICO NA CARGA PARA FALHA EM FADIGA DE UMA CERÂMICA FELDSPÁTICA CIMENTADA

Santa Maria, RS
2018

Andressa Borin Venturini

EFEITO DA CONCENTRAÇÃO DE ÁCIDO FLUORÍDRICO NA CARGA PARA FALHA EM FADIGA DE UMA CERÂMICA FELDSPÁTICA CIMENTADA

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

Orientador: Prof. Dr. Luiz Felipe Valandro

Santa Maria, RS
2018

Venturini, Andressa Borin
Efeito da concentração de ácido fluorídrico na carga para falha em fadiga de uma cerâmica feldspática cimentada / Andressa Borin Venturini.- 2018.
87 p.; 30 cm

Orientador: Luiz Felipe Valandro
Tese (doutorado) - Universidade Federal de Santa Maria, Centro de Ciências da Saúde, Programa de Pós Graduação em Ciências Odontológicas, RS, 2018

1. Ácido fluorídrico 2. Cerâmica feldspática 3. Carregamento cíclico 4. Fadiga I. Valandro, Luiz Felipe II. Título.

Sistema de geração automática de ficha catalográfica da UFSM. Dados fornecidos pelo autor(a). Sob supervisão da Direção da Divisão de Processos Técnicos da Biblioteca Central. Bibliotecária responsável Paula Schoenfeldt Patta CRB 10/1728.

Andressa Borin Venturini

EFEITO DA CONCENTRAÇÃO DE ÁCIDO FLUORÍDRICO NA CARGA PARA FALHA EM FADIGA DE UMA CERÂMICA FELDSPÁTICA CIMENTADA

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

Aprovado em 16 de março de 2018:

____

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

____

Marília Pivetta Rippe, Dra. (UFSM)

____

César Dalmolin Bergoli, Dr. (UFPEL)

____

Iana Lamadrid Aurélio, Dra. (UNIFACVEST)

____

Sara Fraga, Dra. (UNIFRA)

Santa Maria, RS
2018

AGRADECIMENTOS

À **Deus**, pela dádiva da vida, por tudo o que Ele me proporcionou e por todas as pessoas maravilhosas que colocou no meu caminho... por me dar forças para prosseguir a cada obstáculo encontrado, por ajudar a alcançar os meus objetivos e por permitir que eu chegasse até aqui.

Aos meus amados pais, **Gilberto Venturini e Teresinha Borin Venturini**, que são meus grandes exemplos, meu suporte e amor incondicional. O amor e os ensinamentos que vocês me deram por toda vida construíram as bases dos meus princípios e os alicerces da minha história. A vontade de orgulhar vocês me trouxe até aqui e me levará sempre além. Isso é o que me move. Muito obrigada por tudo... por não medirem esforços para me proporcionar sempre o melhor e por acreditarem em mim.

Ao meu querido irmão **André Borin Venturini**, sempre presente apesar da distância, pelo suporte, amizade e companhia durante toda minha vida. Obrigada por todo acolhimento na Holanda, por dividir seu estúdio comigo, por me ensinar a cultura e costumes desse país maravilhoso. Com certeza ter teu suporte nos primeiros dias em solo estrangeiro foi essencial.

Ao meu namorado **Rodrigo Dutra Segala**, pelo amor, incentivo e compreensão. Por me fortalecer e motivar a ir sempre em frente, por ser abrigo nos momentos difíceis e suporte nas quedas (literalmente). Obrigada por me tornar alguém melhor, por me fazer crescer e aceitar o desafio de viver em outro país ao meu lado, eu não teria conseguido sem teu apoio.

Aos **meus parentes próximos** e à **minha segunda família** que adquiri por meio do Rodrigo, agradeço o carinho, apoio e compreensão nos momentos em que não pude estar presente.

Ao meu orientador, **Prof. Dr. Luiz Felipe Valandro**, pelos anos de amizade, ensinamentos e orientação. Muito obrigada por todas as oportunidades e pela confiança depositada ao longo dos anos. É graças ao seu empenho, disponibilidade e dedicação que esta tese pode ser executada e que diversas oportunidades de crescimento pessoal e intelectual foram me proporcionadas.

Ao meu supervisor estrangeiro, **Prof. Dr. Cornelis Johannes Kleverlaan**, pela oportunidade, pelos ensinamentos concedidos e por toda disponibilidade apesar da agenda tão cheia de compromissos. Não tenho palavras para agradecer o convite feito ao Rodrigo para auxiliar na clínica do departamento e participar das pesquisas laboratoriais ao meu lado.

À **Universidade Federal de Santa Maria (UFSM)** e ao **Programa de Pós-Graduação em Ciências Odontológicas (PPGCO)** pela oportunidade, confiança e por todos os aprendizados adquiridos.

Ao **Academisch Centrum Tandhelkunde Amsterdam (ACTA)** pela oportunidade, receptividade e confiança em me receber para a execução do período de sanduíche (1 ano). Adicionalmente, ao **Departamento de Ciências dos Materiais Dentários (Dental Materials Science)** pela amizade, paciência e pelos momentos de confraternização desfrutados.

À **CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior)**, ao **CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico)** e ao **NUFFIC (Netherlands Organisation for International Cooperation in Higher Education)** pela concessão de suporte financeiro que viabilizaram a execução desta tese, trabalhos paralelos, além do período de doutorado sanduíche executado na ACTA.

À **FGM e Ivoclar**, pela doação dos materiais utilizados para realização deste trabalho.

Aos **professores do Curso de Odontologia e do Programa de Pós-Graduação em Ciências Odontológicas da Universidade Federal de Santa Maria**, por cada ensinamento e por alimentarem meu amor pela docência, em especial à **Prof. Dra. Liliana Gressler May**, pela contribuição intelectual e enriquecimento deste trabalho. Aos **professores membros da banca**, por disponibilizarem de seu tempo e conhecimento na avaliação deste trabalho. Aos meus **colegas professores da disciplina de prótese** pela recepção, ensinamentos e por tornarem o ambiente de trabalho agradável e de fácil convívio.

À **Catina Prochnow e Gabriel Pereira**, pela amizade, dedicação, disponibilidade, apoio e contribuição, que foram imprescindíveis para a realização desta tese. Tenho profunda admiração por vocês e sou eternamente grata por tudo. Estar ao lado de vocês é um privilégio!

À **Jéssica Dalcin da Silva e Simoni de Lima**, pela presteza, disponibilidade, cuidado e carinho de mãe com os alunos do PPGCO. Vocês nunca medem esforços para nos ajudar e são incansáveis em contribuir com o bom funcionamento do curso e do laboratório de biomateriais. Muito obrigada pela amizade e por todo o suporte.

A todos os **colegas ao longo da Pós-Graduação,** em especial **aos queridos da prótese** e **do laboratório de materiais dentários**, muito obrigada pelos momentos ímpares que passamos juntos, pela troca constante de conhecimentos e experiências, pelos momentos de confraternização, pela torcida e companheirismo. Desejo muito sucesso e conquistas a todos vocês.

Enfim, a todos àqueles que fizeram parte desta conquista, que me deram uma oportunidade e contribuíram para a minha formação. Muito obrigada!

"Somos todos anjos de uma asa só, e somente podemos voar quando abraçados uns aos outros."

Luciano de Crescenzo

RESUMO

EFEITO DA CONCENTRAÇÃO DE ÁCIDO FLUORÍDRICO NA CARGA PARA FALHA EM FADIGA DE UMA CERÂMICA FELDSPÁTICA CIMENTADA

AUTORA: Andressa Borin Venturini
ORIENTADOR: Luiz Felipe Valandro

A presente tese é composta por dois artigos científicos que avaliaram a influência de diferentes concentrações de ácido fluorídrico na carga para falha em fadiga de coroas e discos de cerâmica feldspática cimentados à um material análogo a dentina (resina epóxi). Para tanto, oitenta coroas com geometria simplificada e oitenta discos de cerâmica feldspática (Vita Mark II, Vita Zahnfabrik) foram divididos aleatoriamente, e a superfície interna de cada coroa/disco foi tratada de acordo com um dos quatro métodos de condicionamento de superfície (n=20): sem condicionamento/controle (CTRL), ou condicionamento por 60 s com diferentes concentrações de ácido fluorídrico: 1% (HF1), 5% (HF5) ou 10% (HF10). A superfície cerâmica tratada recebeu a aplicação de um agente de união silano. A superfície de cimentação dos preparos para coroas totais simplificadas e dos discos (ambos em resina epóxi) foram condicionados com ácido fluorídrico 10% por 60 s seguido da aplicação de uma camada de adesivo específico do sistema de cimentação. Após os tratamentos de superfície, as coroas e discos cerâmicos foram cimentados adesivamente aos respectivos preparos protéticos e discos de resina epóxi, os quais foram submetidos a cargas cíclicas em água pelo método da escada (500.000 ciclos, 20 Hz). Os dados de carga para falha em fadiga foram analisados pela análise de variância (ANOVA-1 fator) e testes de Tukey (α=0,05). Para as coroas cerâmicas, as cargas médias para falha em fadiga dos grupos CTRL (245,0 ± 15,1 N), HF1 (242,5 ± 24,7 N) e HF10 (255,7 ± 53,8 N) foram estatisticamente semelhantes (P>0,05), enquanto que a carga média do grupo HF5 (216,7 ± 22,5 N) foi significativamente inferior. Na condição de discos cimentados, a carga de falha média do grupo HF5 (255,0 ± 23,0 N) também foi significativamente inferior; o grupo HF1 (301,7 ± 71,0 N) apresentou valores intermediários e os valores mais altos foram alcançados nos grupos CTRL (351,7 ± 13,4 N) e HF10 (341,7 ± 20,6 N). O condicionamento com ácido fluorídrico 5% teve um efeito deletério nos valores de carga para falha em fadiga de coroas e discos de cerâmica feldspática cimentados adesivamente, visto que o condicionamento com ácido fluorídrico 10% não teve influência negativa em ambas as condições testadas (coroas e discos).

Palavras-chave: Ácido Fluorídrico. Carregamento Cíclico. Cerâmica Feldspática. Fadiga. Tratamento de superfície.

ABSTRACT

EFFECT OF HYDROFLUORIC ACID CONCENTRATION ON THE FATIGUE FAILURE LOAD OF A CEMENTED FELDSPATHIC CERAMIC

AUTHOR: Andressa Borin Venturini
ADVISOR: Luiz Felipe Valandro

The present thesis is composed by two scientific articles that evaluated the influence of different hydrofluoric acid concentrations on the fatigue failure loads of feldspathic ceramic crowns and discs adhesively cemented to a dentin analogue material (epoxy resin). Therefore, eighty crowns with simplified geometry and eighty feldspathic ceramic discs (Vita Mark II, Vita Zahnfabrik) were randomly allocated, and the intaglio surface of each crown/disc was treated with one of the four surface conditioning methods (n=20): nonetched / control (CTRL), or etched for 60 s with different hydrofluoric acid concentrations: 1% (HF1), 5% (HF5), or 10% (HF10). The treated ceramic surface received the application of a silane coupling agent. The cementation surface of simplified complete crown preparations and discs (both in epoxy resin) were etched with 10% hydrofluoric acid for 60 s and received a primer coating specific to the cementation system. After the surface treatments, the ceramic crowns and discs were adhesively cemented to the respective prosthetic preparations and epoxy resin discs, which were subjected to cyclic loads in water by a staircase approach (500,000 cycles, 20 Hz). Fatigue failure load data were analyzed by 1-way ANOVA and Tukey's tests (α= .05). For the ceramic crowns, mean failure loads of groups CTRL (245.0 ± 15.1 N), HF1 (242.5 ± 24.7 N) and HF10 (255.7 ± 53.8 N) were statistically similar (P>.05), while the mean load of the HF5 group (216.7 ± 22.5 N) was significantly lower. In the condition of cemented discs, mean failure load of the HF5 group (255.0 ± 23.0 N) was also significantly lower; HF1 group (301.7 ± 71.0 N) presented intermediate values, and the highest values were achieved in CTRL (351.7 ± 13.4 N) and HF10 (341.7 ± 20.6 N) groups. Etching with 5% hydrofluoric acid had a deleterious effect on the fatigue failure loads of adhesively cemented feldspathic ceramic crowns and discs, while etching with 10% hydrofluoric acid had no negative influence in both tested conditions (crowns and discs).

Keywords: Cyclic Loading. Fatigue. Feldspathic Ceramic. Hydrofluoric Acid. Surface treatment.

SUMÁRIO

1 INTRODUÇÃO

As cerâmicas têm ganhado papel de destaque na Odontologia Restauradora por satisfazerem a crescente exigência estética dos pacientes (KELLY, 2004), pelo desenvolvimento de materiais mais confiáveis e utilização de sistemas CAD/CAM (Computer Aided Design/Computer Aided Machine). A longevidade clínica das restaurações totalmente cerâmicas é dependente de uma combinação de fatores, tais como a durabilidade da união adesiva, a confiabilidade mecânica do material cerâmico (GUESS et al., 2009), e as cargas cíclicas às quais os materiais são submetidos quando em função (ZHANG et al., 2006).

Os blocos cerâmicos pré-fabricados para usinagem em sistemas CAD/CAM (*Computer Aided Design/Computer Aided Machine*) são confeccionados sob condições industriais padronizadas e reproduzíveis, consistindo em um material mais confiável estruturalmente e homogêneo devido a menores chances de incorporação de defeitos e poros (GIORDANO, 2006). Dentre os blocos cerâmicos disponíveis, Vitablocs Mark II (Vita Zahnfabrik, BadSäckingen, Alemanha) são produzidos com uma cerâmica de estrutura feldspática fina, e indicados para confecção de facetas, *inlays/onlays*, coroas anteriores e posteriores (VITA ZAHNFABRIK, 2012). Embora esse material apresente uma resistência flexural relativamente baixa de aproximadamente 86 MPa, possui elevado módulo de Weibull (m=23,6) (TINSCHERT et al., 2000). Clinicamente, coroas monolíticas de Vita Mark II apresentam taxas de sobrevivência semelhante às coroas com infraestrutura de Vita In Ceram Spinell em um período 2 a 5 anos (BINDL; MÖRMANN, 2004). Além disso, essas coroas (Vita Mark II) são significativamente reforçadas por procedimentos adesivos adequados (BINDL; LÜTHY; MÖRMANN, 2006).

Nesse sentido, a qualidade da união estabelecida entre cerâmica e cimento é um fator que pode afetar a resistência à fratura de restaurações cerâmicas (BINDL; LÜTHY, H.; MÖRMANN, 2006). Estudos que compararam cimentação adesiva e convencional demonstraram que coroas cimentadas adesivamente suportaram cargas mais altas para fratura (MÖRMANN et al., 1998; BINDL; LÜTHY; MÖRMANN, 2006). Em um estudo clínico de sobrevivência de coroas cerâmicas vítreas (Dicor), Malament e Socranski (2001) observaram que as coroas submetidas ao condicionamento ácido, previamente à cimentação, tiveram maior probabilidade de sobrevivência (75%) quando comparadas àquelas sem condicionamento (43%), em um período superior a 16 anos.

O processo adesivo das cerâmicas baseadas em sílica (vítreas) aos cimentos resinosos parece estar bem estabelecido, visto que a união é proporcionada pelo condicionamento com ácido fluorídrico (HORN, 1983) e potencializada pelo agente de união silano (BRENTEL et al., 2007). O condicionamento com ácido fluorídrico, realizado na superfície interna da peça cerâmica, ataca seletivamente a fase vítrea das cerâmicas, expondo óxidos de sílica (SiO_2), e produzindo alterações topográficas que contribuem para a retenção micromecânica a materiais resinosos (PHOENIX; SHEN, 1995; ROULET; DEGRANGE, 1996; THORDRUP et al., 1999). Esse condicionamento ácido modifica a superfície cerâmica em função da concentração de ácido fluorídrico utilizado e do tempo de condicionamento (ADDISON; MARQUIS; FLEMING, 2007b). Entretanto, grande parte das tensões de tração, responsáveis pelo início da falha em coroas cerâmicas, concentram-se na superfície de cimentação da peça cerâmica, na qual o procedimento de condicionamento ácido é realizado (KELLY et al., 1990; THOMPSON et al., 1994; QUINN et al., 2005; MAY et al., 2012).

Embora o condicionamento com ácido fluorídrico promova a rugosidade de superfície necessária para criar retenção micromecânica, estudos *in vitro* têm relatado que esse procedimento poderia enfraquecer o material cerâmico (ADDISON; FLEMING, 2004; ADDISON; MARQUIS; FLEMING, 2007b; HOOSHMAND; PARVIZI; KESHVAD, 2008; VENTURINI et al., 2015b). Em contrapartida, o condicionamento com ácido fluorídrico não afetou a resistência à fratura de cerâmicas vítreas quando um procedimento adesivo foi empregado após o condicionamento da superfície (YEN et al., 1993; PAGNIANO et al., 2005; POSRITONG, et al., 2013). Além disso, esse ácido também é conhecido como um produto químico com efeitos extremamente perigosos devido à sua toxicidade (OZCAN; ALLAHBEICKARAGHI; DUNDAR, 2012), o que tem motivado pesquisadores a testar concentrações mais baixas de ácido fluorídrico.

Os estudos laboratoriais (YEN et al., 1993; ADDISON; FLEMING, 2004; PAGNIANO et al., 2005; ADDISON; MARQUIS; FLEMING, 2007b; HOOSHMAND; PARVIZI; KESHVAD, 2008; POSRITONG, et al., 2013; VENTURINI et al., 2015b) que avaliaram o efeito de diferentes regimes de condicionamento (tempo e concentração de ácido fluorídrico) na resistência flexural de cerâmicas vítreas utilizaram testes monotônicos com espécimes na forma de barras ou discos. Testes monotônicos consistem na aplicação única de carga crescente até a fratura do corpo de prova, e são amplamente utilizados para a caracterização de materiais e avaliação da influência de variáveis nas propriedades mecânicas. Desta forma, os materiais não falham devido a um dano cumulativo (fadiga), como ocorre quando os materiais estão em função (SCHERRER et al., 2003). Em contrapartida, os ensaios de fadiga cíclica são testes complementares aos ensaios monotônicos tradicionais, pois reproduzem uma condição mais próxima ao cenário clínico (WISKOTT; NICHOLLS;

BELSER, 1995; MAY et al., 2015). Um dos ensaios baseados na aplicação de carga cíclica é o método da escada ("staircase method" ou "up and down method") que estabelece a resistência à fadiga do material (valor de tensão em que a falha ocorrerá após um tempo de vida específico). Nesse método, um número de ciclos é pré-determinado para aplicação de uma carga inicial de magnitude inferior à tensão máxima suportada pelo material e, dependendo da ocorrência ou não de fratura, a carga é aumentada ou diminuída, empregando-se um incremento fixo nas fases seguintes (COLLINS, 1993).

Portanto, os estudos laboratoriais citados anteriormente além de utilizarem apenas ensaios monotônicos, não levaram em consideração a influência da geometria da restauração na distribuição de tensões, nem o método de confecção dos corpos-de-prova (KELLY, 1999). Além disso, os procedimentos de cimentação e o suporte dentinário (módulo de elasticidade da dentina) não foram considerados na maioria desses estudos. Até o presente momento, não há estudos na literatura que tenham avaliado a influência de diferentes concentrações de ácido fluorídrico em espécimes de cerâmica feldspática cimentados adesivamente e submetidos a ensaios de fadiga cíclica, cujo contexto de teste apresenta maior relevância clínica.

Ainda no sentido da influência da geometria da restauração, alguns estudos (MAY et al., 2012; ANAMI et al., 2015) validaram ensaios com espécimes em forma de coroas simplificadas, que dispensam o uso de uma completa anatomização oclusal para avaliar desfechos clínicos. Em relação à facilidade metodológica, Chen et al. (2014) utilizaram uma metodologia de ensaio simplificada para simulação de restaurações em dentes posteriores, no qual discos cerâmicos são cimentados adesivamente a discos de um substrato análogo a dentina obtendo-se uma espessura final de 3,5 mm, equivalente à espessura média da parede pulpar à superfície oclusal. Entretanto, comparações sobre a eficácia de simulação desse método simplificado (disco sobre disco proposto por Chen) em relação ao uso de restaurações na forma de coroas cimentadas não foram investigadas.

A fim de prever de maneira mais confiável o comportamento mecânico da superfície cerâmica após os procedimentos de condicionamento com ácido fluorídrico e cimentação, espécimes na forma de coroas e discos de cerâmica feldspática foram submetidos a ensaios de fadiga cíclica em meio úmido. Por consequência, fatores como a distribuição de tensões, propagação de trincas, e a interação da população de defeitos da cerâmica com o cimento resinoso serão mais semelhantes à situação encontrada clinicamente. Portanto, o presente trabalho visa avaliar, através do método de "*staircase*", o impacto do condicionamento com ácido fluorídrico e suas diferentes concentrações na carga para falha em fadiga de coroas e de discos de cerâmica feldspática cimentados adesivamente a um material análogo a dentina.

2 ARTIGO 1 – FATIGUE FAILURE LOAD OF FELDSPATHIC CERAMIC CROWNS AFTER HYDROFLUORIC ACID ETCHING AT DIFFERENT CONCENTRATIONS

Publicado em “*The Journal of Prosthetic Dentistry*”, ISSN: 0022-3913, Fator de impacto = 2.095; Qualis A1. As normas para publicação estão descritas no Anexo A.

Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations

Andressa B. Venturini, DDS, MSD,[a] Catina Prochnow, DDS, MSD,[b] Liliana G. May, DDS, PhD,[c] Cornelis J. Kleverlaan, PhD,[d] and Luiz Felipe Valandro, DDS, PhD[e]

[a] Doctoral student, Graduate Program in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.
[b] Doctoral student, Graduate Program in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.
[c] Adjunct Professor, Graduate Program in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.
[d] Professor, Department of Dental Materials Science, Academic Centre for Dentistry Amsterdam (ACTA), University of Amsterdam and Vrije Universiteit Amsterdam, Amsterdam, The Netherlands.
[e] Associate Professor, Graduate Program in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.

Corresponding author:
Luiz Felipe Valandro, D.D.S, M.S.D., Ph.D., Associate Professor,
Federal University of Santa Maria
Faculty of Odontology
MSO-PhD Graduate Program in Oral Science
Prosthodontic Unit
Floriano Peixoto, 1184, 97015-372
Rio Grande do Sul State, Santa Maria, Brazil.
Phone: +55-55-3220-9276, Fax: +55-55-3220-9272
E-mail: lfvalandro@hotmail.com (Dr LF Valandro)

Authors'addresses:

Andressa BorinVenturini (andressa.venturini@hotmail.com)

Catina Prochnow (catinaprochnow@hotmail.com)

Liliana Gressler May (liligmay@gmail.com)

Cornelis Johannes Kleverlaan (c.kleverlaan@acta.nl)

Luiz Felipe Valandro (lfvalandro@gmail.com)

Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations

ABSTRACT

Statement of problem: Hydrofluoric acid etching modifies the cementation surface of ceramic restorations, which is the same surface where failure is initiated. Information regarding the influence of hydrofluoric acid etching on the cyclic loads to failure of ceramic crowns is lacking. **Purpose:** The purpose of this in vitro study was to evaluate the influence of different hydrofluoric acid concentrations on the fatigue failure loads of feldspathic ceramic crowns. **Materials and Methods:** Eighty feldspathic ceramic crowns were cemented with resin cement to identical simplified complete crown preparations machined in a dentin-like polymer. The preparations were etched with 10% hydrofluoric acid for 60 seconds and received a primer coating. Before cementation, the intaglio of the ceramic crowns was treated with 1 of 4 surface conditionings (n=20): nonconditioned (control, CTRL), or etched for 60 seconds with different hydrofluoric acid concentrations: 1% (HF1), 5% (HF5), and 10% (HF10). A silane coupling agent was applied on this surface of all crowns, which were cemented to the preparations. Each crown was cyclically loaded in water with a G10 epoxy-glass piston positioned in the center of the occlusal surface. Fatigue failure loads of ceramic crowns were obtained by the staircase approach after 500 000 cycles at 20 Hz. Mean failure loads were analyzed by 1-way ANOVA and the Tukey test (α=.05). **Results:** Mean failure loads of groups CTRL (245.0 ±15.1 N), HF1 (242.5 ±24.7 N), and HF10 (255.7 ±53.8 N) were statistically similar ($P>.05$), while that of the HF5 group (216.7 ±22.5 N) was significantly lower ($P<.05$). **Conclusion:** HF5 acid had a negative effect on the fatigue loads of the tested feldspathic ceramic crowns, while HF1 and HF10 acids did not change the fatigue resistance.

Clinical Implications

Hydrofluoric acid etching does not have a weakening effect on resin-bonded feldspathic ceramic crowns. However, the clinical use of 5% hydrofluoric acid for etching this ceramic should be considered carefully.

1. INTRODUCTION

The long-term success of ceramic restorations is determined by the durability of the adhesive bond and by the mechanical reliability of the ceramic material.[1] Ceramic restorations are constantly subjected to cyclic loads in wet conditions, and the combination of these factors has been identified as one of the main causes of crack initiation and subsequent growth, which may decrease the strength and increase the failure of the restorative materials.[2,3]

Vitablocs Mark II (Vita Zahnfabrik) are fine-structure feldspar ceramic blocks used to produce inlays, onlays, veneers, and crowns with computer-aided design and computer-aided manufacturing (CAD-CAM) systems. Industrially prepared ceramic blocks are more structurally reliable materials for dental applications, although the machining process may induce flaws.[4] Vita Mark II has a relatively low flexural strength of 86.3 MPa, but it has a high Weibull modulus (m=23.6).[4] Clinically, complete crowns of Vita Mark II showed similar survival rates to crowns with Vita In-Ceram Spinell copings over a period of 2 to 5 years.[5] Furthermore, appropriate bonding procedures can improve not only the bond strength but also the fracture strength of CAD-CAM crowns.[6]

During the bonding procedure, the intaglio surface of feldspathic ceramic restorations should be etched with hydrofluoric acid to provide the necessary surface alterations and surface roughness for mechanical interlocking.[7] These surfaces are further primed with a silane coupling agent for chemical bonding of the ceramic, silane, and resin cement.[7,8] The experimental evaluation of clinically failed complete ceramic crowns and finite element analysis results suggests that the majority of bulk fractures of single-unit crowns are initiated from the intaglio of the ceramic (cementation surface), where high tensile stresses develop during cyclic loading[2,9-13] and where hydrofluoric acid etching is performed. Therefore, the internal surface characteristics of ceramic crowns are believed to play a crucial role in their probability of failure.[14]

Although hydrofluoric acid etching modifies the intaglio surface of ceramic restorations to promote micromechanical interlocking, the impact of this acid on the ceramic strength remains uncertain. It appears to have a weakening effect on glass ceramics[15-18] because of the modification of the resident surface flaw population,[15] with a progressive increase in this effect as a function of the hydrofluoric acid concentration used for etching.[16,19] With higher hydrofluoric acid concentrations, the increased number of defects can be attributed to newly introduced flaws.[16] If these flaws reach a critical size, they can propagate on the internal surface and cause ceramic failure. Thus, the flaw distribution present in the material is directly related to the fracture strength of the ceramic.[20] However, some studies have reported that hydrofluoric acid in different etching regimens did not negatively impact the strength of ceramics[21,22] and that unfilled resin or resin cement application had a positive effect on flexural strength after hydrofluoric acid etching and silane treatment,[14,22] minimizing the influence of flaws.[23] The strengthening effect occurs as a consequence of the interaction of the resin with the entire surface defect population[24] and may depend on the behavior of the resin penetrating the ceramic surface.[25]

In addition, this acid is also known as a chemical with extremely hazardous effects because of its toxicity.[26] Therefore, potential damage to health has motivated dental research to test low hydrofluoric acid concentrations.[27]

The performance of complete ceramic crowns is determined by a complex combination of factors including the material selected, thickness, damage introduced, adhesive/luting system used, tooth substrate (natural dentin or foundation restoration[28]), and the fatigue response to complex loading.[29] In terms of failure prediction, cyclic mechanical loads applied under wet conditions could simulate the damage accumulation that occurs in ceramic restorations in an oral environment[30] and can lead to modes of crack initiation and growth not seen under monotonic loads.[31] However, the authors are unaware of studies that did not perform monotonic tests to evaluate the effect of hydrofluoric acid etching on the ceramic surface, which can be less clinically relevant than fatigue failure tests. Likewise, the authors are unaware of studies that tested the influence of the restoration geometry on stress distribution or the cementation effects, such as flaw "healing" by resin cement. In addition, the influence of dentin elastic properties and the manufacturing method of the ceramic specimens–related to population defects–need to be studied. Currently, studies that replicated clinical situations that have assessed the influence of hydrofluoric acid etching in different concentrations on the fatigue failure loads of adhesively cemented feldspathic ceramic crowns are lacking.

Therefore, the purpose of this in vitro study was to evaluate the influence of different hydrofluoric acid concentrations on the failure loads of feldspathic ceramic crowns machined by CAD-CAM systems using wet mechanical cyclic tests. Two hypotheses were tested: acid etching would not reduce the fatigue failure loads in comparison with untreated crowns, and mean failure loads would not be influenced by the different hydrofluoric acid concentrations.

2. MATERIAL AND METHODS

The design of a simplified complete crown was adapted from one previously developed by Gressler May et al.[30] They observed that fractures were initiated from the cementation surface of feldspathic crowns. Furthermore, the lack of contact damage on the loaded surface was attributed to the use of a flat-end piston made from G10 epoxy glass.

Eighty identical dentin analog prosthetic preparations (preparation height=5.32 mm, internal angle radii=0.5 mm, axial wall convergence=16 degrees, cervical preparation depth=1.2 mm, and round shoulder radii=0.5 mm) were machined from 11 mm diameter rods of an epoxy-glass cloth (NEMA G10; International Paper) in a mechanical lathe (Diplomat 3001; Nardini), as seen in Figure 1A. One G10 preparation was scanned, and the 3-dimensional images were processed in CAD software (CEREC in-Lab 3D, v4.1; Sirona Dental Systems GmbH) with an occlusal cementation space of 60 µm and an occlusal thickness of 1.5 mm.

A CAM machine (CEREC inLab MC XL; Dentsply Sirona) was used to mill ceramic blocks (10×12×15 mm) (Vita Mark II 4M2C/I12; Vita Zahnfabrik) into identical crowns using diamond rotary instruments and water. Four nominally identical pairs of diamond instruments, each containing 1 cylindrical (Cylinder pointed bur 12S; Dentsply Sirona) and 1 stepped pattern (Step bur 12S; Dentsply Sirona), were used to generate 80 crowns. Each rotary instrument set machined the sample size for each group (n=20). The specimen machining sequence (1 to 20) was recorded for each individual crown, and they were randomly assigned according to the same instrument set using an online tool (http://www.randomizer.org/). After machining, each crown was seated on its respective preparation to evaluate the marginal fit. However, no internal adjustment was needed in the crowns. Nevertheless, their occlusal surface was finished with silicon carbide grit (#600), resulting in a final occlusal thickness of 1.5 ±0.01 mm. The preparations were cleaned in an ultrasonic bath with distilled water for 3 minutes, and the crowns were cleaned with isopropyl alcohol for 5 minutes to remove the polishing residue.

The intaglio surfaces were etched with different hydrofluoric acid concentrations, as summarized in Table 1, for 60 seconds, rinsed with an air-water spray for 30 seconds, dried, and ultrasonically cleaned in distilled water for 5 minutes. Subsequently, the silane-based primer (Monobond Plus; Ivoclar Vivadent AG) was applied on the intaglio of all crowns for 1 minute and then air dried.

The G10 preparations were etched with 10% hydrofluoric acid for 60 seconds, washed for 30 seconds, and ultrasonically cleaned for 5 minutes. Multilink Primer A and B (Ivoclar Vivadent AG) were mixed in a 1:1 ratio, scrubbed on the dies for 30 seconds, and dried until a thin film was obtained.

One centimeter of resin cement (Multilink Automix; Ivoclar Vivadent AG) was measured with a ruler, mixed, and applied to the intaglio crown surface. The crowns were seated under a load of 7.5 N. The excess cement was removed, and the remaining cement was light polymerized with 5 exposures of 20 seconds each.

After the cementation procedures, all specimens, as seen in Figure 1B, were embedded in both polyurethane (F16, Fast Cast Polyurethane; Axson Technologies) and PVC cylinders to 2 mm below the cervical margin. They were then stored in distilled water at 37°C for 7 to 14 days before the staircase load testing was conducted.

Cyclic failure loads were established in an electric machine (Instron ElectroPuls E3000; Instron Corp) using the staircase sensitivity (up and down) approach method described by Collins.[32] Each crown was centrally and perpendicularly loaded under water using a 2-mm diameter piston made from G10 epoxy-glass cloth (Fig. 2). A sheet of polyethylene (0.1 mm thick) was placed between the piston and the ceramic to reduce contact stress concentration.

Sinusoidal cyclic loading was applied to ceramic specimens, with amplitudes ranging from a minimum of 10 N to the maximum tensile load, at a frequency of 20 Hz, and for 500 000 cycles. The initial load and the step size were determined based on the results of the monotonic tests from the 10% hydrofluoric acid (HF10) group (n=3; mean monotonic load for fracture=300 N). A load that was 60% of the mean monotonic failure load was assumed as the initial load (180 N). A step size of 20 N was applied up or down to the next specimen, according to the examination for subsurface crack formation by transillumination. If the tested specimen failed, the next specimen was cycled at a lower load by decreasing 1 step size. If the specimen survived the 500 000 cycles, the subsequent specimen was cycled at a higher load by increasing 1 step size.

The mean failure load (Lf) and the standard deviation (s) were calculated on the basis of the data of the least frequent event (survival or failure) by using the method described by Collins.[32]

$$L_f = L_{f0} + d\left[{\Sigma i n_i}/{\Sigma n_i} \pm {1}/{2}\right] \qquad \text{Eq. (1)}$$

$$s = 1{,}62d\left\{\left[\left(\Sigma n_i \Sigma i^2 n_i - ([\Sigma i n_i)]^2\right)/(\Sigma n_i)^2\right] + 0{,}029\right\} \qquad \text{Eq. (2)}$$

if: $\left[\left(\Sigma n_i \Sigma i^2 n_i - ([\Sigma i n_i)]^2\right)/(\Sigma n_i)^2\right] \geq 0.3$

where L_{f0} is the lowest load level considered in the analysis, d is the step size, and n_i is the number of failures or survivals at the given load level. In Eq. (2), the negative sign is used if the least frequent event is a failure; otherwise, the positive sign is used. The lowest load level considered is designated as i=0, the next level as i=1, and so on. n_i is the number of failures or survivals at a given load level.

Topographical analysis was performed by field emission scanning electron microscopy (FE-SEM) (FEI Inspect F50; FEI) at different magnifications. For this analysis, a machined crown was sectioned into 4 pieces. Each piece was treated using different conditioning methods, and the occlusal surfaces of cementation (internal surface) were sputter coated with a gold-palladium alloy before being examined.

After the fatigue test, the crowns were analyzed under a light microscope (Stereo Discovery V20; Carl Zeiss) to determine the region of crack origin. Then the failure crowns were longitudinally sectioned into halves perpendicular to the track of the radial crack. Representative specimens from each group were analyzed by FE-SEM.

Statistical analysis was performed using statistical software (IBM SPSS Statistics for Windows v21; IBM Corp). All load values (failure or survival steps) were analyzed by 1-way ANOVA and the post hoc Tukey test (α=.05) because the data presented homogeneity of variances (P>.05 based on the Levene test) and normal distribution (P>.05 based on the Shapiro-Wilk test).

3. RESULTS

Significant differences were found among the groups (ANOVA, P<.001). The crowns etched by 5% hydrofluoric acid (HF5) had a lower mean fatigue failure load than that of the CTRL, 1% hydrofluoric acid (HF1), and HF10 groups, which were statistically similar (Table 2). The patterns of runouts (survivals) and failures from the staircase experimental design for the different groups after 500 000 cycles are shown in Fig. 3.

The FE-SEM analysis revealed that slight topographical changes were promoted by HF1 compared with the untreated condition (CTRL) (Fig. 4). A progressive effect of the different hydrofluoric acid concentrations was observed on the ceramic microstructure, indicating that higher concentrations promoted larger and deeper craters and pits.

The crown failure analysis under a light microscope showed that all fatigue cracks were radial cracks starting from the cemented surface, and there was no Hertzian cone crack. Representative FE-SEM micrographs of the fracture surfaces are presented in Fig. 5. The irregularities created by acid etching on the ceramic surface were often not filled by resin cement, especially in the HF5 group.

4. DISCUSSION

The first hypothesis–that the acid etching would not reduce the fatigue failure loads in comparison with untreated crowns–was partially accepted. Mean fatigue failure loads were significantly lower when ceramic surfaces were etched with HF5, while the groups CTRL, HF1, and HF10 were not statistically different (Table 2). The second hypothesis was rejected because the fatigue failure loads were influenced by the distinct hydrofluoric acid concentrations.

Malament and Socransky[28] reported that the intraoral survival rates (Kaplan-Meier) of glass-ceramic crowns (Dicor) over 16 years were higher when they were acid etched before being bonded to the dentin cores, as opposed to not being acid etched. Of the luting agents tested in that study, acid-etched Dicor restorations luted with composite resin exhibited more favorable survivor functions than restorations luted with glass ionomer or zinc phosphate cement.[28] These data highlight the importance of resin-bonded adhesion and topographical changes promoted by hydrofluoric acid etching to achieve better clinical results.

Nevertheless, hydrofluoric acid can be harmful and particularly aggressive to soft tissues.[26] Considering the potential hazards of hydrofluoric acid in dental applications, low concentrations of this acid have been studied to promote durable bond strengths without weakening the ceramics. Venturini et al[27] tested 4 hydrofluoric acid concentrations for etching a feldspathic ceramic (Vita Mark II), concluding that 3%, 5%, and 10% hydrofluoric acid promoted stable resin adhesion after long-term aging, while 1% hydrofluoric acid showed a significant decrease in bond strength after aging/thermocycling. In another study,[18] they tested the effect of the same hydrofluoric acid concentrations on the flexural strength and reported that acid etching has a weakening effect on feldspathic ceramic when compared with untreated ceramic, regardless of its concentration. Regardless of these previous monotonic findings, the mean fatigue failure loads were not different among the groups CTRL, HF1, and HF10 in the current study. The explanation for this could be the cementation procedure, which provides support to the ceramic crown by a cement layer. A positive influence of the resin cement application on the ceramic flexural strength has been proposed by some authors.[22,24,25] The theory of resin strengthening ceramics may be explained by the combination of the Poisson constraint and the creation of a resin interpenetrating layer sensitive to the elastic modulus of the resin.[25] A resin layer that is bonded to the flawed surface would change the ceramic material to a ceramic-composite resin.[12]

Etching the cementation surface and bonding with a low-viscosity resin cement can minimize the influence of flaws at cementation surfaces.[23] However, crowns etched with 5% hydrofluoric acid were significantly less resistant than the other groups tested here. The FE-SEM fractography images (Fig. 5) show nonhomogeneous penetration of the resin cement into the ceramic irregularities created by 5% hydrofluoric acid through the cement voids present at the interface, mainly in the failure origin area. The presence of a large flaw and cement voids along the internal surface of a glass-ceramic crown may raise internal stresses, leading to failure.[12] Under loading, the stress at the flaw tip in an air space cannot be transferred to the resin, thereby increasing the susceptibility of the ceramic to crack initiation.[12] The resin cement viscosity is also important, as more fluid resin cement could penetrate irregularities on the intaglio ceramic surface.

Previous investigations of clinically failed glass-ceramic crowns revealed that the majority of failures were initiated from flaws and tensions existing at the cementation surface, indicating this surface as the location of the highest tensile stress and/or the largest flaws.[9,11,13] In the current study, all failures occurred as radial cracks from the cementation surface, which approximate to the clinical failure reports. In addition, the use of a flat-end piston made from G10 epoxy glass and a plastic strip between the piston and the crown during the test may justify the absence of contact damage on the loaded occlusal surface.

The load to initiate a radial fracture is influenced by the crown thickness and the relative elastic modulus between crown and the supporting tooth substrate.[29] Hence, the current study used a crown design with a simple geometric configuration because an irregular geometry makes it difficult to determine the effect of crown thickness. In addition, the substrate supporting material was an epoxy filled with woven glass fibers (G10), which has an elastic modulus similar to that of dentin. Kelly et al[10] reported that G10 was not significantly different from hydrated dentin in terms of blunt contact elastic behavior or resin cement bond strength. They found a small difference (approximately 5%) in fatigue behavior when testing was performed at 20 Hz versus 2 Hz and concluded that further tests can be performed at 20 Hz, greatly increasing the rate of data accumulation. Therefore, the current fatigue test was performed at 20 Hz.

Regarding the fatigue test, the staircase approach was used to determine the fatigue strength; it involved subjecting specimens to a high number of cycles at a low load. At least 15 specimens were necessary to evaluate the fatigue strength at the life of interest.[32] Coefficients of variation of approximately 10% were achieved by Kelly et al,[10] with a modest number of specimens (≤20) in a staircase sensitivity testing. Because of its low variability, a sample size of 20 crowns was used in the present study.

There are limitations in this in vitro study, and some clinical conditions were not simulated. The direction of the load application was only axial, without simulation of lateral forces and sliding that may occur clinically during mastication or clenching. Furthermore, the brief water storage times (7 to 14 days), without long-term aging, might be insufficient to damage bond strength. The chemical bond between silane and resin cement might deteriorate over time because of hydrolysis, and the micromechanical retention promoted by the minimal acid etching (HF1) or nonacid etching (CTRL) may be insufficient to provide stable bond strength. Therefore, the adhesion in CTRL and HF1 may have been promoted mainly by chemical bonds, with minimum micromechanical bonding. Thus, these findings should be considered carefully, and future studies should stress the clinical plausibility of using lower concentrations of hydrofluoric acid.

5. CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Etching with 5% hydrofluoric acid on the intaglio surface of feldspathic ceramic crowns reduced the fatigue failure loads and thus would not be recommended for the tested ceramic.

2. The mean fatigue failure loads of the feldspathic crowns was not influenced by 1% and 10% hydrofluoric acid, since these etched crowns did not differ from untreated crowns.

ACKNOWLEDGMENTS

CAPES (Brazilian Higher Education Agency, Brazil) Foundation supported this investigation (PhD scholarship). The authors thank FGM for providing the hydrofluoric acids, and Ivoclar Vivadent for donation the resin cement.

REFERENCES

1. Guess PC, Strub JR, Steinhart N, Wolkewitz M, Stappert CF. All-ceramic partial coverage restorations-midterm results of a 5-year prospective clinical splitmouth study. J Dent 2009;37:627-37.

2. Kelly JR. Clinically relevant approach to failure testing of all-ceramic restorations. J Prosthet Dent 1999;81:652-61.

3. Drummond JL, Lin L, Al-Turki LA, Hurley RK. Fatigue behaviour of dental composite materials. J Dent 2009;37:321-30.

4. Tinschert J, Zwez D, Marx R, Anusavice KJ. Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics. J Dent 2000;28:529-35.

5. Bindl A, Mörmann WH. Survival rate of mono-ceramic and ceramic-core CAD/CAM-generated anterior crowns over 2-5 years. Eur J Oral Sci 2004;112:197-204.

6. Mörmann WH, Bindl A, Lüthy H, Rathke A. Effects of preparation and luting system on all-ceramic computer-generated crowns. Int J Prosthodont 1998;11:333-9.

7. Brentel AS, Ozcan M, Valandro LF, Alarça LG, Amaral R, et al. Microtensile bond strength of a resin cement to feldpathic ceramic after different etching and silanization regimens in dry and aged conditions. Dent Mater 2007;23:1323-31.

8. Phoenix RD, Shen C. Characterization of treated porcelain surfaces via dynamic contact angle analysis. Int J Prosthodont 1995;8:187-94.

9. Kelly JR, Giordano R, Pober R, Cima MJ. Fracture surface analysis of dental ceramics: clinically failed restorations. Int J Prosthodont 1990;3:430-40.

10. Kelly JR, Rungruanganunt P, Hunter B, Vailati F. Development of clinically validated bulk failure test for ceramic crowns. J Prosthet Dent 2010;104:228-38.

11. Kelly JR, Campbell SD, Bowen HK. Fracture-surface analysis of dental ceramics. J Prosthet Dent 1989;62:536-41.

12. Anusavice KJ, Hojjatie B. Tensile stress in glass-ceramic crowns: effect of flaws and cementation voids. Int J Prosthodont 1992;5:351-8.

13. Thompson JY, Anusavice KJ, Naman A, Morris HF. Fracture surface characterization of clinically failed all-ceramic crowns. J Dent Res 1994;73:1824-32.

14. Pagniano RP, Seghi RR, Rosenstiel SF, Wang RT, Katsube N. The effect of a layer of resin luting agent on the biaxial flexure strength of two all-ceramic systems. J Prosthet Dent 2005;93:459-66.

15. Addison O, Fleming GJ. The influence of cement lute, thermocycling, and surface preparation on the strength of a porcelain laminate veneering material. Dent Mater 2004;20:286-92.

16. Addison O, Marquis PM, Fleming GJ. The impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. Dent Mater 2007;23:461-8.

17. Hooshmand T, Parvizi S, Keshvad A. Effect of surface acid etching on the biaxial flexural strength of two hot-pressed glass ceramics. J Prosthodont 2008;17:415-9.

18. Venturini AB, Prochnow C, May LG, Bottino MC, Valandro LF. Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic. J Mech Behav Biomed Mater 2015;48:241-8.

19. Della Bona A, Anusavice KJ, Hood JAA. Effect of ceramic surface treatment on tensile bond strength to resin cement. Int J Prosthodont 2002;15:248-53.

20. Thompson JY, Anusavice KJ. Effect of surface etching on the flexure strength and fracture toughness of Dicor disks containing controlled flaws. J Dent Res 1994;73:505-10.

21. Yen T, Blackman RB, Baez RJ. Effect of acid etching on the flexural strength of a feldspathic porcelain and castable glass ceramic. J Prosthet Dent 1993;70:224-33.

22. Posritong S, Borges AL, Chu TM, Eckert GJ, Bottino MA, Bottino MC. The impact of hydrofluoric acid etching followed by unfilled resin on the biaxial strength of a glass-ceramic. Dent Mater 2013;29:e281-90.

23. Kelly JR, Nishimura I, Campbell SD. Ceramics in dentistry: historical roots and current perspectives. J Prosthet Dent 1996;75:18-32.

24. Addison O, Marquis PM, Fleming GJ. Resin strengthening of dental ceramics-the impact of surface texture and silane. J Dent 2007;35:416-24.

25. Addison O, Marquis PM, Fleming GJ. Resin elasticity and the strengthening of all-ceramic restorations. J Dent Res 2007;86:519-23.

26. Özcan M, Allahbeickaraghi A, Dündar M. Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review. Clin Oral Investig 2012;16:15-23.

27. Venturini AB, Prochnow C, Rambo D, Gundel A, Valandro LF. Effect of hydrofluoric acid concentration on resin adhesion to a feldspathic ceramic. J Adhes Dent 2015;17:313-20.

28. Malament KA, Socransky SS. Survival of Dicor glass-ceramic dental restorations over 16 years. Part III: effect of luting agent and tooth or toothsubstitute core structure. J Prosthet Dent 2001;86:511-9.

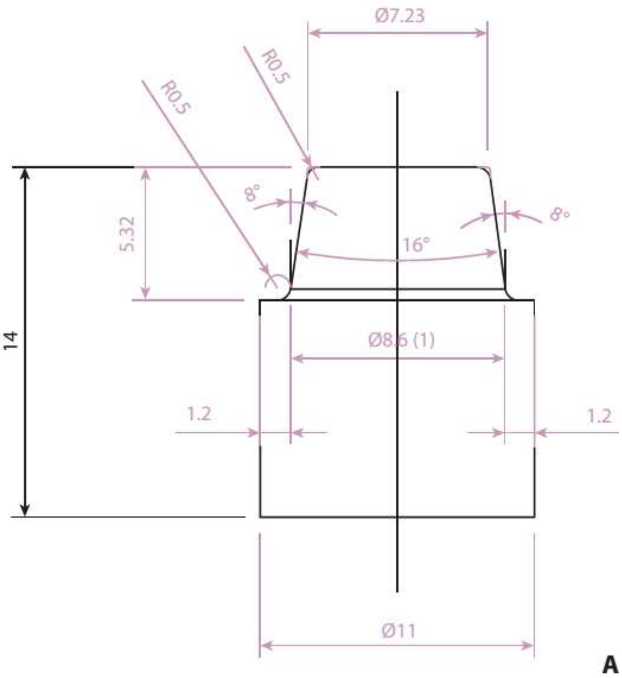
29. Thompson VP, Rekow DE. Dental ceramics and the molar crown testing ground. J Appl Oral Sci 2004;12:26-36.

30. Gressler May L, Kelly JR, Bottino MA, Hill T. Influence of the resin cement thickness on the fatigue failure loads of CAD/CAM feldspathic crowns. Dent Mater 2015;31:895-900.

31. Suresh S. Fatigue of materials. 2nd ed. Cambridge: Cambridge University Press; 1998.

32. Collins JA. Failure of materials in mechanical design. 2nd ed. New York: John Wiley & Sons; 1993. p. 383-90.

FIGURES



A

Figure 1. Illustrations of a G10 preparation for crown cementation. A, Sketch lines and dimensions (in mm) for the axial-symmetric drawing of the G10 preparation. B, Specimen after crown cementation.

A

B

Figure 2. Experimental arrangement. Specimen centrally loaded with G10 piston in fatigue machine.

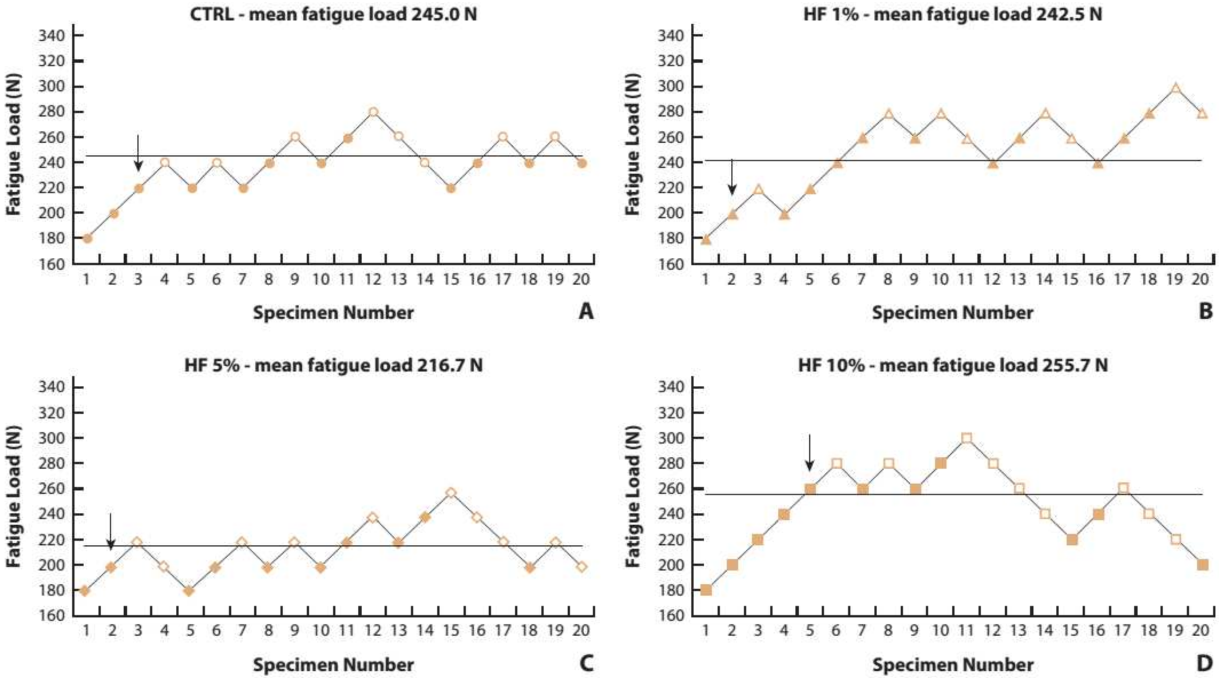


Figure 3. Pattern of runouts (survival) and failures for each group (CTRL, HF1, HF5, HF10) observed during wet mechanical cycling (500 000 cycles). Arrows indicate load level at which up-and-down character started. Horizontal lines indicate mean fatigue load, filled scorers show runout, and empty scorers are failure.

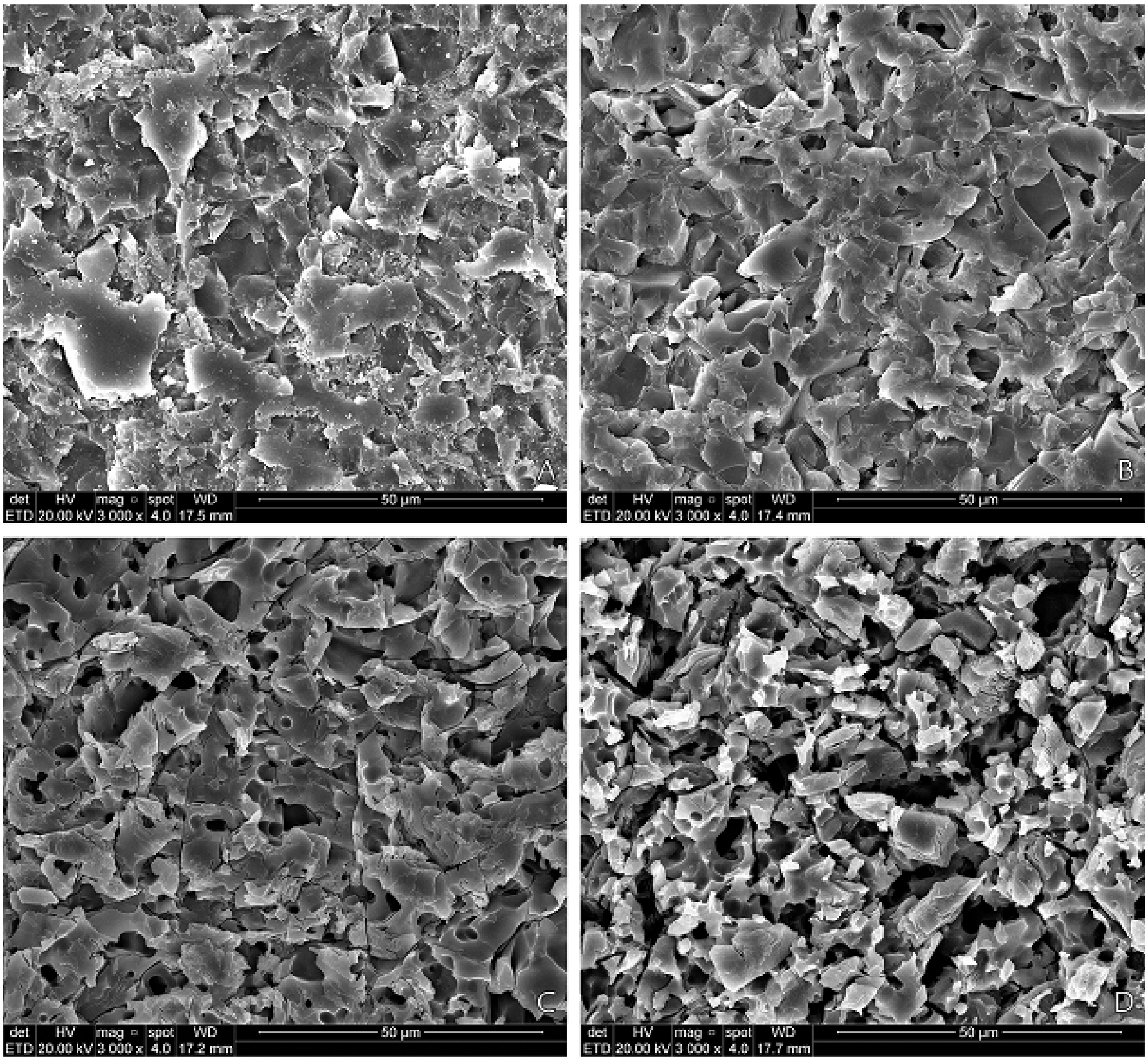


Figure 4. Representative field emission scanning electron microscopy micrographs (original magnification, ×3000) of different ceramic surface treatments showing topography pattern alteration generated by machining procedure and hydrofluoric acid etching. A, CTRL group. B, HF1 group. C, HF5 group. D, HF10 group.

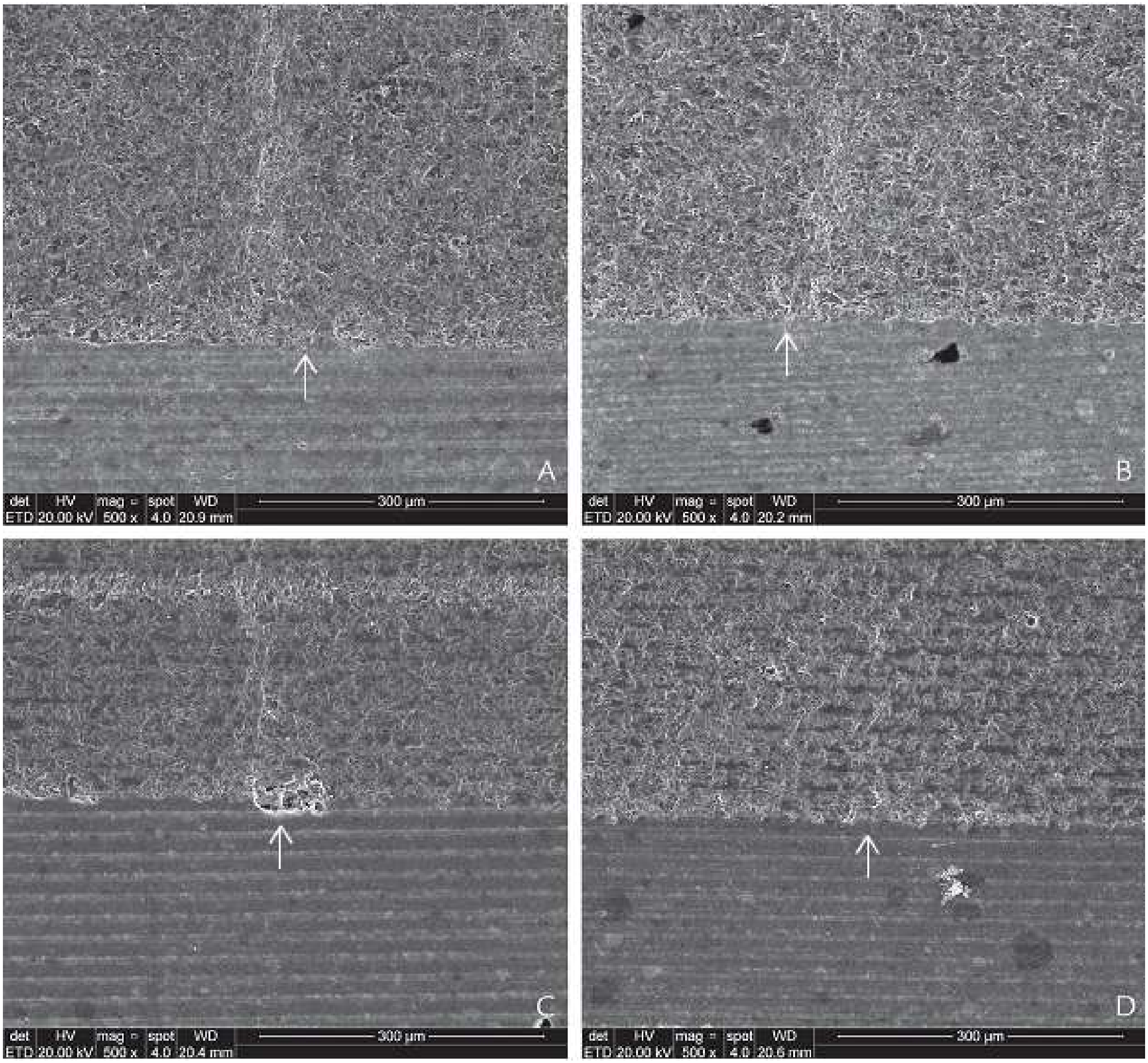


Figure 5. Representative images (original magnification, ×500) of fracture surfaces from crowns subjected to fatigue. A, CTRL group. B, HF1 group. C, HF5 group. D, HF10 group. Radial cracks pointing to fracture origin can be observed in cementation surface under tensile stresses**.**

TABLES

Table 1. Experimental design.

Group	Surface Treatment
CTRL	Unconditioned control, only silane
HF1	Etching with gel 1% hydrofluoric acid[a]
HF5	Etching with gel 5% hydrofluoric acid[b]
HF10	Etching with gel 10% hydrofluoric acid[b]

[a]Experimentally formulated (FGM). [b]Condac Porcelana 5% and 10% (FGM).

Table 2. Mean fatigue failure loads (L_f) and standard deviation (SD) of CAD-CAM feldspathic crowns from staircase tests.

Group	L_f (N) (± SD)
CTRL	245.0 (±15.1)^{A}
HF1	242.5 (±24.7)^{A}
HF5	216.7 (±22.5)^{B}
HF10	255.7 (±53.8)^{A}

CAD-CAM, computer-aided design and computer-aided manufacturing. Different superscript letters indicate statistically significant differences ($P<.05$).

3 ARTIGO 2 – THE EFFECT OF HYDROFLUORIC ACID CONCENTRATION ON THE FATIGUE FAILURE LOAD OF ADHESIVELY CEMENTED FELDSPATHIC CERAMIC DISCS

Publicado em "*Dental Materials*", ISSN: 0109-564, Fator de impacto = 4.070; Qualis A1. As normas para publicação estão descritas no Anexo B.

The effect of hydrofluoric acid concentration on the fatigue failure load of adhesively cemented feldspathic ceramic discs

Andressa B. Venturini[a], Catina Prochnow[a], Gabriel K. R. Pereira[a,b], Arie Werner[c], Cornelis J. Kleverlaan[c], Luiz Felipe Valandro[a*]

[a] Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Rio Grande do Sul, Brazil

[b] School of Dentistry, Meridional Faculty – IMED, Passo Fundo, Rio Grande do Sul, Brazil

[c] Department of Dental Materials Science, Academic Centre for Dentistry Amsterdam (ACTA), Universiteit van Amsterdam and Vrije Universiteit, Amsterdam, The Netherlands

* Corresponding author at: Federal University of Santa Maria, Prosthodontics Unit, Floriano Peixoto, 1184, 97015-372 Santa Maria, Brazil.

E-mail addresses: andressa.venturini@hotmail.com (A.B. Venturini), catinaprochnow@hotmail.com (C. Prochnow), gabrielkrpereira@hotmail.com (G.K.R. Pereira), a.werner@acta.nl (A. Werner), c.kleverlaan@acta.nl (C.J. Kleverlaan), lfvalandro@hotmail.com (L.F. Valandro).

The effect of hydrofluoric acid concentration on the fatigue failure load of adhesively cemented feldspathic ceramic discs

ABSTRACT

Objectives: This study investigated the influence of hydrofluoric acid (HF) etching at different concentrations on the fatigue failure load of adhesively cemented feldspathic ceramic discs (Vita Mark II). Besides, their effect on the micromorphology of ceramic surface was investigated.

Methods: Eighty ceramic discs (ϕ= 10 mm; thickness= 1.5 mm) were cemented to epoxy supporting discs (ϕ= 10 mm; thickness= 2.0 mm) using different surface conditioning methods (n=20): nonetched control (CTRL), or etched for 60 s with different HF concentrations: 1% (HF1), 5% (HF5), or 10% (HF10). All the ceramic discs received a silane application (Monobond Plus). The epoxy discs were etched with 10% HF for 60 s and received a primer coating (Multilink Primer A+B). Adhesively cementation was performed (Multilink Automix), and the assemblies (ceramic discs/epoxy discs) were subjected to cyclic loads in water by a staircase approach (500,000 cycles; 20 Hz; initial load= 290 N; step size= 30 N). Fatigue failure load data were analyzed using 1-way ANOVA and *post-hoc* Tukey's tests (α=.05).

Results: Mean failure load of the HF5 group (255.0 ± 23.0 N) was significantly lower; HF1 group (301.7 ± 71.0 N) presented intermediate values, and the highest values were achieved in CTRL (351.7 ± 13.4 N) and HF10 (341.7 ± 20.6 N) groups. All the failures were radial cracks starting from the bonding surface**.**

Significance: In terms of fatigue failure load, etching with 1% and 5% HF had a deleterious effect on the fatigue behavior of an adhesively cemented feldspathic ceramic, while 10% HF had no negative influence.

KEYWORDS: Acid etching. Glass ceramics. Fatigue. Surface treatment. Mechanical cycling.

HIGHLIGHTS

- Hydrofluoric acid (HF) concentration on the fatigue failure load was investigated.
- 5% and 1% HF etching for 60 seconds have demonstrated a deleterious effect.
- 10% HF had no negative influence and seems the recommended protocol for etching.

1. Introduction

Among a wide range of CAD/CAM blocks available to produce monolithic restorations, the feldspathic ceramic Vitablocs Mark II (Vita Zanhfabrik, Bad Säckingen, Germany) is fabricated using fine-structure ceramic powders under industrial sintering process, resulting in good polishing properties, decreased enamel wear, and increased strength due to a nearly pore free ceramic [1]. Also, the fracture resistance of the feldspathic ceramic restorations may be improved with appropriate bonding procedures [2]. To achieve successful bonding, the intaglio surface of these ceramics requires micro-mechanical interlocking by hydrofluoric acid etching, and chemical bonding by a silane coupling agent [3,4].

Even though that adhesive approach is a well-known and recommended method to increase bond strength to feldspathic ceramics, the high toxicity of hydrofluoric acid remains a concern among the clinicians and scientific literature [5] motivating the investigation of lower concentrations of hydrofluoric acid and alternative methods [6-9]. Besides, previous studies showed that mechanical strength of glass-ceramic materials may be negatively influenced by the hydrofluoric acid etching approaches [10-14]. The alterations of the surface flaws (defects' population) as consequence of the etching time and hydrofluoric acid concentration [11] could be the predictive factor of ceramic bulk fractures, mainly if the surface defects are not entirely filled by resin cement [15]. Thereby, only the flexural strength of the ceramic etched by hydrofluoric acid may not exactly reflect the actual strength of all-ceramic restoration [16] since it will be cemented to the tooth. The resin cement could improve the probability of survival of ceramic restorations to a certain extent by healing the cracks [17,18], which depends on the behavior of the resin penetrating the ceramic surface [19].

From the clinical perspective, ceramic restorations are constantly subjected to cyclic loading under wet conditions during chewing function, which should be considered during in vitro tests, since these conditions decrease the fracture load of restorative materials by slow-crack growth of intrinsic defects [20,21]. Studies about clinically failed glass-ceramic crowns reported that the majority of bulk fractures started from flaws and stresses at the cementation surface [22-24]. From that standpoint, it is important to investigate the influence of the etching protocols in a bonded ceramic surface in attempt to predict the clinical behavior using cyclic loading fatigue tests. Furthermore, limited information is available as to how the use of different hydrofluoric acid concentrations influences the fatigue failure load of adhesively cemented feldspathic ceramic.

Therefore, the current study aimed to evaluate the effect of different hydrofluoric acid concentrations on the fatigue failure loads of feldspathic ceramic discs adhesively bonded. Additionally, micro-morphological evaluations of the ceramic surface in response to the etching protocol and fractographic examinations of the failed specimens were executed. The hypotheses tested were: (1) acid etching would influence the fatigue failure loads compared with nonetched ceramic discs, and (2) different hydrofluoric acid concentrations would promote different fatigue failure loads.

2. Materials and methods

The materials used in this investigation are described in Table 1.

2.1 Specimens preparation and cementation procedure

Feldspathic ceramic blocks (VitaBlocks Mark II for CEREC/inLab, Vita Zahnfabrik, Bad Säckingen, Germany) were shaped into cylinders using a diamond drill (ϕ= 10 mm; Diamant Boart, Brussels, Belgium) coupled to a bench drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) under refrigeration. The cylinders were cut under water-cooling (Isomet 1000, Buehler, Lake Bluff, United States), resulting in 80 discs with an initial thickness of 1.6 mm. The ‘occlusal’ surface of the discs was polished with 600-grit SiC polish papers (Ecomet

Polisher, CarbiMet SiC Abrasive Paper grit 600–P1200, Buehler) until the thickness of 1.5 ± 0.1 mm to remove the irregularities of the surface in contact with the piston. The cementation surface was kept 'as-cutting'. Then, the ceramic samples were cleaned (isopropyl alcohol; 5 min) in an ultrasonic bath (Vitasonic, Vita Zahnfabrik).

The epoxy resin discs were shaped into cylinders using a cylindrical diamond drill with an internal diameter of 10 mm in the same way as aforementioned for the ceramic blocks, from plates with 2.0 mm thickness (Epoxy Plate 150 × 350 × 2.0 mm; Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany). A simplified bi-layer setup [25] was designed simulating the restoration of a posterior tooth. On this setup, the ceramic disc represents an occlusal restoration with 10 mm diameter, which is the average surface area of molars according Ferrario et al. [26]; and the epoxy resin disc simulates dentin (ϕ= 10 mm; thickness= 2.0 mm). The bonded two-layer discs had a final thickness of 3.5 mm, which is equivalent to the average thickness from upper pulp wall of the tooth to occlusal surface of the restoration [27-29].

Prior the cementation procedures, the discs were randomly assigned into four groups (n=20) according to the surface conditioning method: nonetched control (CTRL); etched with gel 1%, 5% and 10% hydrofluoric acid (HF1, HF5, and HF10, respectively). The bonding surface of each ceramic disc was etched with one of the different hydrofluoric acid concentrations (1%, 5%, or 10%; FGM, Joinville, Brazil) for 60 s, rinsed with an air-water spray for 30 s, dried, and ultrasonically cleaned in distilled water for 5 min, while the discs of the control group remained nonetched. Subsequently, the silane-based primer (Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) was applied on the bonding surfaces of all ceramic discs scrubbing it on the surfaces for 15 s and then kept to react for 45 s (completing 60 s for the entire procedure), followed by 15 s of air-drying.

The epoxy resin surface was etched with 10% hydrofluoric acid for 60 s, washed for 30 s, and ultrasonically cleaned for 5 min. Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, scrubbed on the epoxy surface for 30 s, and dried until a thin film was obtained. After, the ceramic discs were adhesively cemented to the corresponding epoxy discs with resin cement (Multilink Automix, Ivoclar Vivadent). The bonded two-layer discs were seated under a load of 2.5 N (to generate a similar cement thickness), followed by removal of excess cement, and light-activation (Radii-cal, SDI, Bayswater, Australia) for five exposures of 20 s each (one in each direction – 4 positions 0◦, 90◦, 180◦, 270◦, and top). All the specimens were stored in distilled water (7 up to 14 days; 37 ◦C) until the staircase load tests were conducted.

2.2 Fatigue failure load tests

The fatigue tests were conducted in an electric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, United States) with a piston made from epoxy filled with woven glass fibers (ϕ= 2 mm; NEMA, grade G10; International Paper, Hampton, USA), which was positioned in the center of the ceramic surface (ceramic up, dentin analogue material down). A sheet of polyethylene (0.1 mm thick) was placed between the piston and the ceramic surface to reduce contact stress concentration [30].

Cyclic failure loads (500,000 cycles at 20 Hz) were applied by the staircase approach [31], with amplitudes ranging from a minimum of 10 N to the maximum load to failure. The initial load and the step size were determined based on the results of the monotonic tests from the 10% hydrofluoric acid (HF10) group (n = 3; mean monotonic load for fracture = 483 N). A load of ~60% of the mean monotonic failure load was assumed as the initial load (290 N). A step size of 30 N (~10% of the initial load) was added or subtracted for the next specimen, according to the examination of subsurface crack formation by transillumination. If the tested specimen failed, the next specimen was cycled at a lower load by decreasing one step size. If the specimen survived to the 500,000 cycles, the subsequent specimen was cycled at a higher load by increasing one step size. According to Collins [31]., it is considered that the test only starts after the first inversion of outcome (survival/fracture); and also that, at least 15 samples should be tested to achieve an accurate measurement after this inversion.

The mean fatigue failure load (L_f) and the standard deviation (s) were calculated based on the data of the least frequent event (survival or failure), using the method described by Collins [31]:

$$L_1 f = L_1 f0 + d[\, [\![\sum in]\!]_{\downarrow} i \,/\, [\![\sum n]\!]_{\downarrow} i \pm 1/2 \,]$$

Eq. (1)

$$s = 1{,}62d \left\{ \left[\left(\sum n_i \sum i^2 n_i - ([\![\sum i n_i)]\!]^2 \right) \Big/ \left(\sum n_i \right)^2 \right] + 0{,}029 \right\}$$ Eq. (2)

If: $\left[\left(\sum n_i \sum i^2 n_i - ([\![\sum i n_i)]\!]^2 \right) \Big/ \left(\sum n_i \right)^2 \right] \geq 0.3$

$$s = 0.53d, if: \left[\left(\sum n_i \sum i^2 n_i - ([\![\sum i n_i)]\!]^2 \right) \Big/ \left(\sum n_i \right)^2 \right] < 0.3$$

where L_{f0} is the lowest load level considered in the analysis, d is the step size and n_i is the number of failures or survivals at the given load level. In Eq. (1), the negative sign is used if the least frequent event is a failure; otherwise the positive sign is used. The lowest load level considered is designated as i = 0, the next level as i = 1, etc. n_i is the number of failures or survivals at a given load level.

2.3 Fractographic analysis

After the fatigue tests, all fractured specimens were analyzed under a light microscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany) to determine the region of crack origin. Then, the failed discs were longitudinally sectioned into two halves, perpendicularly to the direction of the radial crack. Representative specimens from each group were analyzed under scanning electron microscopy (SEM; XL 20, FEI Company, GG Eindhoven, The Netherlands).

2.4 Topographic analysis

Topographical analyses were performed by SEM (EVO LS15, Zeiss, Oberkochen, Germany) at different magnifications. The specimens were treated using different conditioning methods, and then sputter coated with a gold-palladium alloy before being examined. Besides, additional specimens were subjected to SEM analysis (Vega3, Tescan, Czech Republic) to verify the bonding interface and the cement penetration into ceramic irregularities created by hydrofluoric acid etching.

2.5 Data analysis

According to Collins [31], only the data from the least frequent event values (failure or survival steps) observed on the fatigue tests should be considered. So, on this sense, the statistical analysis was performed (IBM SPSS Statistics v21 for Windows; IBM Corp) using One-way ANOVA and the post hoc Tukey's test (α =.05).

3. Results

Significant differences for fatigue failure load were found among the evaluated groups (1-way ANOVA, $P<.001$). On this sense, discs etched by 5% hydrofluoric acid (HF5) presented the lowest mean value in comparison to the CTRL, HF1, and HF10 groups (Table 2). The pattern of runouts (survivals) and failures from the staircase fatigue tests for each group are described in Fig. 1.

The fractographic analysis under a light microscope showed that all failures were radial cracks starting from the cemented surface at the center region, and Hertzian cone cracks were not observed. Representative SEM micrographs of the fracture surfaces are presented in Fig. 2, and also corroborate those assumptions (observed on light microscope).

The SEM analysis revealed a progressive effect of hydrofluoric acid concentration on the ceramic microstructure, indicating that higher concentrations promoted larger and deeper craters and pits (Fig. 3). Fig. 4 shows that the defects (apparently sharper) promoted by low hydrofluoric acid concentrations (1% and 5%) were not entirely filled by the resin cement, while the resin cement penetrated into the irregularities (apparently larger and rounded) introduced by 10% hydrofluoric acid.

4. Discussion

This in vitro study showed a significant effect of hydrofluoric acid concentration on the fatigue failure load of feldspathic ceramic specimens. The first hypothesis that acid etching would influence the fatigue failure loads compared with nonetched ceramic discs was partially accepted, since HF10 and CTRL groups presented statistically similar fatigue failure load values (Table 2). However, a decrease in the fatigue failure load values was observed when ceramic surfaces were etched with 5% and 1% hydrofluoric acid (HF5 < HF1 < HF10). Thus, these findings corroborate the second hypothesis, i.e., different hydrofluoric acid concentrations would promote different fatigue failure loads.

Literature supports that hydrofluoric acid etching mechanism is based on a chemical modification of the ceramic surface, selectively removing the glass matrix, increasing the bonding area (i.e., roughness), and the reactivity to the luting agent (i.e., surface free energy) [32,33]. After etching appropriately the cementation surface of glass ceramics (i.e., producing surface alterations for micromechanical interlocking), and adhesively bonding it to the tooth substrate using low-viscosity resin cements; it may be minimized the influence of critical flaws at this surface [34]. The ability of the resin cement to interpenetrate the ceramic surface defects introduced by etching creates a ceramic-resin composite hybrid layer, which is responsible for the ceramic strengthening effect [19]. Thus, the increase of the fracture resistance of glass ceramics will be in response of an enhanced stress distribution through this assembly, which decreases the risk of stress concentration on the aforementioned flaws [16,18,35].

In the current investigation, the topographic alterations promoted by 10% hydrofluoric acid resulted in no weakening effect in comparison to the nonetched condition (CTRL), while a negative impact on the fatigue failure load was observed for 1% and 5% concentrations. These findings may be explained by the size and shape of the defects introduced by the different hydrofluoric acid concentrations. In the SEM images, it is possible to observe a progressive effect in response to the increase on acid concentrations (larger and deeper craters) on the ceramic microstructure, and a lesser amount of glassy matrix in all etched surfaces compared with nonetched one. Hence, the largest irregularities, promoted by 10% hydrofluoric acid, enabled an adequate penetration of the resin cement creating a ceramic-resin composite layer, which resulted in a higher fatigue failure load in comparison to the other hydrofluoric acid concentrations (Fig. 4). In regards to the low hydrofluoric acid concentrations (1% and 5%), they probably introduced sharper defects that were not entirely filled by the resin cement, so in this scenario it hypothesizes an ineffective stress distribution (higher stress concentration around the existing defects and leading to premature fracture during smaller loads).

Another important factor to be considered is that hydrofluoric acid etching induces an increased wettability on the adherent (resin cement), in response to an increase in surface area [32]. Increased wettability is associated with a lower contact angle and greater bonding potential among substrates [36]. Venturini et al., [6] tested the same acids used in this study with the same ceramic (Vita Mark II) and observed that hydrofluoric acid etching decreased the contact angle in all etched groups (1, 5 and 10%), on which the lowest mean contact angle value was achieved by 10% hydrofluoric acid. These previous findings also corroborate the higher filling potential of the cement observed for 10% hydrofluoric acid etching in the current results (Fig. 4).

As aforementioned, acid etching can reduce the stress concentration at existing flaws tips by changing their shape [37]. However, ceramic discs etched with 5% hydrofluoric acid were significantly less resistant than all the other tested groups. This current finding agrees with Venturini et al. [15], who evaluated the influence of hydrofluoric acid concentration on the fatigue failure load of feldspathic ceramic crowns machined by a CAD/CAM system. They stated that HF5 acid had a negative effect on the fatigue loads of Vita Mark II ceramic crowns. In the FE-SEM fractographic images, they observed cement voids at the interface related to the failure origin area. On this scenario, a fraction of the stress at the flaw tip cannot be properly transferred to the resin in these areas with nonhomogeneous penetration of the resin cement (air space), increasing the susceptibility of the ceramic to crack initiation [37]. Thus, our results support the clinical implication proposed by this previous study: the clinical use of 5% hydrofluoric acid (gel) for etching Vita Mark II ceramic should be considered carefully [15].

Also, Venturini et al., [15] noticed that HF1 produced similar values to HF10 and CTRL groups, which is not corroborated by the present findings (HF1 < HF10 and CTRL). Those differences for fatigue behavior of HF1 groups might have been caused by the topographical differences of the ceramic surface from the sample production: they produced the ceramic crowns from the CAD/CAM milling, which leads to a rougher topography in comparison to the methodology of the present study – in vitro cutting processing using a cutting machine. Although the costs of research are reduced with this simplified disc methodology, the mechanical properties might be misinterpreted since machining of partially or fully sintered CAD/CAM ceramics is deleterious to flexural fatigue strength [38]. The topography achieved by CAD/CAM milling is also different from the one achieved by in vitro laboratorial processing, and that fact could influence on the final mechanical property of the ceramic, so this topographical differences should be considered [39]. It seems that this phenomenon could also be an explanation to the differences between our data and the ones observed previously by Venturini et al., [15]. From the clinical relevance standpoint, studies about the effect of the etching protocols and bonding should consider the simulation of the surface topography promoted by the milling by CAD/CAM system; otherwise an overestimation may be expected if the ceramic surface is not milled by the CAD-CAM system.

Regarding the used materials and test setup, the epoxy supporting material simulates adequately human hydrated dentin in terms of resin cement bond strength and blunt contact elastic behavior as it presents a similar elastic modulus (18 MPa) [40,41]. It is important to highlight that our study used a simplified bi-layer assembly to simulate a monolithic restoration of a posterior first molar (as described by Chen et al., [25]). The main advantage of this assembly is that by controlling the direction of the load application and creating a standardized and fully reproducible scenario, we enable the evaluation of a factor lonely (the effect of etching protocols). This assumption is corroborated by our fractography analysis, which all fractures were radial cracks starting from the cemented surface at the center region, and there was no Hertzian cone crack. Moreover, the use of a personalized piston made from epoxy glass material and a plastic strip may justify the absence of contact damage on the loaded surface [40]. Despite that, this fatigue experiment consists in a laboratorial in vitro study, which cannot fully represent all intraoral conditions, thus containing inherent limitations. Some factors including the direction of the load application such as lateral forces and sliding, anatomical shapes, and hydrolysis of the chemical adhered interface can influence the long-term fracture strength of a ceramic restoration on the clinical intraoral environment. Thus, in vivo researches, to this aim, should be executed in the future.

In conclusion, taking into account the findings depicted by this current fatigue study and the previous adhesion outcomes [15], hydrofluoric acid at 10% concentration remains as the best surface treatment for feldspathic ceramics. Etching with 5% and 1% hydrofluoric acid had a negative effect on the fatigue failure load of adhesively cemented feldspathic discs.

ACKNOWLEDMENTS

CAPES (Brazilian Higher Education Agency, Brazil) Foundation supported this investigation (PhD scholarship). The authors thank FGM for providing the hydrofluoric acids, and Ivoclar Vivadent for donation the resin cement.

REFERENCES

[1] Giordano R. Materials for chairside CAD/CAM-produced restorations. J Am Dent Assoc 2006;137:S14–21.

[2] Mörmann WH, Bindl A, Lüthy H, Rathke A. Effects of preparation and luting system on all-ceramic computer-generated crowns. Int J Prosthodont 1998;11:333–9.

[3] Brentel AS, Ozcan M, Valandro LF, Alarc ¸a LG, Amaral R, et al. Microtensile bond strength of a resin cement to feldpathic ceramic after different etching and silanization regimens in dry and aged conditions. Dent Mater 2007;23:1323–31.

[4] Chen H, Matsumura H, Atsuta M. Effect of etching period and silane priming on bond strength to porcelain of composite resin. Oper Dent 1998;23:250–7.

[5] Özcan M, Allahbeickaraghi A, Dündar M. Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review. Clin Oral Investig 2012;16:15–23.

[6] Venturini AB, Prochnow C, Rambo D, Gundel A, Valandro LF. Effect of hydrofluoric acid concentration on resin adhesion to a feldspathic ceramic. J Adhes Dent 2015;17:313–20.

[7] Sundfeld Neto D, Naves LZ, Costa AR, Correr AB, Consani S, Borges GA, et al. The effect of hydrofluoric acid concentration on the bond strength and morphology of the surface and interface of glass ceramics to a resin cement. Oper Dent 2015;40:470–9.

[8] Román-Rodríguez JL, Perez-Barquero JA, Gonzalez-Angulo E, Fons-Font A, Bustos-Salvador JL. Bonding to silicate ceramics: conventional technique compared with a simplified technique. J Clin Exp Dent 2017;9:e384–6.

[9] El-Damanhoury HM, Gaintantzopoulou MD. Self-etching ceramic primer versus hydrofluoric acid etching: etching efficacy and bonding performance. J Prosthodont Res 2017;62:75–83.

[10] Addison O, Fleming GJ. The influence of cement lute, thermocycling, and surface preparation on the strength of a porcelain laminate veneering material. Dent Mater 2004;20:286–92.

[11] Addison O, Marquis PM, Fleming GJ. The impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. Dent Mater 2007;23:461–8.

[12] Hooshmand T, Parvizi S, Keshvad A. Effect of surface acid etching on the biaxial flexural strength of two hot-pressed glass ceramics. J Prosthodont 2008;17:415–9.

[13] Zogheib LV, Bona AD, Kimpara ET, McCabe JF. Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. Braz Dent J 2011;22:45–50.

[14] Venturini AB, Prochnow C, May LG, Bottino MC, Valandro LF. Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic. J Mech Behav Biomed Mater 2015;48:241–8.

[15] Venturini AB, Prochnow C, May LG, Kleverlaan CJ, Valandro LF. Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations. J Prosthet Dent 2017, http://dx.doi.org/10.1016/j.prosdent.2017.03.021. Epub ahead of print.

[16] Xiaoping L, Dongfeng R, Silikas N. Effect of etching time and resin bond on the flexural strength of IPS e max Press glass ceramic. Dent Mater 2014;30:e330–6.

[17] Marquis PM. The influence of cements on the mechanical performance of dental ceramics. Bioceramics 1992;5:317–24.

[18] Posritong S, Borges AL, Chu TM, Eckert GJ, Bottino MA, Bottino MC. The impact of hydrofluoric acid etching followed by unfilled resin on the biaxial strength of a glass-ceramic. Dent Mater 2013;29:e281–90.

[19] Addison O, Marquis PM, Fleming GJ. Resin elasticity and the strengthening of all-ceramic restorations. J Dent Res 2007;86:519–23.

[20] Kelly JR. Clinically relevant approach to failure testing of all-ceramic restorations. J Prosthet Dent 1999;81:652–61.

[21] Lohbauer U, Krämer N, Petschelt A, Frankenberger R. Correlation of in vitro fatigue data and in vivo clinical performance of a glassceramic material. Dent Mater 2008;24:39–44.

[22] Thompson JY, Anusavice KJ, Naman A, Morris HF. Fracture surface characterization of clinically failed all-ceramic crowns. J Dent Res 1994;73:1824–32.

[23] Kelly JR, Campbell SD, Bowen HK. Fracture-surface analysis of dental ceramics. J Prosthet Dent 1989;62:536–41.

[24] Kelly JR, Giordano R, Pober R, Cima MJ. Fracture surface analysis of dental ceramics; clinically failed restorations. Int J Prosthodont 1990;3:430–40.

[25] Chen C, Trindade FZ, de Jager N, Kleverlaan CJ, Feilzer AJ. The fracture resistance of a CAD/CAM Resin Nano Ceramic (RNC) and a CAD ceramic at different thicknesses. Dent Mater 2014;30:954–62.

[26] Ferrario VF, Sforza C, Tartaglia GM, Colombo A, Serrao G. Size and shape of the human first permanent molar: a Fourier analysis of the occlusal and equatorial outlines. Am J Phys Anthropol 1999;108:281–94.

[27] Harris EF, Hicks JD. A radiographic assessment of enamel thickness in human maxillary incisors. Arch Oral Biol 1998;43:825–31.

[28] Sulieman M, Addy M, Rees JS. Surface and intra-pulpal temperature rises during tooth bleaching: an in vitro study. Br Dent J 2005;199:37–40.

[29] Afonso Camargo SE, Valera MC, Ribeiro Camargo CH, Gasparoto Mancini MN, Menezes MM. Penetration of 38% hydrogen peroxide into the pulp chamber in bovine and human teeth submitted to office bleach technique. J Endod 2007;33:1074–7.

[30] International Organization for Standardization. Dental ceramics. 2nd ed. Switzerland: Geneva; 1995. ISO 6872:1995(E).

[31] Collins JA. Failure of materials in mechanical design. 2nd ed. New York: John Wiley & Sons; 1993. p. 383–90.

[32] Phoenix S, Shen C. Characterization of treated porcelain surfaces via dynamic contact angle analysis. Int J Prosthodont 1995;8:187–94.

[33] Jardel V, Degrange M, Picard B, Derrien G. Surface energy of etched ceramic. Int J Prosthodont 1999;12:415–8.

[34] Kelly JR, Nishimura I, Campbell SD. Ceramics in dentistry: historical roots and current perspectives. J Prosthet Dent 1996;75:18–32.

[35] May LG, Kelly JR, Bottino MA, Hill T. Effects of cement thickness and bonding on the failure loads of CAD/CAM ceramic crowns: multi-physics FEA modeling and monotonic testing. Dent Mater 2012;28:e99–109.

[36] Amaral R, Özcan M, Bottino MA, Valandro LF. Resin bonding to a feldspar ceramic after different ceramic surface conditioning methods: evaluation of contact angle, surface pH, and microtensile bond strength durability. J Adhes Dent 2011;13:551–60.

[37] Anusavice KJ, Hojjatie B. Tensile stress in glass-ceramic crowns: effect of flaws and cementation voids. Int J Prosthodont 1992;5:351–8.

[38] Fraga S, Amaral M, Bottino MA, Valandro LF, Kleverlaan CJ, May LG. Impact of machining on the flexural fatigue strength of glass and polycrystalline CAD/CAM ceramics. Dent Mater 2017;33:1286–97.

[39] Zucuni CP, Guilardi LF, Fraga S, May LG, Pereira GKR, Valandro LF. CAD/CAM machining vs pre-sintering in-lab fabrication techniques of Y-TZP ceramic specimens: effects on their mechanical fatigue behavior. J Mech Behav Biomed Mater 2017;71:201–8.

[40] Kelly JR, Rungruanganunt P, Hunter B, Vailati F. Development of clinically validated bulk failure test for ceramic crowns. J Prosthet Dent 2010;104:228–38.

[41] Wang Y, Katsube N, Seghi RR, Rokhlin SI. Statistical failure analysis of adhesive resin cement bonded dental ceramics. Eng Fract Mech 2007;74:1838–56.

FIGURES

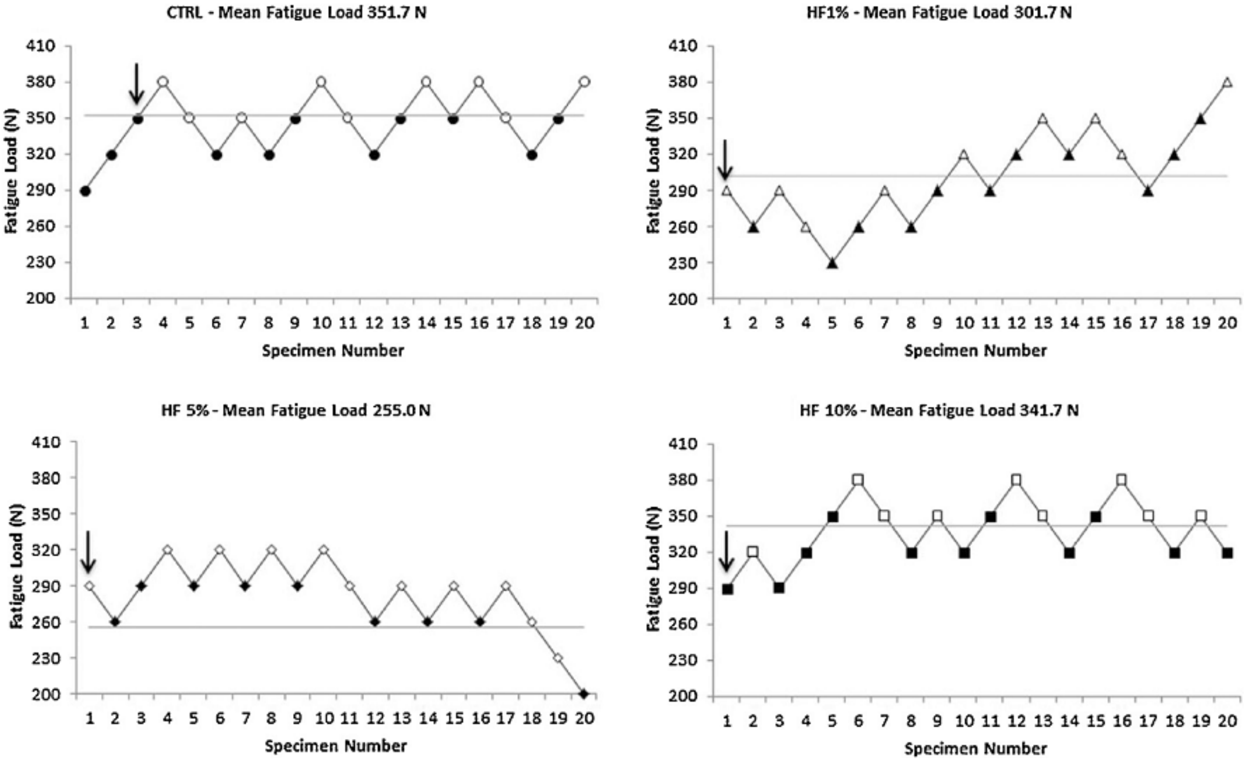


Fig. 1 - Pattern of runouts (survival) and failures for each group (CTRL, HF1, HF5, HF10) observed during wet mechanical cycling (500,000 cycles at 20 Hz). Arrows indicate the load level at which up-and-down characters started. Horizontal lines indicate mean fatigue failure load, filled scorers show runout, and empty scorers are failure.

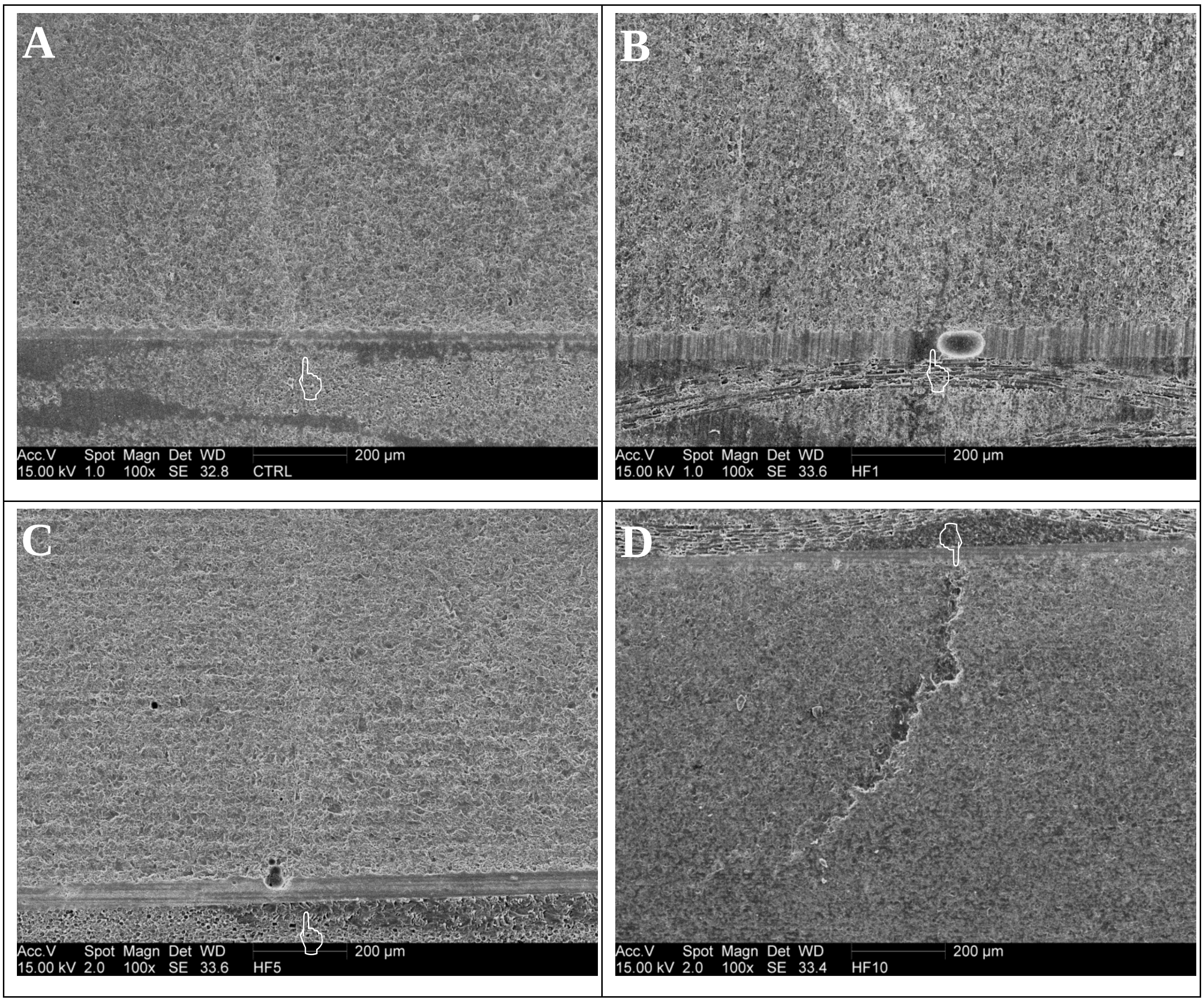


Fig. 2 - Scanning electron microscopy images (original magnification: ×100) of fracture surfaces from discs subjected to fatigue. A, CTRL group. B, HF1 group. C, HF5 group. D, HF10 group. Radial cracks pointing to fracture origin can be observed in cementation surface under tensile stresses.

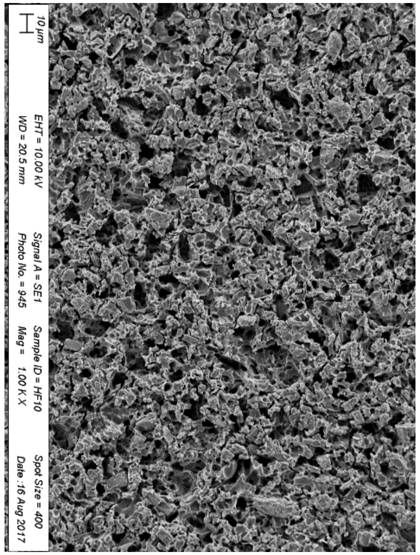
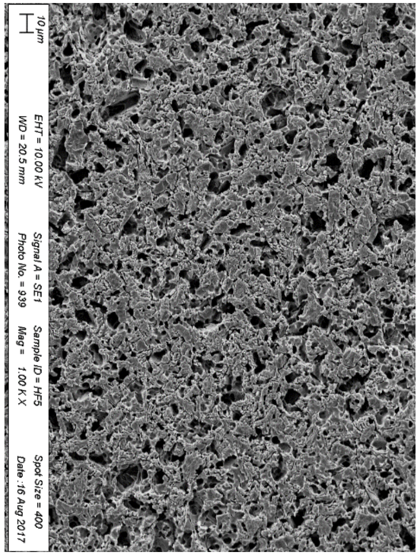
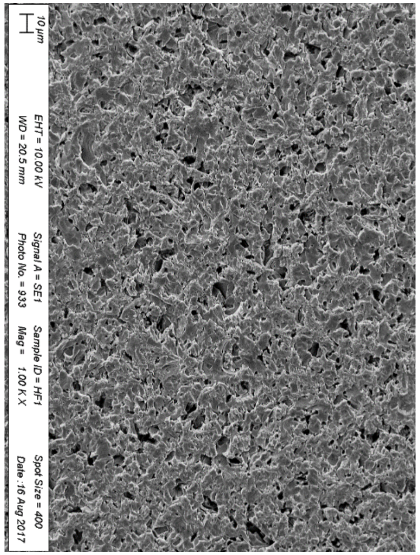
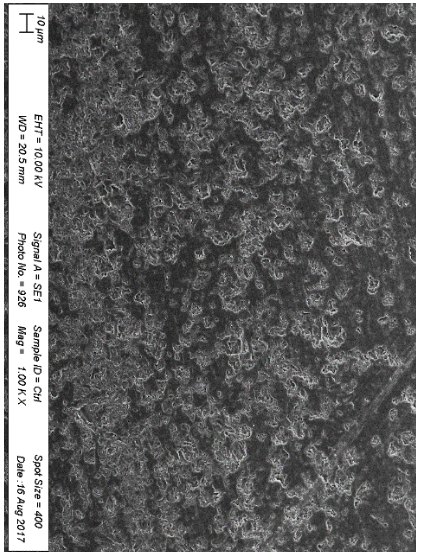
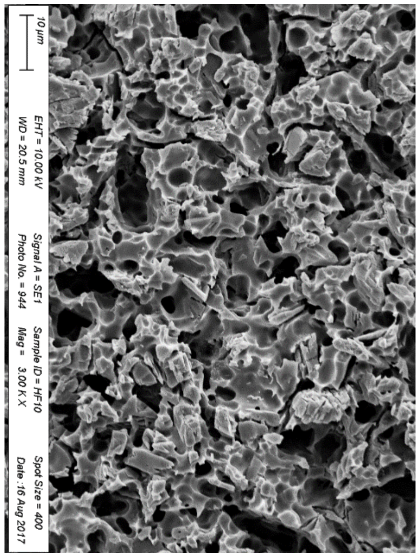
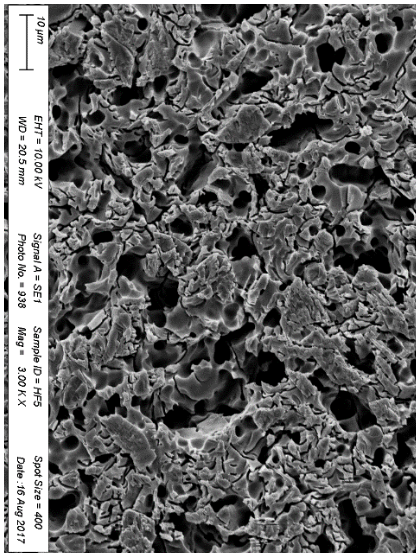
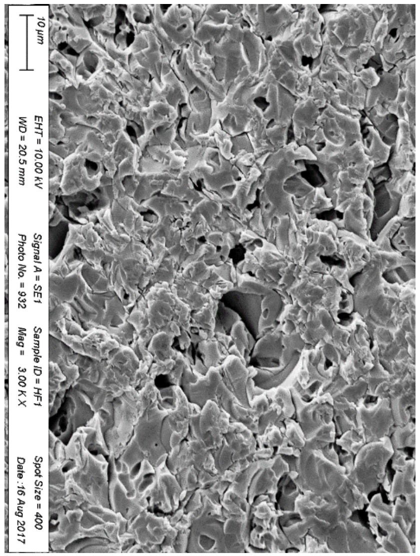
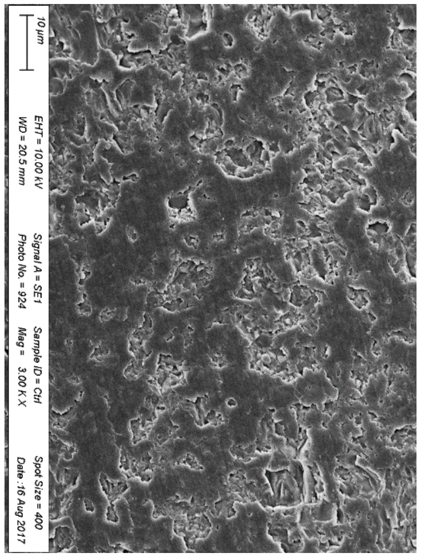
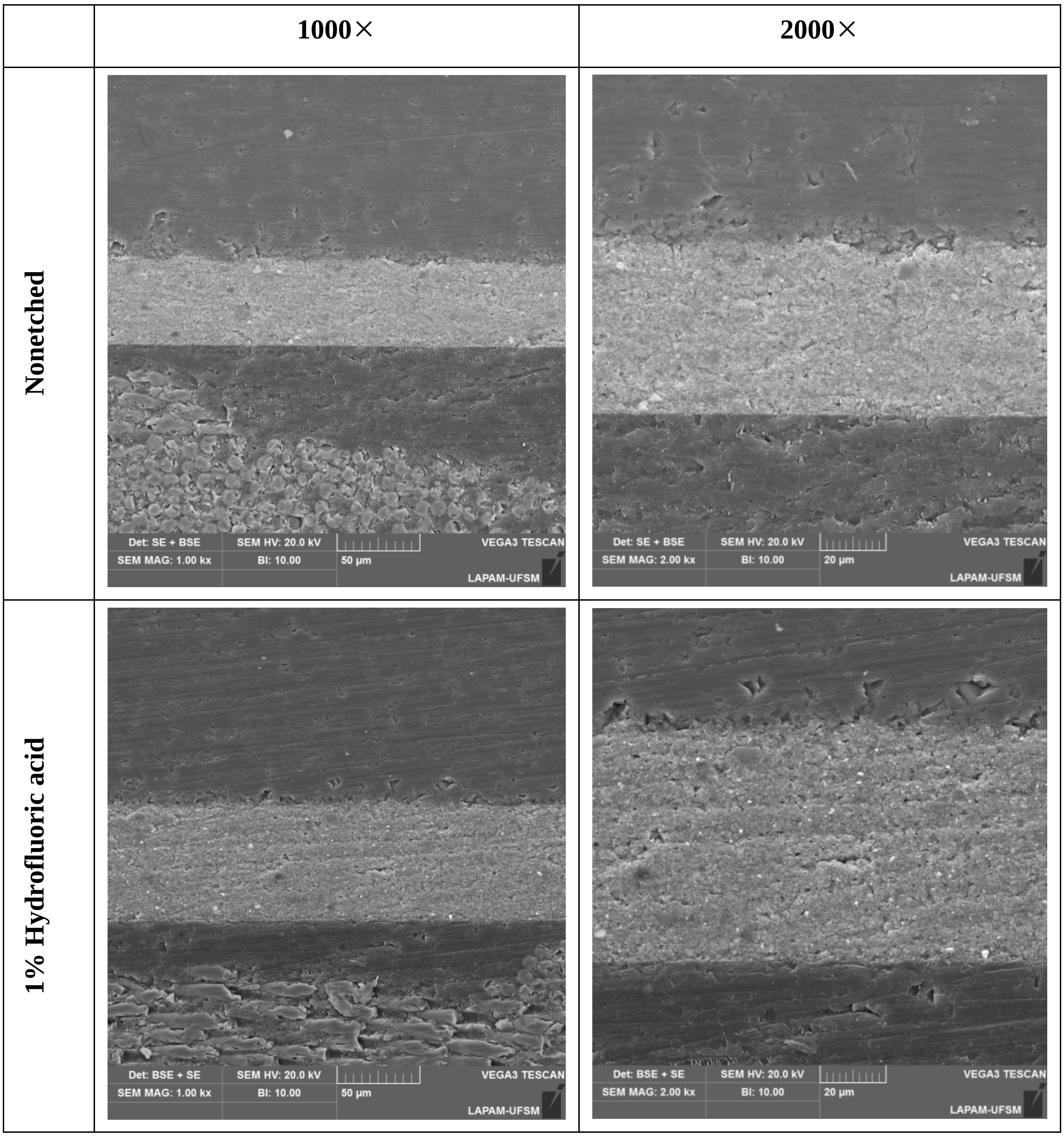
<table>
<tr><th>10% Hydrofluoric acid</th><th>5% Hydrofluoric acid</th><th>1% Hydrofluoric acid</th><th>Nonetched</th><th></th></tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1000×</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3000×</td>
</tr>
</table>

Fig. 3 - Representative SEM micrographs (original magnification, ×1000 and ×3000) of different ceramic surface treatments showing topography pattern alteration generated due to dissolution of the glassy matrix by hydrofluoric acid etching.



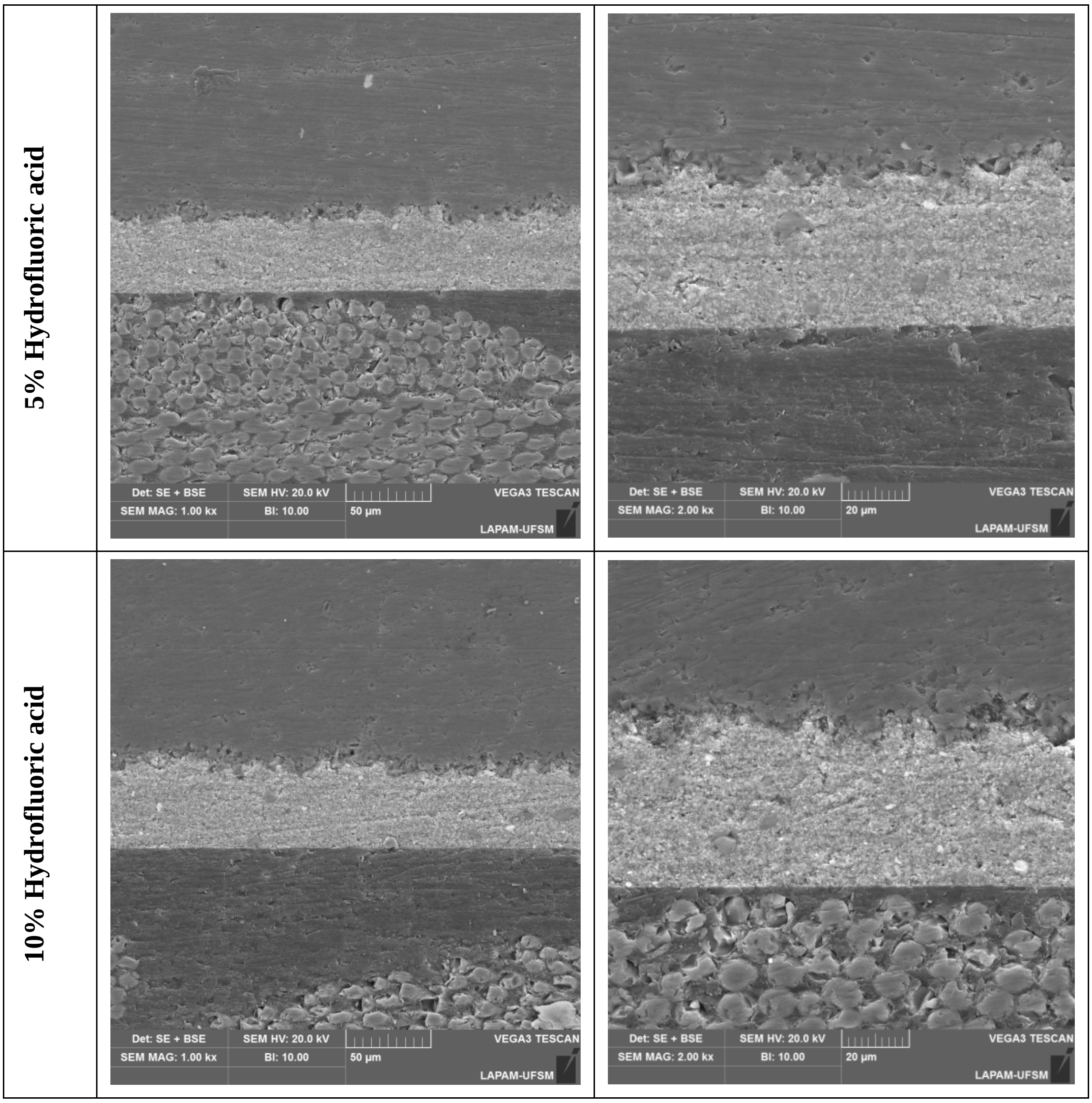


Fig. 4 - SEM images (original magnification, ×1000 and ×2000) of bonding interfaces from tested groups showing cement penetration into ceramic irregularities.

TABLES

Table 1 - Materials used in the study with manufacturer data and composition.

Material	Brand name; Manufacturer (Lot Number)	Composition[a]
Fine-particle feldspar ceramic	VITABLOCS Mark II; Vita Zahnfabrik (LOT 28650)	Al_2O_3, SiO_2, K_2O, Na_2O, CaO, TiO_2, other oxides
Epoxy resin plate	Epoxy platte 150x350x2,0 mm; Carbotec GmbH & Co. KG, Germany	Epoxy resin
Dual-cured resin cement	Multilink Automix; Ivoclar Vivadent AG, Liechtenstein (LOT U19980)	Dimethacrylates, HEMA, barium glass filler, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments
Primer	Multilink Primer A and Primer B; Ivoclar Vivadent AG, Liechtenstein (LOT U22990)	Primer A: water, initiators; Primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabiliser
Silane	Monobond Plus; Ivoclar Vivadent AG, Liechtenstein (LOT U25466)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate
Hydrofluoric acid	Experimentally formulated 1% Condac Porcelana 5% Condac Porcelana 10% FGM, Brazil (LOT 030216)	Hydrofluoric acid at 1%, 5%, and 10%, water, thickener, surfactant, and coloring

[a]The chemical compositions are described according to the manufacturers' information.

Table 2 - Mean fatigue failure loads (L_f) and standard deviation (SD) of tested groups from staircase tests.

Group	L_f (N) (± SD)
CTRL	351.7 (± 13.4)[A]
HF1	301.7 (± 71.0)[B]
HF5	255.0 (± 23.0)[C]
HF10	341.7 (± 20.6)[A]

Different letters indicate statistically significant differences (P<.05).

4 DISCUSSÃO

A união entre a estrutura dental e as restaurações cerâmicas indiretas é um fator imprescindível para o sucesso e longevidade das restaurações cerâmicas (MALAMENT; SOCRANSKY; 2001). Embora o condicionamento com ácido fluorídrico seja necessário para promover o embricamento micro mecânico com os materiais resinosos e a estrutura dental, pouco se sabe sobre os efeitos que as irregularidades criadas por esse ácido podem provocar nas propriedades mecânicas das cerâmicas submetidas a cargas cíclicas em ambiente oral úmido. O condicionamento ácido deveria, ao mesmo tempo, criar uma topografia de superfície suficientemente irregular para uma união adequada com o cimento resinoso sem causar o enfraquecimento da estrutura cerâmica. Neste sentido, o uso de concentrações mais baixas de ácido fluorídrico tem sido considerado juntamente ao fato desse ácido ser perigoso e particularmente agressivo aos tecidos devido à alta toxicidade (OZCAN; ALLAHBEICKARAGHI; DUNDAR, 2012).

Considerando que o tratamento de superfície com ácido fluorídrico pode criar zonas de fragilidade nas restaurações cerâmicas, as quais são submetidas a cargas cíclicas em ambiente oral, uma trinca poderá iniciar e propagar-se para a fratura catastrófica do material. Nesse sentido, os ensaios de fadiga têm se colocado como uma alternativa aos testes monotônicos de resistência flexural, pois se aproximam mais da realidade clínica que os materiais restauradores são submetidos.

Assim, a realização dos dois artigos que compõem esta tese foi impulsionada pela necessidade de prever de maneira mais confiável o comportamento mecânico à fadiga da superfície cerâmica após os procedimentos de condicionamento com ácido fluorídrico e cimentação. Diante disso, objetivou-se avaliar a influência de diferentes concentrações de ácido fluorídrico no condicionamento de espécimes de cerâmica feldspática na forma de discos e de coroas usinadas pelo sistema CAD/CAM, os quais foram cimentados adesivamente a um material análogo a dentina, e submetidos a cargas cíclicas na presença de água.

A partir da evidência de que o condicionamento com ácido fluorídrico tem um efeito de enfraquecimento na resistência de cerâmicas vítreas (ADDISON; FLEMING, 2004; ADDISON; MARQUIS; FLEMING, 2007b; HOOSHMAND; PARVIZI; KESHVAD, 2008; ZOGHEIB, 2011; VENTURINI et al., 2015b), uma das hipóteses testadas foi de que o condicionamento ácido influenciaria negativamente as cargas para falha em fadiga em comparação aos espécimes cerâmicos não condicionados. Entretanto, as coroas não condicionadas (CTRL) e as coroas condicionadas com ácido fluorídrico 1% e 10% (HF1 e HF10) apresentaram valores de carga para falha em fadiga estatisticamente similares, assim como os discos não condicionados e os discos condicionados com HF10. A explicação para esses achados pode estar no procedimento de cimentação mediante a capacidade do cimento resinoso em penetrar nos defeitos da superfície cerâmica e criar uma camada híbrida cerâmica-resina, que é responsável pelo efeito de reforço cerâmico (ADDISON; MARQUIS; FLEMING, 2007a). Investigações prévias já haviam relatado uma influência positiva da aplicação de cimento resinoso na resistência flexural de cerâmicas vítreas (ADDISON; MARQUIS; FLEMING, 2007a; POSRITONG et al., 2013). Além disso, o potencial de penetração do cimento resinoso está diretamente associado a uma maior molhabilidade do aderente, menor ângulo de contato e maior potencial de ligação entre os substratos (AMARAL et al., 2011). Logo, a justificativa para a adequada e homogênea penetração do cimento resinoso nas irregularidades da superfície cerâmica condicionada com ácido fluorídrico 10% deve-se ao maior tamanho e formato arredondado das irregularidades criadas pelo HF10 juntamente ao fato desse ácido ter apresentado um menor ângulo de contato comparado às outras concentrações em estudo prévio (VENTURINI et al., 2015a).

Em relação à influência das diferentes concentrações de ácido fluorídrico, as cargas médias para falha em fadiga foram significativamente inferiores quando as superfícies cerâmicas foram condicionadas com ácido fluorídrico 5% (HF5) tanto nas coroas como nos discos testados. As imagens realizadas em microscópio eletrônico de varredura mostram que o cimento resinoso não conseguiu penetrar completamente nas irregularidades mais pontiagudas criadas pelo ácido fluorídrico 5%, sendo observados vazios de cimento na interface relacionados à área de origem da falha. Nesse cenário, a presença de falhas e vazios de cimento ao longo da superfície de cimentação faz com que as tensões internas se concentrem nessa região e não possam ser distribuídas para o conjunto cerâmica/cimento/dente (ANUSAVICE; HOJJATIE, 1992), o que aumenta a suscetibilidade da cerâmica ao início da trinca e, consequentemente, leva à falha prematura durante cargas baixas.

Como mencionado anteriormente, as coroas cerâmicas condicionadas com ácido fluorídrico 1% apresentaram cargas para falha em fadiga similares as coroas não condicionadas e as condicionadas com ácido fluorídrico 10% (HF1 = CTRL e HF10), diferentemente ao observado nos discos tratados com a concentração de 1% (HF1 < CTRL e HF10). Uma explicação plausível para a diferença entre o comportamento de fadiga das coroas e discos condicionados com HF1 é a topografia da superfície cerâmica decorrente do processo de usinagem das coroas pelo sistema CAD/CAM prévio ao condicionamento. A usinagem dos blocos cerâmicos leva a uma topografia mais rugosa comparada à metodologia do segundo artigo, no qual os discos foram apenas seccionados em uma máquina de corte. Embora os custos da pesquisa sejam reduzidos com essa metodologia de disco simplificada e os resultados sejam praticamente os mesmos entre os estudos (HF5 < CTRL e HF10), as diferenças topográficas devem ser consideradas visto que a usinagem de blocos cerâmicos para CAD/CAM parcialmente ou totalmente sinterizados afeta negativamente a resistência à fadiga flexural (FRAGA et al., 2017). Assim, sugere-se que estudos futuros sobre o efeito de diferentes protocolos de condicionamento simulem a topografia de superfície promovida pela usinagem por meio do sistema CAD/CAM.

De acordo com a fractografia das coroas e discos testados, todas as falhas ocorreram como trincas radiais a partir da superfície de cimentação, o que se aproxima dos estudos que avaliaram coroas cerâmicas que falharam clinicamente (KELLY et al., 1990; THOMPSON et al., 1994), visto que é nesta superfície que se concentram grande parte das tensões de tração responsáveis pelo início da falha (KELLY, 1999). Ademais, o uso de um pistão de resina epóxi com fibra de vidro e de uma tira plástica entre o pistão e o espécime durante o teste pode justificar a ausência de danos de contato na superfície cerâmica (KELLY et al., 2010).

Os estudos *in vitro* que compõem esta tese não simularam completamente todas as condições clínicas, e, desta maneira, contêm limitações inerentes. Nesse sentido, os espécimes testados receberam aplicação de cargas cíclicas apenas no sentido axial, sendo que as forças laterais e de deslizamento, que ocorrem clinicamente durante a mastigação, não foram simuladas. Além disso, o tempo de armazenamento (7 – 14 dias) pode ter sido insuficiente para promover a hidrólise da interface química e interferir na durabilidade da resistência adesiva da restauração cerâmica. Desta maneira, a retenção micromecânica mínima promovida pelo condicionamento com ácido fluorídrico 1%, ou apenas a união química decorrente da aplicação do silano na cerâmica não condicionada, pode ter sido insuficiente para uma resistência de união estável ao longo do tempo. Assim, ensaios clínicos randomizados e investigações adicionais devem ser conduzidos para confirmar os achados destes estudos clinicamente.

5 CONCLUSÃO

A partir dos dois artigos apresentados nesta tese, pode-se concluir que o condicionamento com ácido fluorídrico a 5% reduziu as cargas para falha em fadiga tanto das coroas como dos discos de cerâmica feldspática cimentados adesivamente e, portanto, o uso clínico da concentração de 5% do ácido investigado deve ser considerado cuidadosamente para o condicionamento dessa cerâmica. Assim, com base nos achados desta tese e da necessidade de adesão micromecânica entre os substratos, o condicionamento com ácido fluorídrico a 10% é o tratamento de superfície recomendado para a cerâmica testada visto que não apresentou efeito deletério nas cargas para falha em fadiga em ambos os estudos.

REFERÊNCIAS

ADDISON, O.; FLEMING, G.J.P. The influence of cement lute, thermocycling and surface preparation on the strength of a porcelain laminate veneering material. **Dental Materials**, v.20, n.3, p.286-292, 2004.

ADDISON, O.; MARQUIS, P.M.; FLEMING, G.J.P. Resin elasticity and the strengthening of all-ceramic restorations. **Journal of Dental Research**;v. 86, n.6, p.519-523, 2007a.

ADDISON, O.; MARQUIS, P.M.; FLEMING, G.J.P. The impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. **Dental Materials**, v.23, n.4, p.461-468, 2007b.

AMARAL, R. et al. Resin bonding to a feldspar ceramic after different ceramic surface conditioning methods: evaluation of contact angle, surface pH, and microtensile bond strength durability. **The Journal of Adhesive Dentistry**, v.13, n.6, p.551-560, 2011.

ANAMI, L.C. Finite element analysis of the influence of geometry and design of zirconia crowns on stress distribution. **Journal of Prosthodontics**, v.24, n.2, 146-151, 2015.

ANUSAVICE, K.J.; HOJJATIE, B. Tensile stress in glass-ceramic crowns: effect of flaws and cementation voids. **The International Journal of Prosthodontics** v.5, n.4, p.351-358, 1992.

BINDL, A.; LÜTHY, H.; MÖRMANN, W.H. Thin-wall ceramic CAD/CAM crown copings: strength and fracture pattern. **Journal of Oral Rehabilitation**, v.33, n.7, p.520-528, 2006.

BINDL, A.; MÖRMANN, W.H. Survival rate of mono-ceramic and ceramic-core CAD/CAM-generated anterior crowns over 2-5 years. **European Journal of Oral Sciences**, v.112, n.2, p.197-204, 2004.

BRENTEL, A.S. et al. Microtensile bond strength of a resin cement to feldpathic ceramic after different etching and silanization regimens in dry and aged conditions. **Dental Materials**, v.23, n.11, p.1323-1331, 2007.

CHEN, J.H.; MATSUMURA, H.; ATSUTA, M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. **Journal of Dentistry**, v.28, n.1, p.53-58, 1998.

COLLINS, J.A. Staircase or up-and-down methods. In:__. **Failure of Materials in Mechanical Design**. 2. Ed. New York: John Wiley & sons, 1993. p. 383-390.

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

GIORDANO, R. Materials for chairside CAD/CAM-produced restorations. **The Journal of The American Dental Association**, v.137, n.9, p.14S-21S, 2006.

GUESS, P.C. et al. All-ceramic partial coverage restorations-midterm results of a 5-year prospective clinical splitmouth study. **Journal of Dentistry**, v. 37, n.8, p.627-637, 2009.

HORN, H.R. Porcelain laminate veneers bonded to etched enamel In**:__. Phillips RW editor. Symposium on Dental Material**. Dental Clinics of North America, 1983. v.27, p.671-684.

HOOSHMAND, T.; PARVIZI, S.; KESHVAD, A. Effect of surface acid etching on the biaxial flexural strength of two hotpressed glass ceramics. **Journal of Prosthodontics**, v.17, n.5, p.415-419, 2008.

KELLY, J.R. Clinically relevant approach to failure testing of all-ceramic restorations. **The Journal of Prosthetic Dentistry**, v.81, n.6, p.652-661, 1999.

KELLY, J.R. Dental ceramics: current thinking and trends. **The Dental Clinics of North America**, v.48, n.2, p.513-530, 2004.

KELLY, J.R. et al. Development of clinically validated bulk failure test for ceramic crowns. **The Journal of Prosthetic Dentistry**, v.104, n.4, p.228-238, 2010.

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

MALAMENT, K.A.; SOCRANSKI, S.S. Survival of Dicor glass-ceramic dental restorations over 16 years. Part III: effect of luting agent and tooth or tooth substitute core structure. **The Journal of Prosthetic Dentistry**, v.86, n.5, p.511-519, 2001.

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

MAY, L.G. et al. Influence of the resin cement thickness on the fatigue failure loads of CAD/CAM feldspathic crowns. **Dental Materials**, v.31, n.8, p.895-900, 2015.

MÖRMANN, W.H. et al. Effects of preparation and luting system on all-ceramic computer-generated crowns. **The International Journal of Prosthodontics**, v. 11, n.4, p.333-339, 1998.

OZCAN, M.; ALLAHBEICKARAGHI, A.; DUNDAR, M. Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review. **Clinical Oral Investigations**, v.16, n.1, p.15-23, 2012.

PAGNIANO, R.P. et al. The effect of a layer of resin luting agent on the biaxial flexure strength of two all ceramic systems. **The Journal of Prosthetic Dentistry**, v.93, n.5, p.459-466, 2005.

PHOENIX, R.D.; SHEN C. Characterization of treated porcelain surfaces via dynamic contact angle analysis. **The International Journal of Prosthodontics**, v.8, n.2, p.187-194, 1995.

POSRITONG, S. et al. The impact of hydrofluoric acid etching followed by unfilled resin on the biaxial strength of a glass-ceramic. **Dental Materials**, v.29, n.11, p.e281–e90, 2013.

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

ROULET, J.F., DEGRANGE, M. Inlay restorations. **Journal of the California Dental Association**, v.24, n.9, p.48-62, 1996.

SCHERRER, S.S. et al. Monotonic flexure and fatigue strength of composites for provisional and definitive restorations. **The Journal of Prosthetic Dentistry**, v.89, n.6, p.579-588, 2003.

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

THOMPSON, J.Y. et al. Fracture surface characterization of clinically failed all-ceramic crowns. **Journal of Dental Research**, v.73, n.12, p.1824-1832, 1994.

THORDRUP, M.; ISIDOR, F.; HORSTED-BINDSLEV, P. A 3-year study of inlays milled from machinable ceramic blocks representing 2 different inlay systems. **Quintessence International**, v.30, n.12, p.829-861, 1999.

VENTURINI, A.B. et al. Effect of Hydrofluoric Acid Concentration on Resin Adhesion to a Feldspathic Ceramic. **The Journal of Adhesive Dentistry**, v.17, n.4, p.313-320, 2015a.

VENTURINI, A.B. et al. Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic. **Journal of the Mechanical Behavior of Biomedical Materials**, v.48, p.241-248, 2015b.

VITA ZHANFABRIK: Vitablocks_instructions (pdf file) [homepage na internet]: Bad Säckingen, Alemanha; 2012. Disponível em: <www.carestreamdental.com/ImagesFileShare/.sitecore.media_library.files.CS_Solutions.Certified_Blocks_and_Labs.VITABLOCS_Working_Instructions-EN.pdf>. Acesso em: 29 de maio de 2016.

YEN, T.W.; BLACKMAN, R.B.; BAEZ, R.J. Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic. **The Journal of Prosthetic Dentistry**, v.70, n.3, p.224-233, 1993.

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

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

ZOGHEIB, L.V. et al. Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. **Brazilian Dental Journal**, v.22, n.1, p.45-50, 2011.

ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *THE JOURNAL OF PROSTHETIC DENTISTRY*

Article Types

Articles are classified as one of the following: research/clinical science article, clinical report, technique article, systematic review, or tip from our readers. Required sections for each type of article are listed in the order in which they should be presented.

Research and Education/Clinical Research

The research report should be no longer than 10-12 double-spaced, typed pages and be accompanied by no more than 12 high-quality illustrations. Avoid the use of outline form (numbered and/or bulleted sentences or paragraphs). The text should be written in complete sentences and paragraph form. *Abstract* (approximately 400 words): Create a structured abstract with the following subsections: Statement of Problem, Purpose, Material and Methods, Results, and Conclusions. The abstract should contain enough detail to describe the experimental design and variables. Sample size, controls, method of measurement, standardization, examiner reliability, and statistical method used with associated level of significance should be described in the Material and Methods section. Actual values should be provided in the Results section.

Clinical Implications: In 2-4 sentences, describe the impact of the study results on clinical practice.

Introduction: Explain the problem completely and accurately. Summarize relevant literature, and identify any bias in previous studies. Clearly state the objective of the study and the research hypothesis at the end of the Introduction. Please note that, for a thorough review of the literature, most (if not all references) should first be cited in the Introduction and/or Material and Methods section.

Material and Methods: In the initial paragraph, provide an overview of the experiment. Provide complete manufacturing information for all products and instruments used, either in parentheses or in a table. Describe what was measured, how it was measured, and the units of measure. List criteria for quantitative judgment. Describe the experimental design and variables, including defined criteria to control variables, standardization of testing, allocation of specimens/subjects to groups (specify method of randomization), total sample size, controls, calibration of examiners, and reliability of instruments and examiners. State how sample sizes were determined (such as with power analysis). Avoid the use of group numbers to indicate groups. Instead, use codes or abbreviations that will more clearly indicate the characteristics of the groups and will therefore be more meaningful for the reader. Statistical tests and associated significance levels should be described at the end of this section.

Results: Report the results accurately and briefly, in the same order as the testing was described in the Material and Methods section. For extensive listings, present data in tabular or graphic form to help the reader. For a 1-way ANOVA report of, F and *P* values in the appropriate location in the text. For all other ANOVAs, per guidelines, provide the ANOVA table(s). Describe the most significant findings and trends. Text, tables, and figures should not repeat each other. Results noted as significant must be validated by actual data and P values.

Discussion: Discuss the results of the study in relation to the hypothesis and to relevant literature. The Discussion section should begin by stating whether or not the data support rejecting the stated null hypothesis. If the results do not agree with other studies and/or with accepted opinions, state how and why the results differ. Agreement with other studies should also be stated. Identify the limitations of the present study and suggest areas for future research.
Conclusions: Concisely list conclusions that may be drawn from the research; do not simply restate the results. The conclusions must be pertinent to the objectives and justified by the data. In most situations, the conclusions are true for only the population of the experiment. All statements reported as conclusions should be accompanied by statistical analyses.

References: See Reference Guidelines and Sample References page.
Tables: See Table Guidelines.

Illustrations: See Figure Submission and Sample Figures page.

Clinical Report
The clinical report describes the author's methods for meeting a patient treatment challenge. It should be no longer than 4 to 5 double-spaced, pages and be accompanied by no more than 8 high-quality illustrations. In some situations, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Abstract: Provide a short, nonstructured, 1-paragraph abstract that briefly summarizes the problem encountered and treatment administered.

Introduction: Summarize literature relevant to the problem encountered. Include references to standard treatments and protocols. Please note that most, if not all, references should first be cited in the Introduction and/or Clinical Report section.

Clinical Report: Describe the patient, the problem with which he/she presented, and any relevant medical or dental background. Describe the various treatment options and the reasons for selection of the chosen treatment. Fully describe the treatment rendered, the length of the follow-up period, and any improvements noted as a result of treatment. This section should be written in past tense and in paragraph form.

Discussion: Comment on the advantages and disadvantages of the chosen treatment and describe any contraindications for it. If the text will only be repetitive of previous sections, omit the Discussion.

Summary: Briefly summarize the patient treatment.

References: See Reference Guidelines and Sample References page.

Illustrations: See Figure Submission and Sample Figures page.

Dental Technique
The dental technique article presents, in a step-by-step format, a unique procedure helpful to dental professionals. It should be no longer than 4 to 5 double-spaced, typed pages and be accompanied by no more than 8 high-quality illustrations. In some situations, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Abstract: Provide a short, nonstructured, 1-paragraph abstract that briefly summarizes the technique.

Introduction: Summarize relevant literature. Include references to standard methods and protocols. Please note that most, if not all, references should first be cited in the Introduction and/or Technique section.

Technique: In a numbered, step-by-step format, describe each step of the technique. The text should be written in command rather than descriptive form ("Survey the diagnostic cast" rather than "The diagnostic cast is surveyed.") Include citations for the accompanying illustrations.

Discussion: Comment on the advantages and disadvantages of the technique, indicate the situations to which it may be applied, and describe any contraindications for its use. Avoid excessive claims of effectiveness. If the text will only be repetitive of previous sections, omit the Discussion.

Summary: Briefly summarize the technique presented and its chief advantages.

References: See Reference Guidelines and Sample References page
Illustrations: See Figure Submission and Sample Figures page.

Systematic Review
The author is advised to develop a systematic review in the Cochrane style and format. The *Journal* has transitioned away from literature reviews to systematic reviews. For more information on systematic reviews, please see www.cochrane.org. An example of a Journal systematic review: Torabinejad M, Anderson P, Bader J, Brown LJ, Chen LH, Goodacre CJ, Kattadiyil MT, Kutsenko D, Lozada J, Patel R, Petersen F, Puterman I, White SN. Outcomes of root canal treatment and restoration, implant-supported single crowns, fixed partial dentures, and extraction without replacement: a systematic review. JProsthet Dent 2007;98:285-311.

The systematic review consists of:
An Abstract using a structured format (Statement of Problem, Purpose, Material and Methods, Results, Conclusions).

Text of the review consisting of an introduction (background and objective), methods (selection criteria, search methods, data collection and data analysis), results (description of studies, methodological quality, and results of analyses), discussion, authors' conclusions, acknowledgments, and conflicts of interest. References should be peer reviewed and follow JPD format.

Tables and figures, if necessary, showing characteristics of the included studies, specification of the interventions that were compared, the results of the included studies, a log of the studies that were excluded, and additional tables and figures relevant to the review.

Tips From Our Readers
Tips are brief reports on helpful or timesaving procedures. They should be limited to 2 authors, no longer than 250 words, and include no more than 2 high quality illustrations. Describe the procedure in a numbered, step-by-step format; write the text in command rather than descriptive or passive form ("Survey the diagnostic cast" rather than "The diagnostic cast is surveyed").

Contact Information
The Journal of Prosthetic Dentistry
Editorial Office
The Journal of Prosthetic Dentistry
The Dental College of Georgia at Augusta University
1120 15th St., GC3094
Augusta, GA 30912-1255
Phone: (706) 721-4558
E-mail: JPD@augusta.edu
Website: http://www.prosdent.org
Online submission: http://www.ees.elsevier.com/jpd/

Submission Guidelines
Thank you for your interest in writing an article for *The Journal of Prosthetic Dentistry*. In publishing, as in dentistry, precise procedures are essential. Your attention to and compliance with the following policies will help ensure the timely processing of your submission.

Length of Manuscripts
Manuscript length depends on manuscript type. In general, research and clinical science articles should not exceed 10 to 12 double-spaced, typed pages (excluding references, legends, and tables). Clinical Reports and Technique articles should not exceed 4 to 5 pages, and Tips articles should not exceed 1 to 2 pages. The length of systematic reviews varies.

Number of Authors

The number of authors is limited to 4; the inclusion of more than 4 *must be justified* in the letter of submission. (Each author’s contribution must be listed.) Otherwise, contributing authors in excess of 4 will be listed in the Acknowledgments. There can only be one corresponding author.

General Formatting All submissions must be submitted via the EES system in Microsoft Word with an 8.5×11 inch page size.
The following specifications should also be followed:
• Times Roman, 12 pt
• Double-spaced
• Left-justified
• No space between paragraphs
• 1-inch margins on all sides
• Half-inch paragraph indents
• Headers/Footers should be clear of page numbers or other information
• Headings are upper case bold, and subheads are upper/lower case bold. No italics are used.
• References should not be automatically numbered. Endnote or other reference-generating programs should be turned off.
• Set the Language feature in MS Word to English (US). Also change the language to English (US) in the style named Balloon Text.

Ethics in publishing
Please see our information pages on Ethics in publishing and Ethical guidelines for journal publication.

Declaration of interest
All authors must disclose any financial and personal relationships with other people or organizations that could inappropriately influence (bias) their work. Examples of potential conflicts of interest include employment, consultancies, stock ownership, honoraria, paid expert testimony, patent applications/registrations, and grants or other funding. If there are no conflicts of interest then please state this: 'Conflicts of interest: none'. More information.

Submission declaration and verification
Submission of an article implies that the work described has not been published previously (except in the form of an abstract or as part of a published lecture or academic thesis or as an electronic preprint, see 'Multiple, redundant or concurrent publication' section of our ethics policy for more information), that it is not under consideration for publication elsewhere, that its publication is approved by all authors and tacitly or explicitly by the responsible authorities where the work was carried out, and that, if accepted, it will not be published elsewhere in the same form, in English or in any other language, including electronically without the written consent of the copyright-holder. To verify originality, your article may be checked by the originality detection service CrossCheck.

Changes to authorship
Authors are expected to consider carefully the list and order of authors **before** submitting their manuscript and provide the definitive list of authors at the time of the original submission. Any addition, deletion or rearrangement of author names in the authorship list should be made only **before** the manuscript has been accepted and only if approved by the journal Editor. To request such a change, the Editor must receive the following from the **corresponding author**: (a) the reason for the change in author list and (b) written confirmation (e-mail, letter) from all authors that they agree with the addition, removal or rearrangement. In the case of addition or removal of authors, this includes confirmation from the author being added or removed. Only in exceptional circumstances will the Editor consider the addition, deletion or rearrangement of authors **after** the manuscript has been accepted. While the Editor considers the request, publication of the manuscript will be suspended. If the manuscript has already been published in an online issue, any requests approved by the Editor will result in a corrigendum.

Copyright
Upon acceptance of an article, authors will be asked to complete a 'Journal Publishing Agreement' (see more information on this). An e-mail will be sent to the corresponding author confirming receipt of the manuscript together with a 'Journal Publishing Agreement' form or a link to the online version of this agreement.
Subscribers may reproduce tables of contents or prepare lists of articles including abstracts for internal circulation within their institutions. Permission of the Publisher is required for resale or distribution outside the institution and for all other derivative works, including compilations and translations. If excerpts from other copyrighted works are included, the author(s) must obtain written permission from the copyright owners and credit the source(s) in the article. Elsevier has preprinted forms for use by authors in these cases.
For open access articles: Upon acceptance of an article, authors will be asked to complete an 'Exclusive License Agreement' (more information). Permitted third party reuse of open access articles is determined by the author's choice of user license.

Author rights
As an author you (or your employer or institution) have certain rights to reuse your work. More information.
Elsevier supports responsible sharing
Find out how you can share your research published in Elsevier journals.

Role of the funding source
You are requested to identify who provided financial support for the conduct of the research and/or preparation of the article and to briefly describe the role of the sponsor(s), if any, in study design; in the collection, analysis and interpretation of data; in the writing of the report; and in the decision to submit the article for publication. If the funding source(s) had no such involvement then this should be stated.

Funding body agreements and policies
Elsevier has established a number of agreements with funding bodies which allow authors to comply with their funder's open access policies. Some funding bodies will reimburse the author for the Open Access Publication Fee. Details of existing agreements are available online.

Creative Commons Attribution (CC BY)
Lets others distribute and copy the article, create extracts, abstracts, and other revised versions, adaptations or derivative works of or from an article (such as a translation), include in a collective work (such as an anthology), text or data mine the article, even for commercial purposes, as long as they credit the author(s), do not represent the author as endorsing their adaptation of the article, and do not modify the article in such a way as to damage the author's honor or reputation.

Creative Commons Attribution-NonCommercial-NoDerivs (CC BY-NC-ND)
For non-commercial purposes, lets others distribute and copy the article, and to include in a collective work (such as an anthology), as long as they credit the author(s) and provided they do not alter or modify the article.
The open access fee for this journal is **USD 2500**, excluding taxes. Learn more about Elsevier's pricing policy: https://www.elsevier.com/openaccesspricing.

Green open access
Authors can share their research in a variety of different ways and Elsevier has a number of green open access options available. We recommend authors see our green open access page for further information. Authors can also self-archive their manuscripts immediately and enable public access from their institution's repository after an embargo period. This is the version that has been accepted for publication and which typically includes author-incorporated changes suggested during submission, peer review and in editor-author communications. Embargo period: For subscription articles, an appropriate amount of time is needed for journals to deliver value to subscribing customers

before an article becomes freely available to the public. This is the embargo period and it begins from the date the article is formally published online in its final and fully citable form. Find out more.

Language (usage and editing services)
Please write your text in good American English. Authors who feel their English language manuscript may require editing to eliminate possible grammatical or spelling errors and to conform to correct scientific English may wish to use the English Language Editing service available from Elsevier's WebShop http://webshop.elsevier.com/languageediting/ or visit our customer support site http://support.elsevier.com for more information.

Informed consent and patient details
Studies on patients or volunteers require ethics committee approval and informed consent, which should be documented in the paper. Appropriate consents, permissions and releases must be obtained where an author wishes to include case details or other personal information or images of patients and any other individuals in an Elsevier publication. Written consents must be retained by the author and copies of the consents or evidence that such consents have been obtained must be provided to Elsevier on request. For more information, please review the Elsevier Policy on the Use of Images or Personal Information of Patients or other Individuals. Unless you have written permission from the patient (or, where applicable, the next of kin), the personal details of any patient included in any part of the article and in any supplementary materials (including all illustrations and videos) must be removed before submission.

Submission
Our online submission system guides you stepwise through the process of entering your article details and uploading your files. The system converts your article files to a single PDF file used in the peerreview process. Editable files (e.g., Word, LaTeX) are required to typeset your article for final publication. All correspondence, including notification of the Editor's decision and requests for revision, is sent by email.

Submit your article
Please submit your article via http://www.ees.elsevier.com/jpd/.

Use of word processing software
It is important that the file be saved in the native format of the MS Word program. The text should be in single-column format. Keep the layout of the text as simple as possible. Most formatting codes will be removed and replaced on processing the article. In particular, do not use the word processor's options to justify text or to hyphenate words. However, do use bold face, italics, subscripts, superscripts etc. When preparing tables, if you are using a table grid, use only one grid for each individual table and not a grid for each row. If no grid is used, use tabs, not spaces, to align columns. The electronic text should be prepared in a way very similar to that of conventional manuscripts (see also the Guide to Publishing with Elsevier: http://www.elsevier.com/guidepublication). Note that source files of figures, tables and text graphics will be required whether or not you embed your figures in the text. See also the section on Electronic artwork.
To avoid unnecessary errors you are strongly advised to use the 'spell-check' and 'grammar-check' functions of your word processor.

Embedded math equations
If you are submitting an article prepared with Microsoft Word containing embedded math equations then please read this related support information (http://support.elsevier.com/app/answers/detail/a_id/302/).

Essential title page information
• *Title.*Concise and informative. Titles are often used in information-retrieval systems. Avoid abbreviations and formulae. Trade names should not be used in the title.

- *Author names and affiliations.* Author's names should be complete first and last names. Where the family name may be ambiguous (e.g., a double name), please indicate this clearly. Present the authors' current title and affiliation, including the city and state/country of that affiliation. If it is private practice, indicate the city and state/country of the practice. Indicate all affiliations with a lower-case superscript letter immediately after the author's name and in front of the appropriate affiliation.
- *Corresponding author.* Clearly indicate who will handle correspondence at all stages of refereeing and publication, also post-publication. Ensure that phone numbers (with country and area code) are provided in addition to the e-mail address and the complete postal address. Contact details must be kept up to date by the corresponding author.

Title page format

- Title: Capitalize only the first letter of the first word. Do not use any special formatting. Abbreviations or trade names should not be used. Trade names should not be used in the title.
- Authors: Directly under the title, type the names and academic degrees of the authors.
- Under the authors' names, provide the title, department and institutional names, city/state and country (unless in the U.S.) of each author. If necessary, provide the English translation of the institution. If the author is in private practice, indicate where with city/state/country. Link names and affiliations with a superscript letter (a,b,c,d).
- Presentation/support information and titles: If research was presented before an organized group, indicate name of the organization and location and date of the meeting. If work was supported by a grant or any other kind of funding, supply the name of the supporting organization and the grant number.
- Corresponding author: List the mailing address, business telephone, and e-mail address of the author who will receive correspondence.
- Acknowledgments: Indicate special thanks to persons or organizations involved with the manuscript.
- See Sample Title page.

Formatting of funding sources

List funding sources in this standard way to facilitate compliance to funder's requirements:

Funding: This work was supported by the National Institutes of Health [grant numbers xxxx, yyyy]; the
Bill & Melinda Gates Foundation, Seattle, WA [grant number zzzz]; and the United States Institutes of Peace [grant number aaaa].

It is not necessary to include detailed descriptions on the program or type of grants and awards. When funding is from a block grant or other resources available to a university, college, or other research institution, submit the name of the institute or organization that provided the funding.

If no funding has been provided for the research, please include the following sentence:

This research did not receive any specific grant from funding agencies in the public, commercial, or notfor-profit sectors.

Units

Follow internationally accepted rules and conventions: use the international system of units (SI). If other units are mentioned, please give their equivalent in SI.

Math formulae

Please submit math equations as editable text and not as images. Present simple formulae in line with normal text where possible and use the solidus (/) instead of a horizontal line for small fractional terms, e.g., X/Y. In principle, variables are to be presented in italics. Powers of e are often more conveniently denoted by exp. Number consecutively any equations that have to be displayed separately from the text (if referred to explicitly in the text).

Embedded math equations
If you are submitting an article prepared with Microsoft Word containing embedded math equations then please read this (related support information).

Artwork
Figure Submission
JPD takes pride in publishing only the highest quality figures in its journal. All incoming figures must pass a thorough examination in Photoshop before the review process can begin. With more than 1,000 manuscripts submitted yearly, the manuscripts with few to no submission errors move through the system quickly. Figures that do not meet the guidelines will be sent back to the author for correction and moved to the bottom of the queue, creating a delay in the publishing process.

File Format
All figures should be submitted as TIF files or JPEG files only.

Image File Specifications
Figure dimensions must be 5.75 × 3.85 inches.
Figures should be size-matched (the same physical size) unless the image type prohibits size matching to other figures within the manuscript, as in the case of panoramic or periapical radiographs, SEM images, or graphs and screen shots. Do not "label" the faces of the figures with letters or numbers to indicate the order in which the figures should appear; such labels will be inserted during the publication process. Do not add wide borders to increase size.

Resolution
The figures should be of professional quality and high resolution. The following are resolution requirements:
- Color and black-and-white photographs should be created and saved at 300 dots per inch (dpi).
- Note: A 5.75 × 3.85-inch image at a resolution of 300 dpi will be approximately 6 megabytes. A figure of less than 300 dpi must not be increased artificially to 300 dpi; the resulting quality and resolution will be poor.
- Line art or combination artwork (an illustration containing both line art and photograph) should be created and saved at a minimum of 600dpi.
- Clarity, contrast, and quality should be uniform among the parts of a multipart figure and among all of the figures within a manuscript.
- A uniform background of nontextured, medium blue should be provided for color figures when possible.

Text within Images
If text is to appear within the figure, labeled and unlabeled versions of the figures must be provided. Text appearing within the labeled versions of the figures should be in **Arial font and a minimum of 10 pt.** The text should be sized for readability if the figure is reduced for production in the Journal. Lettering should be in proportion to the drawing, graph, or photograph. A consistent font size should be used throughout each figure, and for all figures, Please note: Titles and captions should not appear within the figure file, but should be provided in the manuscript text (see Figure Legends).
If a key to an illustration requires artwork (screen lines, dots, unusual symbols), the key should be incorporated into the drawing instead of included in the typed legend. All symbols should be done professionally, be visible against the background, and be of legible proportion should the illustration be reduced for publication.
All microscopic photographs must have a measurement bar and unit of measurement on the image.

Color Figures
Generally, a maximum of 8 figures will be accepted for clinical report and dental technique articles, and 2 figures will be accepted for tips from our reader articles. However, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Clinical figures should be color balanced. Color images should be in CMYK (Cyan/Magenta/Yellow/Black) color format as opposed to RGB (Red/Green/Blue) color format.

Graphs/Screen Captures
Graphs should be numbered as figures, and the fill for bar graphs should be distinctive and solid; no shading or patterns. Thick, solid lines should be used and bold, solid lettering. Arial font is preferred. Place lettering on white background is preferred to reverse type (white lettering on a dark background).Line drawing should be a minimum of 600 dpi. Screen Captures should be a minimum of 300 dpi and as close to 5.75 and 3.85 as possible.

Composites
Composites are multiple images within one Figure file and, as a rule, are not accepted. They will be sent back to the author to replace them with each image sent separately as, Fig. 1A, Fig. 1B, Fig. 1C, etc. Each figure part must meet JPD Guidelines. (Some composite figures are more effective when submitted as one file. These files will be reviewed per case.) Contact the editorial office for more information about specific composites.

Figure Legends
The figure legends should appear within the text of the manuscript on a separate page after Tables and should appear under the heading FIGURES. Journal style requires that the articles (a, an, and the) are omitted from the figure legends. If an illustration is taken from previously published material, the legend must give full credit to the source (see Permissions).

File Naming
Each figure file must be numbered according to its position in the text (Figure 1, Figure 2, and so on) with Arabic numerals. The electronic image files must be named so that the figure number and format can be easily identified. For example, a Figure 1 in TIFF format should be named fig. 1.tif. Multipart figures must be clearly identifiable by the file names: Fig. 1A, Fig. 1B, Fig. 1C, Fig. 1-unlabeled, Fig. 1-labeled, etc.

Callouts
In the article, clearly reference each Figure and Table by including its number in parentheses at the end of the appropriate sentence before closing punctuation. For example: The sutures were removed after 3 weeks (Fig. 4). Or: are illustrated in Table 4.
The *Journal* reserves the right to standardize the format of graphs and tables.
Authors are obligated to disclose whether illustrations have been modified in any way.

Thumbnails
Place thumbnails (reduced size versions) of your figures in Figures section below each appropriate legend.
Thumbnails refers to placing a small (compressed file) copy of your figure into the FIGURES section of the manuscript after each appropriate legend. No smaller than 2" × 1.5" and approximately 72dpi. The goal is to give the editors/reviewers something to review but we want to keep the dimensions and the file size small for easy access. These small images are called thumbnails.

Figures Quick Checklist
- All files are saved as TIFFs or JPEGs (only).
- Figure size: 5.75" × 3.85" (radiographs, SEMS, and screen captures may vary but they must all be size matched).
- Figures are 300 dpi; line or combo line/photo illustrations are minimum 600 dpi.
- For text in figures use Ariel font.
- Label the Figure files according to their sequence in the text.
- Provide figure legends in the manuscript Figure section.

• Place thumbnails (small versions of figure files approx. 2" × 1.5") in Figure section below each legend.
• Submit composite figure parts as separate files.

A detailed guide to electronic artwork is available on our website: You are urged to visit this site; some excerpts from the detailed information about figure preparation are given here. http://www.elsevier.com/artworkinstructions.

Please make sure that artwork files are TIFFs and with the correct resolution. If, together with your accepted article, you submit usable color figures then Elsevier will ensure, at no additional charge, that these figures will appear in color online (e.g., ScienceDirect and other sites) in addition to color reproduction in print. For further information on the preparation of electronic artwork, please see http://www.elsevier.com/artworkinstructions.

Illustration services
Elsevier's WebShop (http://webshop.elsevier.com/illustrationservices) offers Illustration Services to authors preparing to submit a manuscript but concerned about the quality of the images accompanying their article. Elsevier's expert illustrators can produce scientific, technical, and medical-style images, as well as a full range of charts, tables, and graphs. Image 'polishing' is also available, where our illustrators take your image(s) and improve them to a professional standard. Please visit the website to find out more.

Electronic Artwork
General points
• Make sure you use uniform lettering and sizing.
• Embed the used fonts if the application provides that option.
• Use the font Ariel or Helvetica in your illustrations.
• Number the illustration files according to their sequence in the text.
• Use a logical naming convention for your artwork files.
• Provide figure legends in the Figure section.
• Size the illustrations close to the desired dimensions of the published version.
• Submit each illustration as a separate file.

A detailed guide on electronic artwork is available on our website: http://www.elsevier.com/artworkinstructions. **You are urged to visit this site; some excerpts from the detailed information are given here.**

Formats
If your electronic artwork is created in a Microsoft Office application (Word, PowerPoint, Excel) then please supply 'as is' in the native document format.
Regardless of the application used other than Microsoft Office, when your electronic artwork is finalized, please 'Save as' or convert the images to one of the following formats (note the resolution requirements for line drawings, halftones, and line/halftone combinations given below):
TIFF (or JPEG): Color or grayscale photographs (halftones), keep to a minimum of 300 dpi. TIFF (or JPEG): Bitmapped (pure black & white pixels) line drawings, keep to a minimum of 600 dpi. TIFF (or JPEG): Combinations bitmapped line/half-tone (color or grayscale), keep to a minimum of 600 dpi.

Please do not:
• Supply files that are optimized for screen use (e.g., GIF, PNG, PICT, WPG); these typically have a low number of pixels and limited set of colors;
• Supply files that are too low in resolution? or smaller than 5.75 × 3.85-inch.;
• Submit graphics that are disproportionately large for the content.

Color artwork

Please make sure that artwork files are in an acceptable format (TIFF or JPEG) and with the correct size and resolution. If, together with your accepted article, you submit usable color figures then Elsevier will ensure, at no additional charge, that these figures will appear in color online (e.g., ScienceDirect and other sites) in addition to color reproduction in print. For further information on the preparation of electronic artwork, please see http://www.elsevier.com/artworkinstructions.

Illustration services
Elsevier's WebShop offers Illustration Services to authors preparing to submit a manuscript but concerned about the quality of the images accompanying their article. Elsevier's expert illustrators can produce scientific, technical and medical-style images, as well as a full range of charts, tables and graphs. Image 'polishing' is also available, where our illustrators take your image(s) and improve them to a professional standard. Please visit the website to find out more.

Figure captions
Ensure that each illustration has a caption. Supply captions separately, not attached to the figure. A caption should comprise a brief title (**not** on the figure itself) and a description of the illustration. Keep text in the illustrations themselves to a minimum but explain all symbols and abbreviations used. See Sample Figures page.

Tables

- Tables should be self-explanatory and should supplement, not duplicate the text.
- Provide all tables at the end of the manuscript after the reference list and before the Figures. There should be only one table per page. Omit internal horizontal and vertical rules (lines). Omit any shading or color.
- Do not list tables in parts (Table Ia, Ib, etc.). Each should have its own number. Number the tables in the order in which they are mentioned in the text (Table 1., Table 2, etc).
- Supply a concise legend that describes the content of the table. Create descriptive column and row headings. Within columns, align data such that decimal points may be traced in a straight line. Use decimal points (periods), not commas, to mark places past the integer (eg, 3.5 rather than 3,5).
- In a line beneath the table, define any abbreviations used in the table.
- If a table (or any data within it) was published previously, give full credit to the original source in a footnote to the table. If necessary, obtain permission to reprint from the author/publisher.
- The tables should be submitted in Microsoft Word. If a table has been prepared in Excel, it should be imported into the manuscript.

References
Citation in text
Please ensure that every reference cited in the text is also present in the reference list (and vice versa). Any references cited in the abstract must be given in full. Unpublished results and personal communications are not permitted in the reference list, but may be mentioned in the text. Citation of a reference as 'in press' implies that the item has been accepted for publication.

Reference links
Increased discoverability of research and high quality peer review are ensured by online links to the sources cited. In order to allow us to create links to abstracting and indexing services, such as Scopus, CrossRef and PubMed, please ensure that data provided in the references are correct. Please note that incorrect surnames, journal/book titles, publication year and pagination may prevent link creation. When copying references, please be careful as they may already contain errors. Use of the DOI is encouraged.

A DOI can be used to cite and link to electronic articles where an article is in-press and full citation details are not yet known, but the article is available online. A DOI is guaranteed never to change, so you can use it as a permanent link to any electronic article. An example of a citation using DOI for an article not yet in an issue is: VanDecar J.C., Russo R.M., James D.E., Ambeh W.B., Franke M. (2003).

Aseismic continuation of the Lesser Antilles slab beneath northeastern Venezuela. Journal of Geophysical Research, https://doi.org/10.1029/2001JB000884. Please note the format of such citations should be in the same style as all other references in the paper.

Data references
This journal encourages you to cite underlying or relevant datasets in your manuscript by citing them in your text and including a data reference in your Reference List. Data references should include the following elements: author name(s), dataset title, data repository, version (where available), year, and global persistent identifier. Add [dataset] immediately before the reference so we can properly identify it as a data reference. The [dataset] identifier will not appear in your published article.

Acceptable references and their placement
• Most, if not all, references should first be cited in the Introduction and/or Material and Methods section. Only those references that have been previously cited or that relate directly to the outcomes of the present study may be cited in the Discussion.
• Only peer-reviewed, published material may be cited as a reference. Manuscripts in preparation, manuscripts submitted for consideration, and unpublished theses are not acceptable references.
• Abstracts are considered unpublished observations and are not allowed as references unless followup studies were completed and published in peer-reviewed journals.
• References to foreign language publications should be kept to a minimum (no more than 3). **They are permitted only when the original article has been translated into English.** The translated title should be cited and the original language noted in brackets at the end of the citation.
• Textbook references should be kept to a minimum, as textbooks often reflect the opinions of their authors and/or editors. The most recent editions of textbooks should be used. Evidence-based journal citations are preferred.

Reference formatting
• References must be identified in the body of the article with superscript Arabic numerals. At the end of a sentence, the reference number falls *after* the period.
• The complete reference list, double-spaced and in numerical order, should follow the Conclusions section but start on a separate page. Only references cited in the text should appear in the reference list.
• Reference formatting should conform to **Vancouver style** as set forth in "Uniform Requirements for Manuscripts Submitted to Biomedical Journals" (Ann Intern Med 1997;126:36-47).
• References should be manually numbered.
• List up to six authors. If there are seven or more, after the sixth author's name, add et al.
• Abbreviate journal names per the **Cumulative Index Medicus**. A complete list of standard abbreviations is available through the PubMed website: http://www.ncbi.nlm.nih.gov/nlmcatalog/journals.
• Format for journal articles: Supply the last names and initials of all authors; the title of the article; the journal name; and the year, volume, and page numbers of publication. Do not use italics, bold, or underlining for any part of the reference. Put a period after the initials of the last author, after the article title, and at the end of the reference. Put a semicolon after the year of publication and a colon after the volume. *Issue numbers are not used in Vancouver style*.
• Ex: Jones ER, Smith IM, Doe JQ. Uses of acrylic resin. J Prosthet Dent 1985;53:120-9.
• Book References: The most current edition must be cited. Supply the names and initials of all authors/editors, the title of the book, the city of publication, the publisher, the year of publication, and the inclusive page numbers consulted. Do not use italics, bold, or underlining for any part of the reference.
• Ex: Zarb GA, Carlsson GE, Bolender CL. Boucher's prosthodontic treatment for edentulous patients. 11th ed. St. Louis: Mosby; 1997. p. 112-23.

References should not be submitted in Endnote or other reference-generating software. Endnote formatting cannot be edited by the Editorial Office or reviewers, and must be suppressed or

removed from the manuscript prior to submission. Nor should references be automatically numbered. Please number manually.

See Sample Manuscript.
Approved Abbreviations for Journals
Because the *Journal of Prosthetic Dentistry* is published not only in print but also online, authors must use the standard PubMed abbreviations for journal titles. If alternate or no abbreviations are used, the references will not be linked in the online publication. A complete list of standard abbreviations is available through the PubMed website: http://www.ncbi.nlm.nih.gov/nlmcatalog/journals.

Video
Elsevier accepts video material and animation sequences to support and enhance your scientific research. Authors who have video or animation files that they wish to submit with their article are strongly encouraged to include links to these within the body of the article. This can be done in the same way as a figure or table by referring to the video or animation content and noting in the body text where it should be placed. All submitted files should be properly labeled so that they directly relate to the video file's content. In order to ensure that your video or animation material is directly usable, please provide the files in one of our recommended file formats with a preferred maximum size of 150 MB. Video and animation files supplied will be published online in the electronic version of your article in Elsevier Web products, including ScienceDirect. Please supply 'stills' with your files: you can choose any frame from the video or animation or make a separate image. These will be used instead of standard icons and will personalize the link to your video data. For more detailed instructions please visit our video instruction pages. Note: since video and animation cannot be embedded in the print version of the journal, please provide text for both the electronic and the print version for the portions of the article that refer to this content.

Supplementary material
Supplementary material such as applications, images and sound clips, can be published with your article to enhance it. Submitted supplementary items are published exactly as they are received (Excel or PowerPoint files will appear as such online). Please submit your material together with the article and supply a concise, descriptive caption for each supplementary file. If you wish to make changes to supplementary material during any stage of the process, please make sure to provide an updated file. Do not annotate any corrections on a previous version. Please switch off the 'Track Changes' option in Microsoft Office files as these will appear in the published version.

AudioSlides
The journal encourages authors to create an AudioSlides presentation with their published article. AudioSlides are brief, webinar-style presentations that are shown next to the online article on ScienceDirect. This gives authors the opportunity to summarize their research in their own words and to help readers understand what the paper is about. More information and examples are available. Authors of this journal will automatically receive an invitation e-mail to create an AudioSlides presentation after acceptance of their paper.

Submission Checklist
The following list will be useful during the final checking of an article before sending it to the journal for review. Please consult this Guide for Authors for further details of any item. **Ensure the following items are present:**

One author has been designated as the corresponding author with contact details:
• Email address
• Full postal address
• Phone number

All necessary files have been uploaded, and contain the following:
• All figure thumbnails and legends

• All tables (including title, description, footnotes)
• Justification letter for more than 4 authors
• Patient photo permission
• IRB statements

Further considerations:
• Manuscript has been 'spell-checked' and 'grammar-checked'
• References are in the correct format for this journal
• All references mentioned in the Reference list are cited in the text, and vice versa
• There are call-outs for each figure in the text
• Permission has been obtained for the use of copyrighted material from other sources (including the Web)

For any further information please visit our customer support site at http://support.elsevier.com.

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

GUIDE FOR AUTHORS

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

Dental Materials now only accepts online submissions.

The Artwork Quality Control Tool is now available to users of the online submission system. To help authors submit high-quality artwork early in the process, this tool checks the submitted artwork and other file types against the artwork requirements outlined in the Artwork Instructions to Authors onhttp://www.elsevier.com/artworkinstructions. The Artwork Quality Control Tool automatically checks all artwork files when they are first uploaded. Each figure/file is checked only once, so further along in the process only new uploaded files will be checked.

Manuscripts

The journal is principally for publication of Original Research Reports, which should preferably investigate a defined hypothesis. Maximum length 6 journal pages (approximately 20 double-spaced typescript pages) including illustrations and tables.

Systematic Reviews will however be considered. Intending authors should communicate with the Editor beforehand, by email, outlining the proposed scope of the review. Maximum length 10 journal pages (approximately 33 double-spaced typescript pages) including figures and tables. Three copies of the manuscript should be submitted: each accompanied by a set of illustrations. The requirements for submission are in accordance with the "Uniform Requirements for Manuscripts Submitted to Biomedical Journals", Annals of Internal Medicine, 1997,126, 36-47. All manuscripts must be written in American English. Authors are urged to write as concisely as possible. The Editor and Publisher reserve the right to make minimal literary corrections for the sake of clarity. Authors for whom English is not the first language should have their manuscripts read by colleagues fluent in English. If extensive English corrections are needed, authors may be charged for the cost of editing. For additional reference, consult issues of Dental Materials published after January 1999or the Council of Biology Editors Style Manual (1995 ed.).

All manuscripts should be accompanied by a letter of transmittal, signed by each author, and stating that the manuscript is not concurrently under consideration for publication in another journal, that all of the named authors were involved in the work leading to the publication of the paper, and that all the named authors have read the paper before it is submitted for publication. Always keep a backup copy of the electronic file for reference and safety. Manuscripts not conforming to the journal style will be returned. In addition, manuscripts, which are not written in fluent English, will be rejected automatically without refereeing.

Article structure

Subdivision - numbered sections

Divide your article into clearly defined and numbered sections. Subsections should be numbered1.1 (then 1.1.1, 1.1.2, ...), 1.2, etc. (the abstract is not included in section numbering). Use this numbering also for internal cross-referencing: do not just refer to 'the text'. Any subsection may be given a brief heading. Each heading should appear on its own separate line.

Introduction

This must be presented in a structured format, covering the following subjects, although actual subheadings should not be included:

• succinct statements of the issue in question;
• the essence of existing knowledge and understanding pertinent to the issue (reference);

• the aims and objectives of the research being reported relating the research to dentistry, where not obvious.

Materials and methods
• describe the procedures and analytical techniques.
• only cite references to published methods.
• include at least general composition details and batch numbers for all materials.
• identify names and sources of all commercial products e.g.
"The composite (Silar, 3M Co., St. Paul, MN, USA)..."
"... an Au-Pd alloy (Estheticor Opal, Cendres et Metaux, Switzerland)."
• specify statistical significance test methods.

Results
• refer to appropriate tables and figures.
• refrain from subjective comments.
• make no reference to previous literature.
• report statistical findings.

Discussion
• explain and interpret data.
• state implications of the results, relate to composition.
• indicate limitations of findings.
• relate to other relevant research.

Conclusion (if included)
• must NOT repeat Results or Discussion
• must concisely state inference, significance, or consequences

Appendices
If there is more than one appendix, they should be identified as A, B, etc. Formulae and equations in appendices should be given separate numbering: Eq. (A.1), Eq. (A.2), etc.; in a subsequent appendix, Eq. (B.1) and so on. Similarly for tables and figures: Table A.1; Fig. A.1, etc.

Essential title page information
• Title. Concise and informative. Titles are often used in information-retrieval systems. Avoid abbreviations and formulae where possible.
• Author names and affiliations. Please clearly indicate the given name(s) and family name(s) of each author and check that all names are accurately spelled. Present the authors' affiliation addresses (where the actual work was done) below the names. Indicate all affiliations with a lower case superscript letter immediately after the author's name and in front of the appropriate address.
Provide the full postal address of each affiliation, including the country name and, if available, thee-mail address of each author.
• Corresponding author. Clearly indicate who will handle correspondence at all stages of refereeing and publication, also post-publication. Ensure that the e-mail address is given and that contact details are kept up to date by the corresponding author.
• Present/permanent address. If an author has moved since the work described in the article was done, or was visiting at the time, a 'Present address' (or 'Permanent address') may be indicated as a footnote to that author's name. The address at which the author actually did the work must be retained as the main, affiliation address. Superscript Arabic numerals are used for such footnotes.

Abstract (structured format)
• 250 words or less.
• subheadings should appear in the text of the abstract as follows: Objectives, Methods, Results, Significance. (For Systematic Reviews: Objectives, Data, Sources, Study selection, Conclusions). The Results section may incorporate small tabulations of data, normally 3 rows maximum.

Keywords
Up to 10 keywords should be supplied e.g. dental material, composite resin, adhesion.

Abbreviations
Define abbreviations that are not standard in this field in a footnote to be placed on the first page of the article. Such abbreviations that are unavoidable in the abstract must be defined at their first mention there, as well as in the footnote. Ensure consistency of abbreviations throughout the article.

Acknowledgements
Collate acknowledgements in a separate section at the end of the article before the references and do not, therefore, include them on the title page, as a footnote to the title or otherwise. List here those individuals who provided help during the research (e.g., providing language help, writing assistance or proof reading the article, etc.).

Units
Follow internationally accepted rules and conventions: use the international system of units (SI). If other units are mentioned, please give their equivalent in SI.

Math formulae
Please submit math equations as editable text and not as images. Present simple formulae in line with normal text where possible and use the solidus (/) instead of a horizontal line for small fractional terms, e.g., X/Y. In principle, variables are to be presented in italics. Powers of e are often more conveniently denoted by exp. Number consecutively any equations that have to be displayed separately from the text (if referred to explicitly in the text).

Embedded math equations
If you are submitting an article prepared with Microsoft Word containing embedded math equations then please read this related support information (http://support.elsevier.com/app/answers/detail/a_id/302/).

Footnotes
Footnotes should be used sparingly. Number them consecutively throughout the article. Many word processors can build footnotes into the text, and this feature may be used. Otherwise, please indicate the position of footnotes in the text and list the footnotes themselves separately at the end of the article. Do not include footnotes in the Reference list.

Artwork
Electronic artwork
General points
• Make sure you use uniform lettering and sizing of your original artwork.
• Embed the used fonts if the application provides that option.
• Aim to use the following fonts in your illustrations: Arial, Courier, Times New Roman, Symbol, or use fonts that look similar.
• Number the illustrations according to their sequence in the text.
• Use a logical naming convention for your artwork files.
• Provide captions to illustrations separately.
• Size the illustrations close to the desired dimensions of the published version.
• Submit each illustration as a separate file.
A detailed guide on electronic artwork is available on our website: http://www.elsevier.com/artworkinstructions.
You are urged to visit this site; some excerpts from the detailed information are given here.

Formats
If your electronic artwork is created in a Microsoft Office application (Word, PowerPoint, Excel) then

please supply 'as is' in the native document format. Regardless of the application used other than Microsoft Office, when your electronic artwork is finalized, please 'Save as' or convert the images to one of the following formats (note the resolution requirements for line drawings, halftones, and line/halftone combinations given below):
EPS (or PDF): Vector drawings, embed all used fonts.
TIFF (or JPEG): Color or grayscale photographs (halftones), keep to a minimum of 300 dpi.
TIFF (or JPEG): Bitmapped (pure black & white pixels) line drawings, keep to a minimum of 1000 dpi.
TIFF (or JPEG): Combinations bitmapped line/half-tone (color or gray scale), keep to a minimum of 500 dpi. Please do not:
• Supply files that are optimized for screen use (e.g., GIF, BMP, PICT, WPG); these typically have a low number of pixels and limited set of colors;
• Supply files that are too low in resolution;
• Submit graphics that are disproportionately large for the content.

Color artwork
Please make sure that artwork files are in an acceptable format (TIFF (or JPEG), EPS (or PDF), or MS Office files) and with the correct resolution. If, together with your accepted article, you submit usable color figures then Elsevier will ensure, at no additional charge, that these figures will appear in color online (e.g., Science Direct and other sites) regardless of whether or not these illustrations are reproduced in color in the printed version. For color reproduction in print, you will receive information regarding the costs from Elsevier after receipt of your accepted article. Please indicate your preference for color: in print or online only. For further information on the preparation of electronic artwork, please see http://www.elsevier.com/artworkinstructions.
Please note: Because of technical complications that can arise by converting color figures to 'gray scale' (for the printed version should you not opt for color in print) please submit in addition usable black and white versions of all the color illustrations.

Captions to tables and figures
• list together on a separate page.
• should be complete and understandable apart from the text.
• include key for symbols or abbreviations used in Figures.
• individual teeth should be identified using the FDI two-digit system.

Tables
Please submit tables as editable text and not as images. Tables can be placed either next to the relevant text in the article, or on separate page(s) at the end. Number tables consecutively in accordance with their appearance in the text and place any table notes below the table body. Be sparing in the use of tables and ensure that the data presented in them do not duplicate results described elsewhere in the article. Please avoid using vertical rules.

References
Must now be given according to the following numeric system:
Cite references in text in numerical order. Use square brackets: in-line, not superscript e.g. [23]. All references must be listed at the end of the paper, double-spaced, without indents. For example: 1. Moulin P, Picard B and Degrange M. Water resistance of resin-bonded joints with time related to alloy surface treatments. J Dent, 1999; 27:79-87. 2. Taylor DF, Bayne SC, Sturdevant JR and Wilder AD. Comparison of direct and indirect methods for analyzing wear of posterior composite restorations. Dent Mater, 1989; 5:157-160. Avoid referencing abstracts if possible. If unavoidable, reference as follows: 3. Demarest VA and Greener EH .Storage moduli and interaction parameters of experimental dental composites. J Dent Res, 1996; 67:221, Abstr. No. 868.

Citation in text
Please ensure that every reference cited in the text is also present in the reference list (and vice versa). Any references cited in the abstract must be given in full. Unpublished results and personal

communications are not recommended in the reference list, but may be mentioned in the text. If these references are included in the reference list they should follow the standard reference style of the journal and should include a substitution of the publication date with either 'Unpublished results' or 'Personal communication'. Citation of a reference as 'in press' implies that the item has been accepted for publication.

Reference links

Increased discoverability of research and high quality peer review are ensured by online links to the sources cited. In order to allow us to create links to abstracting and indexing services, such as Scopus, CrossRef and PubMed, please ensure that data provided in the references are correct. Please note that incorrect surnames, journal/book titles, publication year and pagination may prevent link creation. When copying references, please be careful as they may already contain errors. Use of the DOI is encouraged.

Web references

As a minimum, the full URL should be given and the date when the reference was last accessed. Any further information, if known (DOI, author names, dates, reference to a source publication, etc.), should also be given. Web references can be listed separately (e.g., after the reference list) under a different heading if desired, or can be included in the reference list.

References in a special issue

Please ensure that the words 'this issue' are added to any references in the list (and any citations in the text) to other articles in the same Special Issue.

Reference management software

Most Elsevier journals have a standard template available in key reference management packages. This covers packages using the Citation Style Language, such as Mendeley (http://www.mendeley.com/features/reference-manager) and also others like EndNote (http://www.endnote.com/support/enstyles.asp) and Reference Manager (http://refman.com/support/rmstyles.asp). Using plug-ins to word processing packages which are available from the above sites, authors only need to select the appropriate journal template when preparing their article and the list of references and citations to these will be formatted according to the journal style as described in this Guide. The process of including templates in these packages is constantly ongoing. If the journal you are looking for does not have a template available yet, please see the list of sample references and citations provided in this Guide to help you format these according to the journal style. If you manage your research with Mendeley Desktop, you can easily install the reference style for this journal by clicking the link below: http://open.mendeley.com/use-citation-style/dental-materials. When preparing your manuscript, you will then be able to select this style using the Mendeley plugins for Microsoft Word or LibreOffice. For more information about the Citation Style Language, visit http://citationstyles.org.

Reference style

Text: Indicate references by number(s) in square brackets in line with the text. The actual authors can be referred to, but the reference number(s) must always be given. Example: '..... as demonstrated [3,6]. Barnaby and Jones [8] obtained a different result ....'List: Number the references (numbers in square brackets) in the list in the order in which they appear in the text.
Examples:
Reference to a journal publication:
[1] J. van der Geer, J.A.J. Hanraads, R.A. Lupton, The art of writing a scientific article, J. Sci. Commun.163 (2010) 51–59.
Reference to a book:
[2] W. Strunk Jr., E.B. White, The Elements of Style, fourth ed., Longman, New York, 2000.
Reference to a chapter in an edited book:
[3] G.R. Mettam, L.B. Adams, How to prepare an electronic version of your article, in: B.S. Jones, R.Z.

Smith (Eds.), Introduction to the Electronic Age, E-Publishing Inc., New York, 2009, pp. 281–304.
Journal abbreviations source
Journal names should be abbreviated according to the List of Title Word Abbreviations: http://www.issn.org/services/online-services/access-to-the-ltwa/.

Submission checklist
The following list will be useful during the final checking of an article prior to sending it to the journal for review. Please consult this Guide for Authors for further details of any item.
Ensure that the following items are present:
One author has been designated as the corresponding author with contact details:
• E-mail address
• Full postal address
All necessary files have been uploaded, and contain:
• Keywords
• All figure captions
• All tables (including title, description, footnotes)
Further considerations
• Manuscript has been 'spell-checked' and 'grammar-checked'
• References are in the correct format for this journal
• All references mentioned in the Reference list are cited in the text, and vice versa
• Permission has been obtained for use of copyrighted material from other sources (including the Internet)
Printed version of figures (if applicable) in color or black-and-white
• Indicate clearly whether or not color or black-and-white in print is required.
• For reproduction in black-and-white, please supply black-and-white versions of the figures for printing purposes.

For any further information please visit our customer support site at http://support.elsevier.com.