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RETENÇÃO DE COROAS DE ZIRCÔNIA: ESTUDO DE DIFERENTES CONDIÇÕES DE TRATAMENTO INTERNO DE RESTAURAÇÕES EM ZIRCONIA E DO NÚCLEO EM RESINA COMPOSTA

Santa Maria, RS
2017

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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 grau de **Doutor em Ciências Odontológicas**.

Orientador: Prof. Dr. Luiz Felipe Valandro

Santa Maria, RS
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Aprovado em 12 de janeiro de 2017:

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

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Liliana Gressler May, Dra. (UFSM)

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Jovito Adiel Skupien, Dr. (UNIFRA)

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Cesar Dalmolin Bergoli, Dr. (UFPel)

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Marina Amaral, Dra. (UNITAU)

Santa Maria, RS
2017

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O pessimista vê dificuldade em cada oportunidade.
O otimista vê oportunidade em cada dificuldade.

(Winston Churchill)

RESUMO

RETENÇÃO DE COROAS DE ZIRCÔNIA: ESTUDO DE DIFERENTES CONDIÇÕES DE TRATAMENTO INTERNO DE COROAS EM ZIRCONIA E DO NÚCLEO EM RESINA COMPOSTA

AUTOR: Vinícius Felipe Wandscher
ORIENTADOR: Luiz Felipe Valandro

A presente tese foi estruturada em dois artigos científicos que investigaram a adesão de coroas de cerâmicas à base de óxido de zircônio. O primeiro artigo objetivou avaliar diferentes métodos de deposição de sílica na superfície interna de coroas de Y-TZP por meio de teste de retenção. Para isso, 100 preparos simplificados para coroa total produzidos a partir de polímeros reforçados por fibras com foram escaneados e 100 coroas em óxido de zircônio com retenções oclusais foram usinadas. Os conjuntos preparo/coroa foram divididos de forma randômica em 5 grupos (n=20) de acordo com o tratamento da superfície interna: TBS- jateamento com partícula de alumina revestida por sílica (30 µm); GHF1- aplicação de fina camada de glaze + ácido fluorídrico por 1 min; GHF5- aplicação de glaze + ácido fluorídrico por 5 min; GHF15- aplicação de glaze + ácido fluorídrico por 15 min; e Nano- deposição de nanofilme de sílica (5 nm) via desbastamento iônico. Todos os grupos receberam aplicação de um agente de união silano. As superfícies dos preparos foram condicionadas com ácido fluorídrico 10% por 30 s e silanizadas. As coroas foram cimentadas com um cimento resinoso, termocicladas (12.000 ciclos; 5 / 55 °C), armazenadas por 60 dias e submetidas ao teste de retenção (0.5 mm/min até a falha). Os dados de retenção (MPa) foram analisados estatisticamente utilizando-se ANOVA- um fator e teste de Tukey ($p \leq 0,05$), além da análise de Weibull. As falhas foram classificadas em 50C (acima de 50% do cimento aderido na coroa) e 50S (acima de 50% de cimento no substrato). Os grupos TBS (5.6 ± 1.7 MPa) e Nano (5.5 ± 1 MPa) apresentaram maiores valores de retenção do que os demais grupos ($p<0.0001$), assim como maiores valores de resistência característica (6.18 e 5.91, respectivamente). Não houve diferença no módulo de Weibull exceto para o grupo GHF1, que apresentou valor estatisticamente inferior. Os grupos TBS e GHF15 apresentaram, respectivamente, 60 e 70% de suas falhas classificadas como 50C, enquanto os outros grupos apresentaram a maior parte das falhas 50S. O jateamento com partículas de alumina revestidas por sílica e a deposição de nanofilme de sílica na superfície interna de coroas de Y-TZP promoveram maiores valores de retenção. O segundo artigo avaliou a retenção de coroas de zircônia cimentadas em núcleos protéticos envelhecidos construídos em resina composta e preparados com brocas diamantadas de diferentes granulações. Para isso, 60 preparos simplificados para coroa total foram confeccionados em resina composta e escaneados. Sessenta coroas à base de óxido de zircônio com retenções oclusais foram usinadas. Os preparos em resina composta foram armazenados por 120 dias em ambiente úmido a 37 °C e randomicamente divididos em 3 grupos (n=20) de acordo com o tipo de finalização do núcleo em resina: CTRL (controle) – sem tratamento; EFB – broca diamantada extrafina (25 µm); e CB – broca grossa (107 µm). O desgaste superficial foi realizado por meio de um paralelômetro adaptado com o objetivo de padronizar a velocidade e a pressão de desgaste. As superfícies internas das coroas foram jateadas com partículas de alumina revestidas por sílica (30 µm) e então um agente de união silano foi aplicado. As coroas foram cimentadas com um cimento resinoso autoadesivo (RelyX U200). Então, os conjuntos núcleo/coroa foram termociclados (12.000 ciclos; 5/55°C), armazenados por 120 dias e submetidos ao teste de retenção (0.5 mm/min até a falha). Os dados de tração (MPa) foram analisados por meio de ANOVA- um fator e teste de Tukey ($p \leq 0,05$), assim como análise de Weibull. As falhas foram classificadas como 50C (mais que 50% de cimento aderido na coroa), 50S (mais que 50% de cimento aderido ao substrato de resina) e COE (fratura coesiva do núcleo). Nenhuma diferença estatística foi observada nos valores de retenção ($p=0.975$), porém o grupo controle (CTRL) apresentou o maior módulo de Weibull. O tipo de falha predominante foi 50S. A ocorrência de falhas coesivas foi maior no grupo controle. A retenção das coroas à base de óxido de zircônio não foi afetada pela rugosidade do núcleo. Concluiu-se que jateamento seguido de silano e deposição de nanofilmes de sílica aumentam a retenção de coroas de zircônia, assim como o acabamento de núcleos de resina com pontas diamantadas não influencia a retenção de coroas de zircônia.

Palavras-chave: Adesão. Resistência de União. Retenção. Condicionamento. Preparo Protético. Zircônia Monolítica.

ABSTRACT

RETENTION OF ZIRCONIA CROWNS: EVALUATION OF DIFFERENT CONDITIONS OF Y-TZP INTAGLIO SURFACE AND THE COMPOSITE CORE

AUTHOR: Vinícius Felipe Wandscher
ADVISOR: Luiz Felipe Valandro

The present thesis was structured in two scientific articles that investigated the adhesion of zirconium oxide ceramic crowns. The first article aimed to evaluate different methods for silica deposition at the inner surface of Y-TZP crowns by means of a retention test. A hundred simplified full-crown preparations obtained from fiber-reinforced polymer were scanned and 100 zirconium oxide crowns with occlusal retention were machined. The preparation/crown assemblies were randomly divided in 5 groups according to the inner surface treatment: TBS – tribochemical silica coating with silica-coated alumina particles (30 µm); GHF1 – application of a thin glaze layer + hydrofluoric acid for 1 min; GHF5 – glaze application + hydrofluoric acid for 5 min; GHF15 – glaze application + hydrofluoric acid for 15 min; and Nano – silica nanofilm deposition (5 nm) via magnetron sputtering. All groups received silane coupling agent application. The preparations surfaces were etched with 10% hydrofluoric acid for 30 s and silanized. Crowns were cemented with resin cement, thermocycled (12.000 cycles; 5 / 55 °C), stored for 60 days and submitted to retention test (0.5 mm/min until failure). Retention data (MPa) were statistically analyzed by one way– ANOVA and Tukey test, as well as Weibull analysis. Failures were classified as 50C (above 50% of cement in the crown) and 50S (above 50% of cement on the substrate). TBS (5.6 ± 1.7 MPa) and Nano (5.5 ± 1 MPa) groups showed higher retention values than the other groups ($p<0.0001$), as well as higher characteristic strength (6.18 e 5.91, respectively). There was no difference for Weibull modulus, except for GHF1 group, which showed statistically inferior value. TBS and GHF15 presented, respectively, 60% and 70% of the failures classified as 50C, while the other groups had 50S as most part of the failures. Air-abrasion with silica-coated alumina particles and silica nanofilm deposition on the inner surface of Y-TZP crowns generated higher retention values. The second article evaluated the retention of zirconia crowns cemented to aged composite cores and prepared with burs of different grit sizes. Sixty simplified full-crown preparations were fabricated with composite resin and scanned. Sixty zirconium oxide crowns with occlusal retention were machined. The composite resin cores were stored for 120 days in humid environment at 37 °C and randomly divided in 3 groups (n=20) according to the finishing of the composite resin core: CTRL (control) – no treatment; EFB – extra-fine diamond bur (25 µm); and CB – coarse diamond bur (107 µm). Superficial grinding was performed with an adapted surveyor to standardize speed and pressure. The inner surfaces of the crowns were air-abraded with silica-coated alumina particles (30 µm) and a silane coupling agent was applied. Crowns were cemented with a self-adhesive resin cement (RelyX U200). Then, the assemblies core/crown were thermocycled (12.000 cycles; 5/55°C), stored for 120 days and submitted to retention test (0.5 mm/min until failure). Tesile date (MPa were analyzed by one way- ANOVA and Tukey test ($p\leq0,05$), as well as Weibull analysis. Failures were classified as 50C (above 50% of cement adhered in the crown), 50S (above 50% of cement adhered on the resin substrate) and COE (cohesive failure of the core). No statistical difference was observed for the retention values ($p=0.975$), but the control group (CTRL) showed higher Weibull modulus. The predominant failure mode was 50S. The occurrence of cohesive failures was higher on the control group. Retention of the zirconium oxide crowns was not affected by the core roughness. It was concluded that air-abrasion followed by silane application and silica nanofilms deposition improve the retention of zircônia crowns, as well as finishing of resin cores with diamond burs does not influence the retention of zirconia crowns.

Keywords: Adhesion. Bond Strength. Retention. Etching. Prosthetic Preparation. Monolithic Zirconia.

SUMÁRIO

1 INTRODUÇÃO

Historicamente, as restaurações metalo-cerâmicas têm sido consideradas a principal opção restauradora para tratamento com próteses fixas unitárias ou múltiplas. Devido a maior exigência pela estética, aos avanços na tecnologia CAD/CAM (*Computer Assisted Design/Computer Assisted Machining*), assim como pelo desenvolvimento e aprimoramento de materiais cerâmicos mais resistentes, próteses à base de zircônia (Y-TZP) se tornaram uma alternativa às próteses metalo-cerâmicas (DENRY; KELLY, 2014). A zircônia é caracterizada por apresentar três formas cristalinas na natureza: monoclínica (*m* – temperatura ambiente até 1170°C), tetragonal (*t* – 1170°C até 2370°C) e cúbica (*c* – acima de 2370°C) (PICONI; MACCAURO, 1999). A manutenção da fase tetragonal a temperatura ambiente foi alcançada com a inclusão de óxidos estabilizadores como CaO, MgO, Y_2O_3 ou CeO_2 na zircônia pura (DENRY; KELLY, 2008).

Embora superior a outras cerâmicas em termos de desempenho mecânico (tenacidade à fratura, resistência à flexão, comportamento sob fadiga mecânica), existem alguns problemas inerentes associados com a zircônia, como a adesão a cimentos resinosos (BLATZ; SADAN; KERN, 2003; BLATZ et al., 2007), em função de sua microestrutura policristalina densa: a zircônia não é condicionável pelo ácido fluorídrico e requer outros métodos de alteração topográfica (união micromecânica) e/ou ativação química da superfície (LUTHARDT et al., 2002; ZHANG et al., 2004), ou seja, a união físico-química não é efetiva em superfícies de ZrO_2 não tratada (ÖZCAN, KERDIJK, VALANDRO, 2008; ÖZCAN, CURA, VALANDRO, 2011), visto que esta apresenta-se apolar e inerte (BLATZ et al., 2003). Contrariamente, a união às cerâmicas vítreas via união micro-mecânica e química é bem pesquisada e a resistência de união é previsível: uma forte união depende da adesão química entre o cimento e a cerâmica (com a união química do silano) e da micro-retenção mecânica criada pelo ácido fluorídrico na superfície cerâmica (BARGHI et al., 2006; NETO et al., 2015).

A abrasão da superfície de zircônia com partículas de óxido de alumínio (Al_2O_3) ou óxido de alumínio revestido por sílica permite a união micro-mecânica e química entre o agente de união silano (quando partículas de sílica são usadas) e o cimento resinoso. Além disso, a abrasão da superfície da zircônia via ar (jateamento) pode gerar microfraturas e ocasionar uma redução na resistência e falhas prematuras da estrutura (LUTHARDT et al., 2002; ZHANG; LAWN, 2004; ZHANG et al., 2004), muito embora esse efeito deletério longitudinal ainda não seja totalmente conclusivo, uma vez que existem achados contraditórios (SCHERRER et al., 2011; ANAMI et al., 2016; AURELIO et al., 2016; CAMPOS et al., 2016). De qualquer forma, diferentes métodos vêm sendo propostos na literatura como alternativa ao jateamento.

Um dos métodos consiste na aplicação de uma fina camada de porcelana de baixa fusão (material rico em sílica, conhecido como '*glaze*') na superfície de cimentação. Essa técnica tem mostrado um aumento na resistência de união entre o cimentos resinoso e a zircônia (DERAND; MOLIN; KVAM, 2005; KITAYAMA et al., 2009). Uma camada de *glaze* é aplicada e durante a sinterização, esse material se infiltra na camada sub-superficial da zircônia levando à discreta separação dos grãos (alteração em nível nanométrico). Assim, posteriormente é possível fazer a dissolução seletiva/parcial dessa camada vítrea com ácido fluorídrico. Essa técnica tem alcançado uma forte e durável união para materiais à base de zircônia (ABOUSHELIB; KLEVERLAAN; FEILZER, 2008; VANDERLEI; BOTTINO; VALANDRO, 2014). Uma evidente desvantagem da técnica é a dificuldade de padronização da espessura do filme de *glaze* na superfície da zircônia, podendo afetar o assentamento de restaurações – um recente estudo observou aumento do desajuste marginal de restaurações de zirconia que receberam a aplicação de *glaze* internamente, mas o desajuste permaneceu dentro do aceitável clinicamente (VANDERLEI; BOTTINO; VALANDRO, 2014). Na nossa opinião, a influência sobre esse desfecho ainda precisa ser melhor investigada.

Aboushelib, Kleverlaan e Feilzer (2007) propuseram uma outra técnica chamada de condicionamento por infiltração seletiva. Essa técnica consiste na utilização do calor para promover uma tensão térmica na superfície da zircônia, causando um tensionamento dos grãos e permitindo a infiltração de vidro fundido nessa superfície. Essa camada seria então condicionada com ácido fluorídrico, provocando a completa remoção do vidro e criando porosidades que favorecem a retenção micro-mecânica com o cimento resinoso (ABOUSHELIB; KLEVERLAAN; FEILZER, 2007). Essa técnica resultou em um aumento da resistência de união por microtração quando comparada ao jateamento da superfície da zircônia. Casucci et al. (2009) demonstraram que a rugosidade de superfície da zircônia aumentou significativamente após a técnica da infiltração seletiva quando comparada com o jateamento ou condicionamento com ácido fluorídrico.

Outro método para promover alterações da superfície de cerâmicas de zircônia é o tratamento via plasma: o "bombardeio" de partículas altamente energéticas em uma superfície sólida cria radicais livres capazes de aumentar as propriedades adesivas entre dois materiais sem alterar topograficamente a superfície do substrato (QUEIROZ et al., 2013; QUEIROZ et al., 2011). Druck et al. (2014) observaram que a resistência adesiva entre cimento resinoso e a zircônia aumentou com a deposição de nanofilme de SiO_2 (5 nm) ou quando a superfície foi jateada com partículas de alumina-sílica e silanização. Outro tipo de plasma utilizado é o plasma de argônio, o qual é capaz de realizar uma limpeza superficial aumentando a molhabilidade dos agentes de cimentação na superfície da zircônia (CANULLO et al., 2014).

A ativação química por uso de agentes químicos baseados em silano já vem sendo empregado. Uma extremidade da molécula de silano é organicamente funcional (por exemplo, vinil –$CH=CH_2$ e amino –NH_2), e pode polimerizar com uma matriz orgânica (por exemplo, um metacrilato). Já a outra extremidade da molécula é geralmente composta de grupos alcóxis (por exemplo, metoxi−OCH_3, etoxi−OCH_2CH_3), que podem reagir com a superfície da cerâmica. Silanos são comumente usados em Odontologia para revestir/cobrir partículas de vidro em compósitos com matriz polimérica e para alcançar a adesão de porcelanas (ou cerâmicas contendo sílica) aos cimentos resinosos. Estes materiais promovem o molhamento da superfície, aumentando o potencial de retenção micromecânica com cimentos resinosos de baixa viscosidade (MATINLINNA; LASSILA; VALLITTU, 2006; YOSHIDA; TSUO; ATSUTA, 2006).

Ainda nesse sentido, está bem estabelecido na literatura que superfícies ricas em sílica (SiO_2) apresentam-se quimicamente reativas com moléculas bifuncionais (agentes de união, silano). Essas superfícies apresentam a composição química Si−O−Si−OH, enquanto um dos términos moleculares do silano apresenta a terminação −OCH_3. O grupo –OH sofrerá um processo de hidrólise e se unirá com a terminação −OCH_3 do silano, formando a ligação Si−O−Si. A outra extremidade da molécula de silano apresenta uma terminação com uma ligação dupla C=C que será quebrada no processo polimerização com o cimento resinoso (THOMPSON et al., 2011) (Anexo A). No entanto, ressalta-se que os silanos apresentam fraca afinidade química com a zircônia, de tal forma que os silanos não deveriam ser utilizados sobre superfícies de zircônia sem tratamento (KERN, WEGNER, 1998; THOMPSON et al., 2011).

Promotores de adesão (*primer*) e materiais resinosos à base de monômeros fosfatados (MDP) também vem sendo estudados, em especial no que se refere à adesão e cimentação de cerâmicas à base de zircônia. Kern e Wegner (1998) foram os primeiros a relatar a união em longo prazo de cimentos resinosos contendo monômeros fosfatados unidos à zircônia. Os autores compararam a resistência de união à tração de cerâmicas à base de zircônia jateadas e cimentadas com diferentes sistemas (com e sem MDP). Após 150 dias, somente dois cimentos contendo fosfato (Panavia EX e Panavia 21) exibiram alta resistência de união e não mostraram diferença estatística após o envelhecimento artificial. Contrariamente, a literatura mostra-se controversa quando são avaliados *primers* ou cimentos resinosos contendo MDP. Derand e Derand (2000) não encontraram uma forte união à zircônia para cimentos à base de MDP comparado com um cimento resinoso à base de polimetilmetacrilato (PMMA) (Superbond C&B). A dificuldade de adesão à zircônia fica evidenciada em outros estudos (ÖZCAN, KERDIJK, VALANDRO, 2008; ÖZCAN, CURA, VALANDRO, 2011), nos quais a falta de durabilidade da adesão zircônia-cimento foi observada.

Além do contexto da união entre cimento e zircônia, outra dificuldade também pode estar presente para aderir cimento e superfície do preparo protético. Dentes com extensas perdas coronárias geralmente necessitam de retenção intrarradicular seguida de reconstrução com pinos pré-fabricados e resina composta para reter a peça protética (DIETSCHI et al., 2008; SINDEL et al., 1999). No entanto, a retenção de coroas de Y-TZP é maior quando o substrato do preparo protético é dentina em relação a núcleos de resina composta (AMARAL et al., 2014).

Cimentos resinosos e resinas compostas são materiais resinosos que apresentam composições químicas similares. Quando um compósito é usado para confeccionar um núcleo, a superfície da resina composta é completamente polimerizada e exposta à saliva e a agentes temporários de cimentação (TEZVERGIL; LASSILA; VALLITTU, 2003). Assim, trabalhos têm mostrado diversos métodos para aumentar a adesão a essa superfície de compósito polimerizada contaminada (COTES et al., 2015). Os achados parecem ser contraditórios: vários estudos têm demonstrado nenhum efeito do ácido fluorídrico (BROSH et al., 1997; LUCENA-MARTÍN; GONZÁLEZ-LÓPEZ; NAVAJAS-RODRÍGUEZ DE MONDELO, 2001; OZCAN et al., 2005; RODRIGUES et al., 2009), jateamento (BONSTEIN et al., 2005) ou aumento da rugosidade com broca (BOUSCHLICHER; REINHARDT; VARGAS, 1997; SHEN et al., 2004), enquanto outros estudos mostraram um efeito positivo do ácido fluorídrico (TRAJTENBERG; POWERS, 2004; YESILYURT et al., 2009), jateamento (BOUSCHLICHER; REINHARDT; VARGAS, 1997; BRENDEKE; OZCAN 2007; BROSH et al., 1997; LUCENA-MARTÍN; GONZÁLEZ-LÓPEZ; NAVAJAS-RODRÍGUEZ DE MONDELO, 2001; RINASTITI et al., 2010; RODRIGUES et al., 2009; TRAJTENBERG; POWERS, 2004; YESILYURT et al., 2009) ou rugosidade com broca (BONSTEIN et al., 2005; BROSH et al., 1997; SHAHDAD; KENNEDY, 1998; YESILYURT et al., 2009). É importante salientar que todos esses estudos utilizaram testes de adesão simplificados (ensaios de tração/cisalhamento com superfície aderida plana) para testarem as suas hipóteses. Até o momento não há estudos que empregaram testes de retenção de coroas para analisar a interface resina composta/cimento resinoso, ou seja, estudos sobre o efeito combinado de tratamentos de superfície do núcleo em resina composta e da fricção decorrente das paredes do preparo protético.

Estudos utilizando testes de retenção de coroas de zircônia exploraram os efeitos de diferentes estratégias de cimentação e tratamento interno nas coroas (ERNST et al., 2005; PALACIOS et al., 2006; ERNST et al., 2009; SHAHIN, KERN, 2010; AMARAL et al., 2014; EHLERS et al., 2015; RIPPE et al., 2015), tipo de substrato (AMARAL et al., 2014) e envelhecimento (ERNST et al., 2009; EHLERS et al., 2015), e os achados expressam diferentes comportamentos quanto a retenção. Por um lado, Rippe et al. (2015) mostraram que a escolha do cimento foi mais importante que o tratamento de superfície interna da coroa quando o substrato for resina composta. Diferentemente, quando o substrato for dentina, Amaral et al. (2014) mostraram maior efeito positivo da silicatização comparado a resina composta.

Alguns estudos empregaram testes de retenção de coroas para avaliar o efeito da rugosidade do substrato (dente) na retenção de coroas fundidas. Quando diferentes tipos de cimentos foram avaliados (fosfato de zinco, ionômero de vidro e cimento resinoso), o cimento resinoso apresentou maiores valores de retenção associados a uma superfície mais rugosa (TUNTIPRAWON, 1999) ou sem qualquer efeito da rugosidade (AYAD; ROSENSTIEL; SALAMA, 1997). Quando um cimento de fosfato de zinco foi usado, a rugosidade da superfície não afetou a retenção de coroas fundidas (DARVENIZA et al., 1987; SMITH, B. G., 1970) exceto para Felton, Kanoy e White (1987), em que brocas diamantadas foram mais eficientes do que brocas carbide. Quando um cimento de ionômero de vidro foi usado, não foi encontrada diferença nos valores de retenção de coroas fundidas para brocas diamantadas de granulações grossa, média, fina ou extrafina (LI et al., 2012).

Diante dos pressupostos teóricos apresentados acima, especialmente no que tange aos reais efeitos de tratamentos de superfície interna de coroas de zircônia e no núcleo em resina composta sobre a retenção das coroas, a presente Tese teve como objetivos:

- Estudo 1: avaliar a retenção de coroas de zircônia variando o método de deposição de sílica da superfície interna da coroa.

- Estudo 2: avaliar a retenção de coroas de zircônia variando a broca diamantada (extrafina ou grossa) para finalização do preparo.

2 ARTIGO 1 – RETENTIVE STRENGTH OF Y-TZP CROWNS: EFFECT OF SURFACE TREATMENTS OF THE CROWNS INTAGLIO WITH SI-BASED METHODS

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Retentive strength of Y-TZP crowns: effect of surface treatments of the crowns intaglio with Si-based methods.

Short title: Tensile strength of Y-TZP crowns.

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Retentive strength of Y-TZP crowns: comparison of different silica coating methods on the intaglio surfaces of the crowns.

Short title: Retention of Y-TZP crowns

Clinical relevance: Tribochemical silica coating and 5-nm thick silica nanofilms conditioning methods can be used on the inner surface of zirconia crowns for retention improvements.

ABSTRACT

Purpose: To evaluate the effect of different methods of silica deposition on the intaglio surface of Y-TZP crowns on the retentive strength of the crowns.

Methods: One hundred simplified full-crown preparations produced from fiber-reinforced polymer material were scanned and 100 Y-TZP crowns with occlusal retentions were milled. Crown/preparation assemblies were randomly allocated into 5 groups (n=20) according to the treatment of the intaglio surfaces: TBS – tribochemical silica coating via air-abrasion with 30 µm silica coated alumina particles; GHF1 – application of thin glaze layer + hydrofluoric acid (HF) etching for 1 min; GHF5 – glaze application + HF for 5 min; GHF15 – glaze application + HF for 15 min; Nano – silica nanofilm deposition (5 nm) via magnetron sputtering. All groups received a silane application. The surfaces of the preparations (polymer) were conditioned with 10% HF for 30 s and silanized. The crowns were cemented with resin cement, thermocycled (12.000 cycles; 5 / 55 °C), stored for 60 days and submitted to a retentive strength test (0.5 mm/min until failure). The retention data (MPa) were analyzed using 1-way ANOVA and Tukey tests and Weibull analysis. Failures were classified as 50C (above 50% of cement in the crown) and 50S (above 50% of cement on the substrate).

Results: The TBS (5.6 ± 1.7 MPa) and Nano groups (5.5 ± 1 MPa) had higher retentive strength than the other groups ($p<0.0001$) and had the highest values of characteristic strength. There was no difference in Weibull modulus, except for the GHF1 group (lower values). The TBS and GHF15 groups, respectively, had 60% and 70% of their failures classified as 50C, while for the other groups most of the failures were 50S.

Conclusion: Tribochemical silica coating and silica nanofilm deposition on the inner surface of zirconia crowns promoted a higher retentive strength.

Key-words: Retention; Adhesion; Zirconia; Surface treatment; Conditionings, Full-contour; Monolithic.

2.1 INTRODUCTION

Yttrium oxide stabilized zirconia polycrystal (Y-TZP) ceramics have been widely used in dentistry owing to their superior mechanical strength (high fracture toughness)[1,2] and biocompatibility.[3] However, due to their physical, chemical and microstructural features, the adhesion of Y-TZP ceramics with resin cements is difficult.[4-7] Hydrofluoric acid etching and silane application can increase the bond strength between vitreous/glass ceramics (based on silica) and composite resins via increasing of the surface free energy and wettability,[8-13] as well as the ceramic primer heat-treatment can promote bond improvements.[14-16] However, as zirconia ceramics are monolithic and lack a silicon dioxide (silica) phase, hydrofluoric acid treatment fails to produce a micro-porous surface for bonding.[9,17,18] Thus a surface pretreatment is necessary to alter the topography increasing the mechanical retention and the chemical adhesion, thereby enhancing the retention of the crowns to the prosthetic preparation.[19,20] Recently, systematic reviews showed that loss of retention is significantly higher for densely sintered zirconia compared to all others types of ceramics and metal-ceramics, for both single and multiple crowns.[21,22]

The most commonly used treatment method is air-abrasion with silicon oxide particles. This method involves the inclusion/incrustation of silica-coated alumina particles on the cementation surface by air-abrasion, followed by the application of a silane primer bonding agent.[23-27] These particles increase the roughness of the zirconia surface, while the silane bonding agent promotes adhesion between the abraded surface and the resin matrix of the cement.[28-30] However, studies have shown that air-abrasion might create superficial defects and cracks that may be fracture or crack initiators.[31,32] Nonetheless, other studies did not find any negative effects when this method was used on Y-TZP ceramics.[33,34] Additionally, clinical failures in Y-TZP crowns (chippings) seem to have no association with the roughness created by the air-abrasion of the intaglio surface.[35-38]

Another recently developed technique involves the application of a thin layer of vitreous porcelain (low-fusing porcelain glaze – vitrification or glaze-on) on the ceramic surface. Basically, the glaze is composed by vitreous porcelain (high silica content or amorphous matrix – SiO_2) and pigments (metallic oxides), making zirconia glazed surface etchable by hydrofluoric acid,[39,40] and than subject to silanization or air-particle abrasion.[41] Vitrification is very effective in promoting the adhesion between the Y-TZP ceramic surface and resin cements.[39,42-46] For glass ceramics[47-49] hydrofluoric acid reacts with the silica phase of the porcelain, creating retentive microchannels. Therefore, the effect of longer etching times on the creation of surface irregularities for bonding depends on the microstructure of the ceramic.[8,50,51] Etching increases the contact area between the adhesive agent and the ceramic[52,53] and the number and size of the irregularities created is associated with the duration of the etching process.[47,54]. However, when low-fusing porcelain glazes on zirconia surfaces are treated with hydrofluoric acid for too long, the acid etching might completely remove the glaze from the surface. The influence of the etching time of porcelain glaze applied on zirconia on the resin bonding strength and the crown retention rate has yet to be investigated.

Recently, the deposition of silica nanofilms on the zirconia surface has been studied.[26,55] For this method, a SiO_2 nanofilm is deposited on the zirconia surface by plasma processing (reactive magnetron sputtering), making it more chemically reactive. Following-up the nanofilm deposition with silanization results in an increase in adhesive strength without damaging the Y-TZP surface. [26,55,56] Moreover, this technique forms a homogeneous film, improves chemical adhesion to the substratum,[57] and does not promote *m*-phase transformation after the film is applied.[26]

Considering the aforementioned silica deposition methods (low-fusing porcelain glaze application, or nanofilm deposition) for improving the bonding to zirconia, the question is: do these treatments of the intaglio surface of zirconia crowns improve crown retention compared to tribochemical silica coating via air-abrasion? This question has not been addressed yet. Thus, the objectives of this *in vitro* study were as follows: (1) to evaluate the effects of different Silica-based coatings of the intaglio surface of zirconia crowns on retentive strength, (2) to compare 3 different hydrofluoric acid etching times for the groups undergoing the vitrification technique, and (3) to evaluate the reliability of the different treatment methods by Weibull modulus. The null hypothesis tested was that there would be no difference among the zirconia surface conditionings.

2.2 METHODS

2.2.1 Sample size calculation

To determinate the number of specimens for group, a sample size calculation was performed, based on a pilot study, with software from the site "*Java applets for power and sample size*" (www.stat.uiowa.edu/~rlenth/Power/). With a statistic power of 80%, a mean of standard deviation of 1.15 MPa and a detectable difference of 1.38 MPa, it was established that n = 20, for a total of 100 specimens divided amongst 5 experimental groups according to table 1.

Thus, the experimental design was based on 1 factor (surface treatment) divided into 5 levels (groups) (n=20) according to the treatment of the intaglio surface of the Y-TZP crowns (Table 1).

This study was blinded for: crown's cementation, aging procedure, retention test, failure analysis.

2.2.2 Prosthetic preparation and crowns production

G10 bars (G10, FR4 Laminate Round Rods Epoxyglass™; NEMA grade FR4, Accurate Plastics, Inc., New York, NY, USA) of 11 mm in diameter and 1.2 m in length were sectioned in small cylinders of 16 mm in height to obtain 100 identical simplified full crown preparations by computer-aided-machining (6 mm in height and a total occlusal convergence angle of 12° with rounded corners, Figure 1A & 1B).[37,38] Taking into account all the preparations in G10 had the identical dimensions/geometry, just one preparation was impressed using a vinyl polysiloxane impression material (Elite HD + Putty and Light Body Normal Setting, Zhermack, Badia Polesine, Italy), followed by obtaining a model in special plaster (CAM-base, type 4, Dentona AG, Dortmund, Germany).

The master die was then scanned (inEos® Blue, Sirona, Bensheim, Germany) and the image was transferred to the inLab software (Version 3.60, Sirona). Equal crowns (N=100) with occlusal retentions were designed considering a resin cement space of 30 µm, followed by milling the Y-TZP crowns (Cerec InLab MC XL, Sirona) (Figure 2A) (VITA In-Ceram YZ, YZ-40/19 cubes with dimension of 15.5 x 19 x 40 mm^3, VITA, Bad Säckingen, Germany).

Sintering was performed according to the manufacturer's instructions (Zircomat oven, VITA). The crowns were checked in their preparations for adaptation (Carbono Arti-Spray, Bausch, Bausch Articulating Papers, Inc., Nashua, USA) and cleaned with an ultrasonic device (1440 D – Odontrobras, Ind. & Com. Equip. Méd. Odonto. LTDA, Ribeirao Preto, Brazil) with distilled water for 10 min.

2.2.3 Crown cementation

After each treatment of the inner surfaces of the Y-TZP crowns (Table 1), methacryloxypropyltrimethoxy-silane (ESPE-Sil Silane, 3M ESPE, Seefeld, Germany) was applied with a microbrush, with a 5-min wait for solvent evaporation.

The preparation surfaces (polymeric material) were conditioned with 10% hydrofluoric acid for 30 sec, cleaned for the same amount of time, and then received an application of silane based-primer (ESPE-Sil, 3M-ESPE), with a 5-min wait time.[59] The resin cement (RelyX ARC, 3M ESPE) was manipulated according to the manufacturer's instructions and applied to the intaglio surface of the crowns, which were positioned on the preparation. With an adapted surveyor (B2, BioArt, Sao Carlos, SP, Brazil), a load of 750 g was applied to the crown, the cement excess was removed, and photo activation was performed for 20 sec on each surface (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia). The specimens were stored in distilled water (37°C) for 24 h before aging.

2.2.4 Aging: Thermocycling + Storage

All specimens were subjected to thermocycling (12.000 cycles; 5°C-55°C; 30 sec per bath and 2 sec between baths; Ethik Technology, Vargem Grande Paulista, Brazil)[19,20,60,61] followed by storage for 60 days in a wet environment at 37°C.

2.2.5 Retentive strength test

After thermocycling, part of the cemented crown was embedded in self-curing acrylic resin (VIPI Flash, VIPI, Pirassununga, Brazil) until total coverage of the retentive part of the crowns. This process was carried out with the aid of an adapted surveyor (B2, BioArt), which maintained a vertical embedding axis. Retentions were made on the apical part of the preparation and the same embedding procedure was performed, keeping the adhesive interface free to the test. The embedding was necessary to make possible the retention test (Figure 2B).

For the retentive strength test, the superior part of the assembly (the crown) was fixed to the movable axle of a universal testing machine (DL-1000, Emic, São José dos Pinhais, Brazil), which was attached to the load cell (1000 N), while the inferior part (the preparation) was positioned at the fixed base of the testing machine (Figure 2C). The retentive strength test was performed until fracture (decementation) at a speed of 0.5 mm/min.[19,20]

2.2.6 Adhesive area calculation

The cementation area was calculated by the SolidWorks software (DS SolidWorks Corporation, Waltham, USA) according to the measures presented in figure 1A, resulting in an adhesive area of 130 mm^2 (Figure 1B). The retentive strength was calculated using the formula: $R = F_{max}/A$, where R = retentive strength, F_{max} = maximum force for failure (decementation) and A = adhesive area.

2.2.7 Failure analysis

The tested assemblies were analyzed under a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, Germany) to evaluate the type of fracture. The fractures were classified according to the localization of the largest portion of cement: 50C - more than 50% of the cement on the crown or 50S - more than 50% of the cement on the substratum (G10 preparation). Representative images were taken with a scanning electronic microscope (SEM) (JSM 5400, Jeol Ltd, Tokyo, Japan). This classification was adapted from Amaral and others[19] and Rippe and others[20].

2.2.8 Micromorphological analysis (zirconia blocks and G10 polymer surfaces)

Extra zirconia samples (small blocks) were produced from VITA In-Ceram YZ blocks (VITA) in a cutting machine (IsoMet 1000; Buehler, Lake Bluff, USA). The standardization of the analysis surface was performed with Sof-Lex disks (3M ESPE) and polished with 1200-grit sandpaper. After sintering according to the manufacturer's guidelines, the blocks presented an analysis area of 5 x 5 mm, which was conditioned with the aforementioned surface methods.

Etched and non-etched axial surfaces of the G10 preparations (10% hydrofluoric acid for 30 sec) were also analysed.

A surface analysis of the both tested strategies and G10 surfaces were performed under an SEM (JSM 5400, Jeol Ltd) with the aim of verifying the topography and superficial alterations of the Y-TZP ceramic and the G10 preparations.

2.2.9 Data analysis

The nominal values of retentive strength were tabulated and statistical analysis was performed with the SPSS software (Version 21, IBM, Chicago, USA). The normality and homoscedasticity were verified and the data were subjected to 1-way analysis of variance (ANOVA) and post-hoc Tukey tests. A Weibull analysis was also performed (Weibull modulus (m): reliability of retention values – represents the variation of strength data and express the size distribution of the flaw population in a structure; and characteristic strength (σ_0): indicates the strength value at which 63,2% of the specimens survive.

2.3 RESULTS

One-way ANOVA (Table 2) showed a statistical difference between the retention values of the groups ($p<0.0001$). According to Tukey's post-hoc test, TBS and Nano groups presented the highest strength values in relation to the other groups. The GHF15 and GHF5 groups presented intermediate values and the GHF1 group had the lowest values (Table 3). The Weibull modulus was lower for the GHF1 group and the characteristic strength was increased for the TBS and Nano groups.

The Figure 3A, 3B, 3C & 3D shows the micromorphological differences between non-etched and etched surface of the polymer material (G10).

We observed the differences between the surface morphological patterns of the zirconia blocks treated with the different strategies (Figure 4A-M). Compared with the untreated surface (Figures 4L & 4M), all the groups presented topographic alterations, except the Nano group (Figures 4I & 4J). The etching time affected the topographic pattern of the glaze groups (the longer the etching time, the greater the presence of pores) (GHF1 - figures 4A & 4B, GHF5 - figures 4C & 4D and GHF15 - figures 4E & 4F). The TBS group presented irregularities promoted by air-abrasion and the Nano group was similar to the untreated surface.

In terms of fracture type, the TBS and GHF15 groups presented a higher percentage of 50C failures in comparison with the other groups (Table 3, Figure 5A, 5B, 5C, 5D & 5E).

2.4 DISCUSSION

The null hypothesis was rejected; the TBS and Nano groups presented the highest retentive strength values followed by the GHF15, GHF5, and GHF1 groups.

The silica nanofilm application and tribochemical silica coating presented the highest retention and characteristic strength values (Table 3) compared to other silica deposition methods. It has been shown that silica coating via air-abrasion followed by silanization improves the bond strength for silica-based,[9,62] glass-infiltrated alumina[9,63] and zirconium ceramics.[9,23,61,64-66] Using a shear bond strength test, some authors showed that SiO_x nanofilms deposited by magnetron sputtering reached values of adhesion similar to air-abrasion with silica[26] or alumina particles.[56] Despite a similar Weibull modulus (*m*), the Nano group had a 40% higher value of *m* than the TBS group, which suggests a high reliability of the silica deposition technique (Table 3). Compared to the vitrification method, deposition of the silica nanofilm via sputtering is rapid, and the thickness and chemical composition of the film can be also controlled. Also, it does not subject the ceramic to high temperatures and avoids the damage associated with sandblasting. However, it requires costly, specialized equipment and specific training for use.[26]

The Nano group generated mostly 50S (more than 50% of the cement in the G10 substratum) failures (80%) in comparison with the TBS group, which presented 60% 50C (more than 50% of the cement in the crown) failures. According to studies conducted on the magnetron sputtering methods of silica deposition, the coating layer is homogeneous and shows a controlled thickness.[57,58] While the magnetron sputtering creates a 5-nm thick homogeneous silica deposition film (Nano group), the 30-µm diameter silica-coated alumina particles (TBS group) penetrate approximately 15 μm into the Y-TZP crowns.[67] Therefore, despite the fact that both methods promote silica deposition and make the surface more reactive, the air-abrasion caused more irregularities, thus favoring micromechanical retentions between the zirconia and cement. These can be seen on the micrographs (Figures 4G, 4H, 4K & 4L)[28-30] and are the cause of the 50C failure mode. In terms of damage effect of the particle air-abrasion on zirconia materials, literature is controversial.[31-34,68]

Among the studied silica deposition methods, the vitrification method (or glaze-on)[42] seems to be a promising technique. *In vitro* shear bond studies showed that the application of a thin layer of low-fusing porcelain glaze on the zirconia surface followed by hydrofluoric acid etching generated similar or higher adhesion values compared to conventional surface treatment methods. However, these studies conditioned the glazed surface for different durations and used different acid concentrations.[39,42-46]

In the current study, we aimed to determine the best acid etching time of the glazed zirconia surface by means of a crowns' pull-out test, which evaluates not only the bond strength, but also all the complex forces involved, including shear and friction. Our results partially agree with other studies that used a similar methodology (vitrification technique + pull-out crown test).[19,20] Rippe and others[20] verified the effect of the surface treatment and type of cement on the retention of Y-TZP crowns on composite cores. They also showed that the vitrification technique presented higher retention values than the group without treatment, but similar to a tribosilicatization group using a 2-hydroxyl methacrylate (HEMA)-based resin cement. However, when a BIS-GMA-based cement was used, there was no difference between the groups. In addition, Amaral and others[19] evaluated the retention of Y-TZP crowns with different inner treatments and different substrate types and found no difference among the treatments when a dentine substrate was used; however, when the crowns were cemented on composite cores, tribosilicatization and vitrification significantly increased the retention force in comparison with the control group (no treatment). However, in the current study, the GHF1 group (Figure 4A & 4B) presented lower values of retention than the silicatized group (TBS) (Figure 4G & 4H), possibly because the conditioning time (1 min) was not enough to generate significant morphological changes on the glazed surface compared to the air-abraded surface.

Among the studied strategies, the GHF5 and GHF15 groups presented statistically similar and intermediate values of retentive strength (Table 3), as well as characteristic strength. An increase in acid etching time improved the retention values (GHF5 and GHF15 groups) compared to the GHF1 group. However, the GHF15 group showed a higher number of 50C failures, indicating a greater adhesive interaction with the crown, similar only to the TBS group. This could have been caused by the topographic alterations (Figure 4E & 4F) promoted by the increased etching time[49] of the glazed surface and consequently a greater surface area for bonding, which facilitates resin cement penetration into the micro-retentive ceramic surface.[69] The glazing procedure is quick, is performed after the clinical prove being applied by dental technician. Thus the clinician received a zirconia crown with an etchable intaglio surface. Standardization of the thickness of the low-fusing porcelain glaze is difficult, thus increasing the marginal gap.[45] However, space for the glaze and cement layer could be measured out by CAD/CAM systems, thus ensuring an adequate fit.[39] Since the thickness of the glass film was approximately 31.8 µm for Ntala and others[39] and 12 µm for Bottino and others[25], the effect on the marginal fit could be not negligible, considering the clinically recommended maximum misfit is around 120 µm.[45,70,71] Even though the vitrification/glaze-on technique has its advantages, we believe that technical improvements are needed, primarily in the standardization of the thickness of the glaze layer, in order to prevent any negative effects on the marginal fit. It is important to highlight that the glaze composition (monophase) may have affected the retention values for glaze groups. If a multiphasic glaze material had been used, maybe better results of crown retention could have been obtained.[39,40] In this sense, the development of multiphasic glaze materials might achieve result improvements.

In the current study, we utilized a woven glass-fiber-filled epoxy material (G10) as a dentine analogue in order to standardize the substrate and the preparation. This material has showed excellent mechanical and adhesive properties[59,72,73] and we understand that standardization of the substrate is crucial to verify the “pretreatment” factor alone in zirconia crowns. Nevertheless, Kelly and others[59] showed that resin cement bond strength to dentin was slightly lower than to the dry and wet analogue material (40-50%) showing that caution is needed when using the G10 to adhesion tests. Up to now, we unknown others studies that have compared the bond strength of dentin and G10, to demonstrate adhesion similarity of these substrates. In this sense, when adhesion studies using G10 are planned, it is very important to evaluate if the G10 as dentin analogue influence the results and its interpretation. In the current study, we evaluated the zirconia/resin cement interface and the most failures were 50S; thus we believe that it was possible to observe the effect of the zirconia surface treatments. Figure 3A, 3B, 3C & 3D shows that etched G10 surfaces present more glass fiber exposed for bonding with the silane couple agent. Additionally, the use of copings in the complex adhesion/retention trial (retentive strength of crowns) better reflects the clinical reality, especially due to the axial loads exerted during the test.

The present study had some limitations. It is difficult to compare our results with the current literature because most of the studies did not present similar geometries of the preparation. Furthermore, those studies that utilized zirconia crowns had a large number of variables: the substrate, preparation angle, cement type (with or without monomer-phosphate), and application of primers. Mechanical cycling or fatigue experiments were not carried out in these studies either – test conditions should be employed in the future.[72,74-76] These new silica deposition methods should be included in prospective clinical trials to evaluate the its bond effectiveness and the effect on retention of zirconia crowns under clinical situations.

2.5 CONCLUSION

1. Tribochemical silica coating (via particles air-abrasion) and silica nanofilm deposition (via magnetron sputtering) as pre-treatments of zirconia crowns in combination with RelyX ARC luting cement promote higher crown's retention compared to the low-fusing porcelain glaze applications (apart from acid etching time).
2. The application of a thin layer of low-fusing porcelain glaze technique showed variable retention results depending on the etching time, thus further studies about glaze coated polycrystalline zirconium oxide ceramics should be performed.

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2.7. TABLES

Table 1. Testing groups and respective descriptions of the surface conditioning protocols on the intaglio surface.

GROUPS CODE (n=20)	PROTOCOLS
TBS	**Tribochemical silica coating**: air-borne particle abrasion with silica-coated aluminum oxide particles (30 µm) (Cojet Sand – 3M ESPE), 15 mm distant from Y-TZP crown for 10 sec at a pressure of 2.8 bar. An adapted device[23] was used to standardize the procedure.
GHF1*	**Procelain glaze + hydrofluoric acid (1 min)**: a single thin layer of low-fusing porcelain glaze (Glaze VITA Akzent, VITA Zahnfabrik, Bad Säckingen, Germany) was applied with a brush (equal proportions of powder and liquid low-fusion porcelains for each 5 crowns). The crowns were submitted to glaze sintering cycle according to the manufacturer. After, the intaglio surface was conditioned with hydrofluoric acid 10% for 1 min, washed with air-water spray during 1 min and dried. The crowns were cleaned in ultrasound (5 min in distilled water) and dried.
GHF5*	**Procelain glaze + hydrofluoric acid (5 min)**: The procedures were the same as for GHF1, but the surfaces were conditioned for 5 min.
GHF15*	**Procelain glaze + hydrofluoric acid (15min):** The procedures were the same that of GHF1, but the surfaces were conditioned for 15 min.
Nano	**Silica nanofilm deposition:** A 5 nm SiO_2 nanofilm was deposited by magnetron sputtering. Prior to deposition, the atmosphere inside the chamber was pumped down to ~ 10^{-7} Torr base pressure. During deposition, pressure was kept at 5.2 mTorr using a 20 sccm argon flow rate. The nanofilm thickness was determined by the exposition time of the crowns to the deposition plasma, as the deposition rate is known.[55]

* The groups with glaze application were made all in the same time (n=60). As the technique is inherent to difficulty of standardization, the 60 crowns were randomized in the 3 glaze groups after the glaze application and sinterization.

Table 2. One-way ANOVA results for retentive strength.

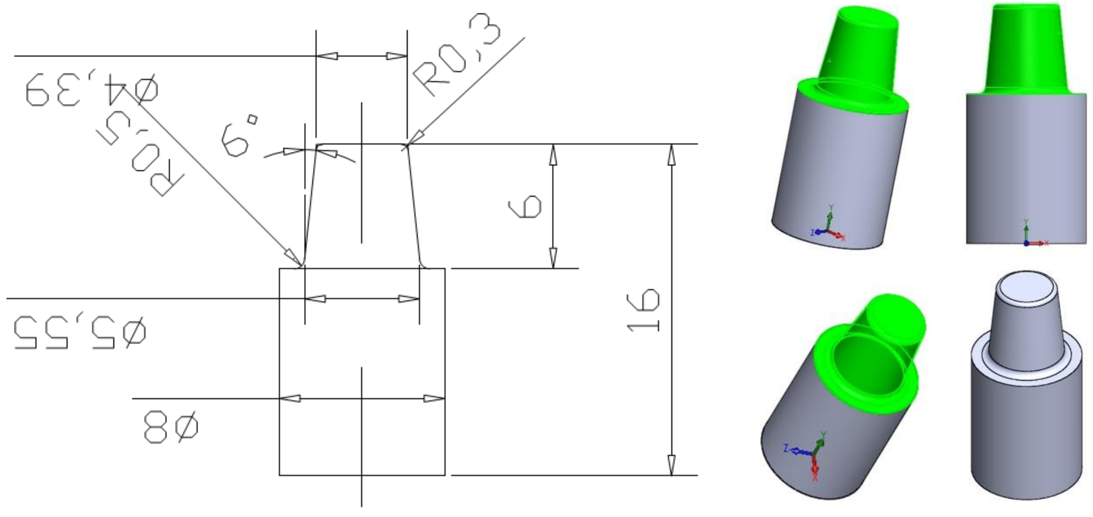
	Sum of squares	df	Mean square	F	Sig.
Between groups	172.203	5	34.441	27.692	.000
Within groups	141.782	114	1.244		
Total	313.985	119			

Table 3. Retentive strength means (MPa) ± standard deviation, Weibull parameters and failure modes of the zirconia crowns.

GROUPS	Retentive strength*	Weibull parameters**				FAILURES***	
		σ_0	CI	*m*	CI	50S (%)	50C (%)
GHF1	2.1 ± 0.95 C	2.55 b	1.76 - 3.66	1.49 B	0.94 - 2	20 (100)	-
GHF5	3.75 ± 1.06 B	4.14 b	3.59 - 4.76	3.86 A	2.44 - 5.19	19 (95)	1 (5)
GHF15	3.78 ± 1.06 B	4.22 b	3.6 - 4.9	3.61 A	2.28 - 4.85	6 (30)	14 (70)
TBS	5.6 ± 1.68 A	6.18 a	5.36 - 7.1	3.9 A	2.46 - 5.24	8 (40)	12 (60)
Nano	5.5 ± 0.98 A	5.91 a	5.43 - 6.41	6.55 A	4.14 - 8.81	16 (80)	4 (20)

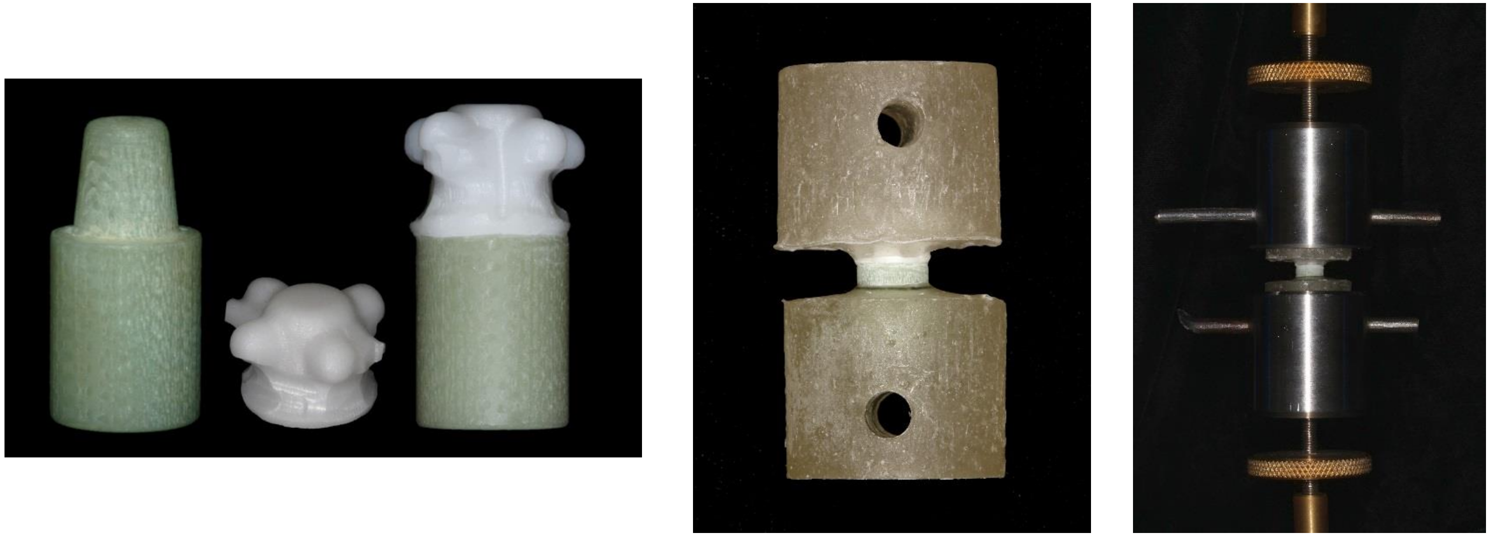
*Different letters in the same column indicate a significant difference.
** Weibull analysis for retention values: σ_0 = characteristic resistance, in MPa, and *m* = Weibull modulus (95% confidence intervals - CI). Lowercase letters were used for σ_0 values and capital letters for *m* values.
*** 50S: more that 50% of cement adhered on the substratum;
50C: more that 50% of cement adhered on the crown.

2.8. FIGURES



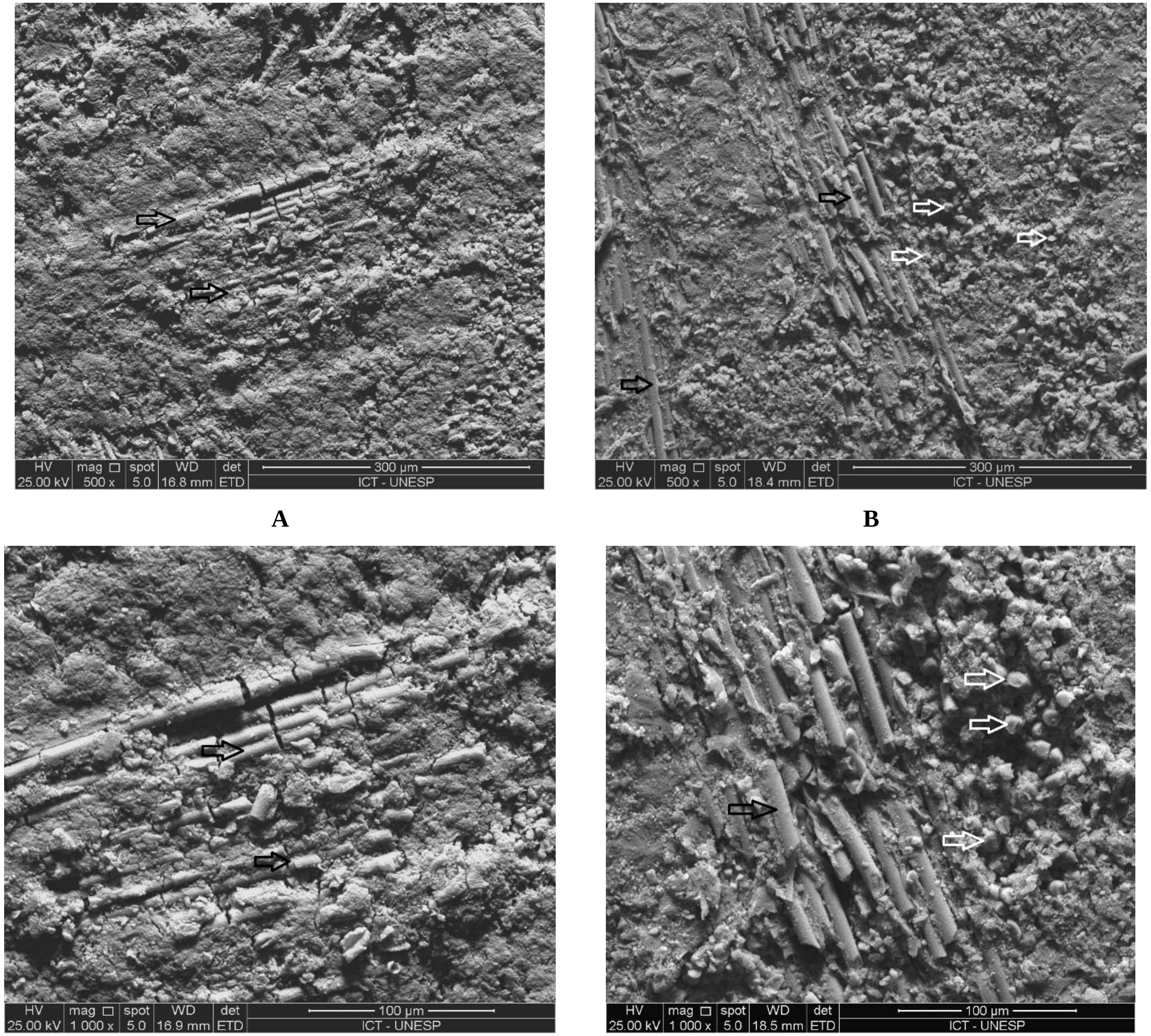
A **B**

Figure 1. A. Schematic drawing of the G10 dies (ø: diameter and R: radius) referents to trunked cone. B. Lateral and frontal views of the G10 die in Solid Works software. The green area corresponds to the adhesive area.



A B C

Figure 2. A. G10 die after milling, zirconia crown with occlusal retentions and cemented crown on the G10 die. B. Specimen embedded for the tensile test. C. Specimen attached on the universal machine test.



C **D**

Figure 3. Representative micrographics of the G10 surfaces with and without hydrofluoric acid etching. A and B, G10 axial surface non etched and etched, respectively (x500). C and D, G10 axial surface non etched and etched (x1000), respectively. G10 analog material presents glass fiber in different senses, it is possible to observe in the non etched surface (A/C) little exposed fiber in only one horizontal direction (black arrow), while in the etched surface presents more exposed glass fiber in different directions (black and white arrows).

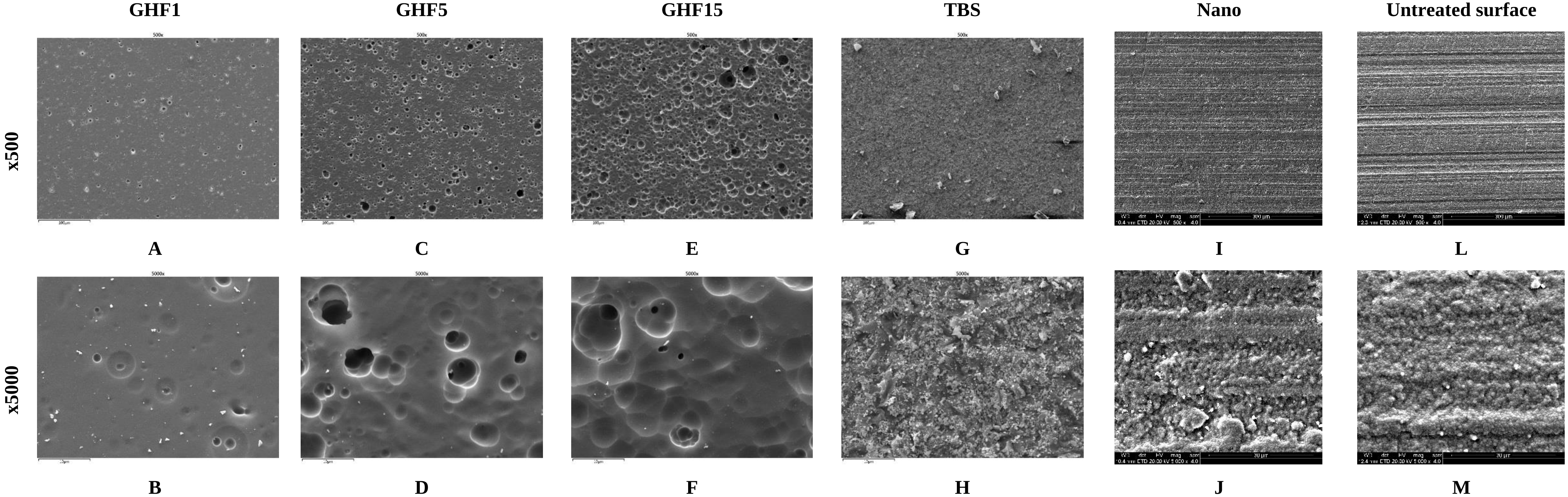


Figure 4. Representative SEM micrographs of the zirconia blocks treated with the same treatments utilized in the intaglio surface of zirconia crowns. GHF1: A (x500) and B (x5000); GHF5: C (x500) and D (x5000); GHF15: E (x500) and F (x5000); TBS: G (x500) and H (x5000); Nano: I (x500) and J (x5000) and zirconia surface without treatment: K(x500) and L (x5000). It is possible to observe in glaze groups greater the etching time, greater the quantity and size of pores, differently to air-abraded surface that presents irregularities promoted by impact of silica coated alumina particles and Nano group presents characteristic of the surface untreated (Sof-Lex disks and 1200-grit sandpaper) possibly because the nanofilm presents a thickness of 5 nm.

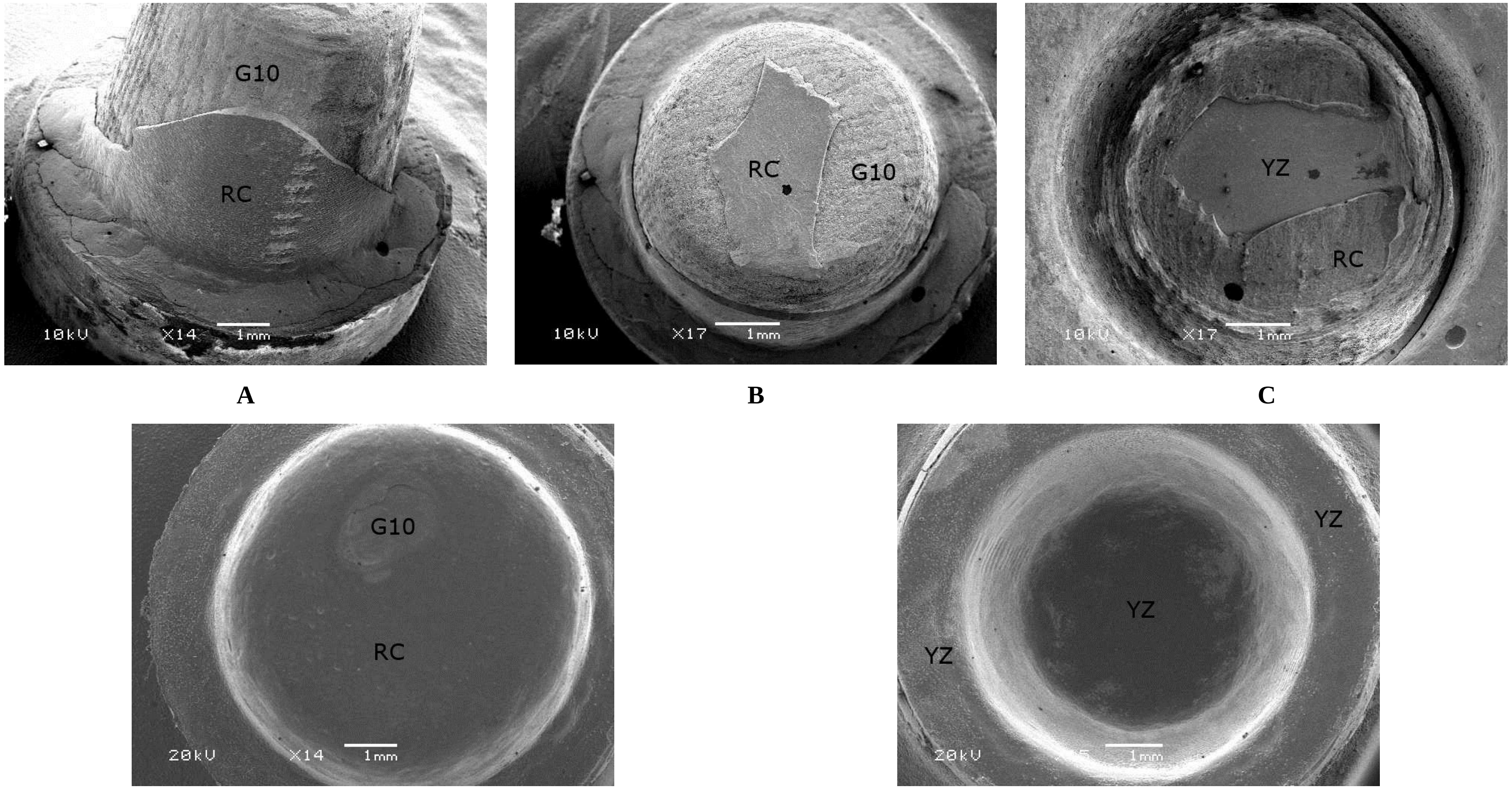


Figure 5. A, B and C: 50C failure of the GH15 group specimen (A and B correspond to G10 die and C to intaglio surface of zirconia crown). D and E: 50S failure of the GH1group specimen (D: G10 die and E: intaglio surface of zirconia crown). G10: G10 analog material; RC: resin cement; YZ: zirconia crown.

3 ARTIGO 2 – GRINDING OF COMPOSITE CORES USING DIAMOND BURS WITH DIFFERENT GRIT SIZES: THE EFFECTS ON THE RETENTIVE STRENGTH OF ZIRCONIA CROWNS

Este artigo foi submetido ao periódico *Journal of Prosthodontics*, ISSN: 1532-849X, Fator de impacto = 1.133; Qualis B1. As normas para publicação estão descritas no Anexo C.

Grinding of composite cores using diamond burs with different grit sizes: the effects on the retentive strength of zirconia crowns

Running title: Effect of grinding on composite cores on the retention of Y-TZP crowns

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Grinding of composite cores using diamond burs with different grit sizes: the effects on the retentive strength of zirconia crowns

Short title: Grinding of composite core and its effect on the retention of Y-TZP crowns

ABSTRACT

Purpose: To evaluate the retentive strength of Y-TZP crowns cemented in aged composite cores finished with burs of different grit sizes.

Materials and Methods: Sixty simplified full-crown preparations were made from composite resin and scanned, while 60 Y-TZP crowns with occlusal retentions were milled. The composite preparations were stored in water at 37°C for 120 days and randomly divided into three groups (n=20 each) according to the type of composite core surface treatment. The groups were defined as follows: CTRL (control: No treatment), EFB (extra-fine diamond bur [25 µm]), and CB (coarse diamond bur [107 µm]). The grinding was performed with an adapted surveyor to standardize both the speed and pressure of the grinding. The intaglio surfaces on the zirconia crowns were air-abraded with silica-coated alumina particles (30 µm) and then received an application of silane coupling agent. The crowns were cemented with self-adhesive resin cement (Relyx U200), thermocycled (12,000 cycles; 5/55°C), stored in wet enrironment at 37°C for 120 days and submitted to a tensile strength test (0.5 mm/min until failure). The retentive strength data (MPa) were analyzed using one-way analysis of variance (ANOVA) and Tukey tests, as well as Weibull analysis. Failures were classified as 50C (above 50% of cement in the crown), 50S (above 50% of cement on the composite core) and COE (composite core cohesive failure).

Results: No statistical difference was observed among the retention values (p=0.975). However, a higher Weibull modulus was observed in the CTRL group. The predominant type of failure was 50S (above 50% of cement on the composite core). The incidence of cohesive failure was highest in the CTRL group.

Conclusion: The retention of zirconia crowns was not affected by different diamond grit sizes burs or when no finishing was performed.

Key-words: Retention; Adhesion; Zirconia; Surface treatment; Conditionings.

3.1. INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics have been increasingly used over the years because of their superior flexural strength, flexural toughness, and phase-transformation toughening mechanism compared with traditional materials.[1] However, in spite of its excellent mechanical properties, zirconia is resistant to acid etching because of its highly crystalline microstructure, hence limiting adhesion to resin materials.[1,2] To improve the bond strength of zirconia to resin cements, tribochemical air-abrasion is commonly employed. This technique uses alumina particles coated with silica to generate micromechanical retention and a reactive surface for silanization.[1]

Although many strategies have been proposed to improve the bond strength of Y-TZP to resin cements, there has been very little focus on the substrate over which the restoration is cemented. Significant loss of coronal tissue is commonly observed in endodontically treated teeth, resulting in the need for post-retained restorations for both aesthetic and functional rehabilitation.[3] These restorations can be performed with prefabricated fiber-post cementation followed by a core build-up with composite resin.[4] Recently, Amaral and others[5] showed that the retention of Y-TZP crowns is higher when cemented to dentin in comparison with composite resin. Preparation for restorations includes core build-up, prosthetic preparation and impression. Following these procedures, composite resin cores with variable conversion grades at their surface, which reduce the potential for their adhesion to the resin cement.[6] During the clinical treatment with prosthetics, dentists will often use provisional restorations in patients before permanent restoration can be performed. During this period, the composite core build-up can be exposed to moisture, variances in pH and temperature[7] and temporary luting cements.[8] As a result, surface alterations of the composite core could be required to improve their adhesion to resin cements and for optimal crown retention.[7]

When a composite core is built up, its external surface is completely cured. The interaction with the surrounding environment may promote water absorption, leading to softening of the matrix, the formation of micro-cracks, resin degradation, debonding of the filler/matrix interface, and leaching of some constituents.[9-11] Some *in vitro* methods, such as thermocycling and water storage for different periods, can be used to simulate the aging process of resin-based composites that occurs *in vivo*.[12-14]

Thus, some studies have proposed that the surface treatment of aged resins could increase their adhesions to fresh resins. Some of these techniques include: grinding with a diamond bur[15-17] or grinding with a diamond bur followed by acid etch/adhesive,[12,18,19] lasers irradiation,[20-24] air abrasion with aluminum oxide particles and silanization,[16,17,19,25], air abrasion with silica-coated alumina particles and silanization,[15,16,25,26] and treatment with hydrofluoric acid.[19,27-29] However, some of these techniques (e.g., air-abrasion and laser) require extra armamentarium in the clinical setting. As a result, the cost of the treatment increases,[30] a rubber dam is required to avoid damage to the patient's periodontium and inhalation of particles,[25] those materials (e.g., silica particles and hydrofluoric acid can be, damaging to patients' health, tooth and soft tissues) are used intra-orally.

In terms of crown retention, the retentive strength of ceramic crowns is associated with tooth preparation, as well as the type of luting material employed.[31,32] Factors such as temperature, exposure to saliva, and mechanical stresses during mastication can influence the longevity of the bond of the zirconia crown-resin cement-dentin complex.[33] In previous studies, when castings were cemented onto the surfaces of teeth using conventional cements (e.g., zinc phosphate), the rough surfaces seemed to influence the retention of crowns.[34,35] Indeed, the retention quality of conventional cements is associated with both their physical strength and the micromechanical retention of the filler particles on the rough surface of the prepared tooth (and not with adhesive quality).[34] However, with advances in adhesive technology for promoting adhesion between different substrates, there could be an increase in interaction between the composites used for core build-up and resin cements.[8]

Until now, no study has evaluated the use of diamond burs with different grit sizes of composite cores for finishing (i.e., grinding) on the retention of Y-TZP crowns cemented with resin-luting cements. Thus, the aim of this study was to evaluate the effect of finishing (i.e., grinding by diamond burs with different grit sizes) of the prosthetic preparation made of composite resin on the retentive strength of zirconia crowns. We tested the hypothesis that grinding with a coarse diamond bur would generate higher retentive strength than grinding with an extra-fine diamond bur.

3.2. METHODS

3.2.1 Composite core prosthetic preparation, aging, and finishing method

Split transparent templates were used to produce sixty composite resin prosthetic preparations (Tetric EvoCeram, Ivoclar Vivadent, Schaan, Liechtenstein) with identical, simplified full-crown preparations (16 mm in total height: 6 mm in preparation height with a total occlusal convergence angle of 12° with rounded corners + 10 mm in base height) (Figure 1A). Small portions of composite resin (2 mm) were inserted incrementally into the templates, until they were filled completely. Next, a screw of 25 mm in height was screwed into the center of the composite base. This screw was used to help with the fixation of the composite preparation sample in the embedding resin for the purpose of a retention test (Figure 1B). Each surface of composite resin preparation was photo-activated for 20 s with a high-power LED (1200 mW/cm^2, RadiiCal, SDI, Bayswater, VIC, Australia) and placed into a vacuum-mirrored polymerization chamber (Visio™ Beta Vario Light Unit, 3M ESPE, Seefeld, BY, Germany) using a specific protocol for photo curing materials (1 min of light followed by 1 min of vacuum and light) to increase the conversion degree. Afterwards, the composite preparations were stored in a bacteriological furnace (wet environment, 37°C) for 120 days.[12-14]

After aging, the composite preparations were assigned to three groups (n=20) according to the grit size of the diamond bur used to finish the surface:

- **CTRL, control:** without roughening the surface,
- **Extra fine bur**: roughening the surface with an extra-fine bur (878EF.314.014 – parallel-chamfer, torpedo – Komet, Brasseler, Lemgo, Germany),
- **Coarse bur**: roughening the surface with a coarse bur (878.314.014 – parallel-chamfer, torpedo – Komet, Brasseler).

To perform the grinding procedures, the composite core preparations were placed in a rotatory mounting of a purposely-built device allowing the core to rotate counter-clockwise around its own axis at a speed of 30 rpm. The diamond burs (Extra-fine and Coarse bur) were installed on an handpiece (Kavo Dental GmbH/Kaltenbach & Voigt GmbH, Biberach an der Riß, BW, Germany) oriented to steadily hold the bur axis parallel to the composite core surface. The bur rotated at 20.000 rpm in the opposite direction (clockwise) of that of the core. The whole rotatory mounting was positioned above a movable X-Y micrometric table: this arrangement allowed a standardized core grinding by setting the cutting depth on a dial caliber (Make, Model, 0.01 mm resolution). A cutting depth pattern of 50 ± 10 µm with a total of three rounds (or revolutions) for each preparation was performed (Figure 2A, 2B, 2C). The abrasion was carried-out under water cooling exclusively on the axial surfaces, preserving the preparation shoulder, chamfer which remained intact. In the manner here described, it was possible to standardize the same geometry and surface type in each composite core specimen.

This study was blinded for: aging procedure, retention test and failure analysis.

3.2.2 Zirconia crowns production, cementation, and aging

Each preparation was scanned (inEos X5, Sirona Dental Systems, Bensheim, Germany) and the images were transferred to the inLab software (Sirona SW 15.0, Sirona). Crowns with occlusal retentions were designed for each preparation and the Y-TZP crowns were milled by a milling machine (Cerec InLab MC XL4, Sirona) (IPS e.max ZirCAD for inLab C-15, dimensions of 14.5 x 15.5 x 18.5 mm^3, Ivoclar Vivadent) with a cement space of 80 µm.

Sintering was produced according to the manufacturer's instructions (Zircomat, VITA Zahnfabrik, Bad Säckingen, Germany). The crowns of each preparation were checked for passive adaptation (Carbono Arti-Spray, Bausch, Bausch Articulating Papers, Inc., Nashua, NH, USA) and cleaned with an ultrasonic device (1440 D – Odontrobras, Ind. & Com. Equip. Méd. Odonto. LTDA, Ribeirao Preto, SP, Brazil) and distilled water for 10 min.

To standardize the procedure, the intaglio surface of Y-TZP crowns were air-abraded with an adapted device using silica-coated aluminum oxide particles (30 µm) (Cojet Sand, 3M ESPE, Seefeld, BY, Germany) at a distance of 15 mm for 10 s and a pressure of 2.8 bar.[36] A coupling agent based on methacryloxypropyltrimethoxysilane (RelyX Ceramic Primer S, 3M ESPE) was applied with a microbrush and crowns were left untouched for 5 min to allow for evaporation of the solvent. Self-adhesive resin cement (RelyX U200, 3M ESPE) was manipulated according to the manufacturer's instructions and applied to the intaglio surface of the crowns, which were positioned on the composite preparation. With an adapted surveyor (B2, BioArt, Sao Carlos, SP, Brazil), a load of 750 g was applied to the crown, the cement excess was removed, and photo-activation was performed for 20 s on each surface (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, VIC, Australia). The specimens were stored in distilled water at 37°C for 24 h, submitted to thermocycling (12.000 cycles; 5°C-55°C; 30 s per bath and 5 s between baths; Ethik Technology, Vargem Grande Paulista, SP, Brazil), and then stored for 120 days in a bacteriological furnace in wet environment at 37°C.

3.2.3 Embedding and retentive strength test

Before a retentive strength test, the specimens were partially embedded inside the acrylic resin to fix the zirconia crown and the composite preparations. The margins of the crown preparations were kept free for testing. First, the crown from the crown/preparation assembly was fixed onto an adapted surveyor perpendicular to the X axis (B2, BioArt) to keep the adequate orientation of the specimen. Subsequently, this preparation allowed the base of the composite preparation to be embedded in self-curing resin (VIPI Flash, VIPI, Pirassununga, SP, Brazil) until 2 mm above the marginal zone. After acrylic-resin polymerization, the previously embedded part was fixed onto the surveyor perpendicular to X axis (for the same aforementioned reason) and the zirconia crown was embedded until the occlusal retentions were covered. Both parts were then embedded using metallic templates with transversal holes that allowed for the attaching of the superior part (crown) and inferior part (composite preparation) in the universal testing machine (DL-1000, Emic, São José dos Pinhais, PR, Brazil). The superior part was fixed to a load cell (1000 N) which was attached to movable axle of the testing machine, while the inferior part was fixed at the fixed base of the machine. Next, a retention force (pull-out) was applied until failure (0.5 mm/min).

3.2.4 Adhesive area calculation

The amount of adhered area (129 mm^2) was calculated by SolidWorks software (DS SolidWorks Corporation, Waltham, MA, USA) according to the measures presented in Figure 1. The retentive strength (R) was calculated using the formula:

$R = F_{max}/A$,

where F_{max} = maximum force for failure (decementation) and A = adhered area.

3.2.5 Failure analysis

To evaluate the type of fracture, the tested assemblies were analyzed under a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, NI, Germany), and the fractures were classified according to the localization of the largest portion of cement. These classifications are described as follows: 50C (more than 50% of the cement on the crown), 50S (more than 50% of the cement on the substratum (composite core preparation)), and COE (cohesive failure of composite preparation) (these data were not included in the statistical analysis). Representative images were taken with a scanning electronic microscope (SEM) (JSM-6360LV, JEOL USA, Inc., Peabody, MA, USA). This classification was adapted from Amaral and others[5] and Rippe and others[7].

3.2.6 Micromorphological analysis

Surface roughness was measured using a 3D optical perfilometer (Leica DCM 3D, Leica Microsystems, Wetzlar Switzerland) with a 20× magnification lens and the images were made with software Leica Map Premium (version 6.2.6266, 2012). The analysis was performed in accordance with ISO 25178.

3.2.7 Data analysis

The retentive strength data were statistically analyzed with the SPSS software (Version 21, IBM, Chicago, IL, USA). Both normality and homoscedasticity were verified, and the data were subjected to one-way analysis of variance (ANOVA) and post-hoc Tukey tests. The reliability of the retentive strength values (m: Weibull modulus) and the characteristic retentive strength (σ_0: strength value at which 63.2% of the specimens survive) were performed by a Weibull analysis.

3.3. RESULTS

A one-way ANOVA showed no statistical difference among the retention values (p=0.975) (Table 1). In addition, no difference in characteristic strength (σ_0) was observed. However, the

Weibull modulus (*m*) was higher in the CTRL group compared with the CB and EFB groups (overlap of confidence intervals). The most common type of failure was 50S (more than 50% of cement adhered to the substratum) (Figure 3).

The perfilometer analysis (Figure 4) showed different surface patterns after grinding with different grit sizes of diamond burs.

3.4. DISCUSSION

The retentive strengths of the three groups were not statistically different (Table 1). Therefore, our formulated research hypothesis was rejected.

Other studies have used bond strength tests with simplified geometry (shear or tensile bond strength on flat surfaces) to evaluate bonding to aged composites. However, these studies have shown conflicting results. In relation to the surface treatment with burs, our results agree with other studies that showed no effect of burs on composite-composite bonding.[37,38] In contrast, Valente and others[39] and Costa and others[17] showed that surface roughening with diamond burs improved the tensile bond strength to new composites. However, these studies used intermediate agents between the aged and fresh composite layers, which could have enhanced the adhesion. Bonstein and others[40] suggested that surface treatment with only a diamond bur on aged composites is simple, efficient, and does not require additional dental materials or instrumentation. Other methods of surface roughening were tested, including sandpapers,[8,25,41] abrasive stone,[12] and pumice,[42] but for these studies the increased bonding is associated with surface grinding, followed by the application of a primer/adhesive. We chose to test burs for their finishing abilities (i.e., surface treatments) because the method is simple, has low cost and is available in the dental office. Notably, we did not apply any intermediate agent since a self-adhesive resin cement was chosen to lute the Y-TZP crowns. This cement does not require any surface treatment, is easy to use, and promotes equivalent bond strength to conventional luting resin cements.[43-45]

Most failures were classified as 50S (more than 50% of cement adhered to substratum) (Table 1). These findings agree with those of both Amaral and others[5] and Rippe and others,[7] who also used composite cores finished with fine diamond burs and observed that failure occurred between the cement and zirconia (adhesive failure). A main explanation for a non-significant result could be the association of resin materials (e.g., composite resin and resin cement) with similar chemical compositions,[8] thus favoring a bond between them. It is possible that the surface produced with an extra-fine bur and coarse bur had no effect on retentive strength because of the similar compositions of resin cement and composite resin. In contrast, other studies demonstrated that the majority of cement was adhered to the intaglios of zirconia crowns after thermocycling.[33,45,46] However, dentin substrate was used in these studies.

In addition, failure analysis showed that the cement remained attached on the internal occlusal surface of the zirconia crown for some samples in the Extra-fine and Course Bur groups (Figure 3). This result possibly occurred because the occlusal surface of the composite core was not prepared and, therefore, the cement remained adhered on ground axial surfaces of the composite. Notably, this result was also observed by Palacios and others.[45] Amaral and others[5] and Rippe and others[7] did not evaluate the roughness of the composite core on zirconia crown retention. However, both of these studies presented failure patterns similar to those observed in our study, which used composite cores and resin cements. Hence, independently of surface treatment, factors including the type of substratum, resin cement, and taper preparation can be more important than surface roughness in influencing the retention of zirconia crowns.

Taper preparation is another factor that could have affected the retention values. Kaufman and others[47] examined the effect of variation of the convergence angle (1°, 5°, 10°, 15° and 20°) on crown retention and showed that retention increases as the convergence angle decreases. In our study, we utilized the total convergence angle of 12° and, consequently, the retentive effect may have been higher than both bonding and roughness effects. This convergence angle could have contributed to cohesive failure as observed by this study, Amaral and others,[5] and Rippe and others.[7]

Despite the similar retention strengths depicted by our findings, the Weibull modulus of control group (CTRL) was higher (higher reliability) than the EFB and CB groups (Table 1). Ayad and others[48] stated that excessive roughness could lead to trapped air between the cement and tooth preparation, which could cause adhesive failure; this event could have occurred in the current study. Furthermore, the standard deviation of the control group was lower than in the EFB and CB groups, possibly due to the fact that the procedure could have promoted heterogeneous morphological surface patterns on the treated cores (Figure 4).

There were some limitations of our study. First, unreal retention values were generated with cohesive failures of the composite cores. As a result, these data were removed from the statistical analysis to avoid overestimation or underestimation of the retention values. In a prior study, cohesive failure occurred before reaching the maximum load supported by adhesive interfaces.[45] In the current study, cohesive failures varied from 15% to 30%, depending on the group (Table 1). It is important to emphasize that if this type of failure had not occurred, the retention values would probably be higher. These failures may be associated with taper preparation — if the convergence angle had been greater, maybe the cohesive failures would not have occurred. Secondly, composite preparations were created using highly standardized procedures. In clinical practice, however, dental tissue will be present at the chamfer preparation when restoring endodontically treated teeth with posts and composite cores. Therefore, it is difficult to compare our results with those in the current literature. Indeed, most studies employ different methodologies including varying geometries of the preparation, as well as different taper preparations, resin cements, and substrates. Further studies should be performed with other types of aging, composite-core surface treatments, luting cements (e.g., zinc phosphate, glass ionomer, resin modified glass ionomer, and resin cement of different compositions), adhesive techniques, and taper preparations. Finally, tests applying intermittent loading and fatigue investigations should also be conducted.[49-54]

3.5. CONCLUSION

The retention of zirconia crowns cemented with self-adhesive resin cement was not affected by grinding using diamond burs with different grit sizes on composite resin preparations with a convergence angle of 12º.

3.6. REFERENCES

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3.7. TABLES

Table 1. Means (standard deviation) of the tensile strength (MPa), Weibull analysis (*m*= modulus; σ_0= characteristic tensile resistance (MPa); IC= confidence interval), and percentage of failure types.

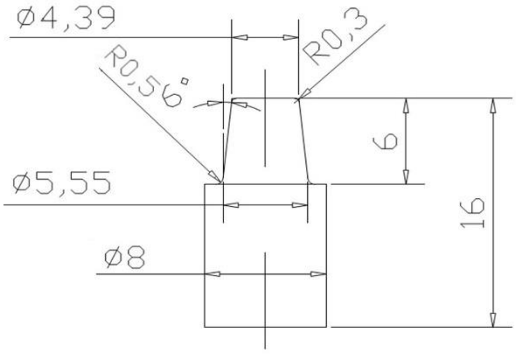
Groups	Tensile Strength*	Weibull Parameters**				Failures***		
		m	IC	σ_0	IC	50C (%)	50S (%)	COE (%)
No-treatment (control)	2.1 (0.41)[a]	5.9[a]	3.3 – 8.3	3.4[a]	3 – 3.8	5 (25)	9 (45)	6 (30)
Coarse diamond bur	2.03 (1.03)[a]	2.1[b]	1.2 – 2.9	3.3[a]	2.4 – 4.5	-	15 (75)	5 (25)
Extra-fine diamond bur	2.04 (0.97)[a]	2.2[b]	1.3 – 3	3.3[a]	2.5 – 4.5	1 (5)	16 (80)	3 (15)

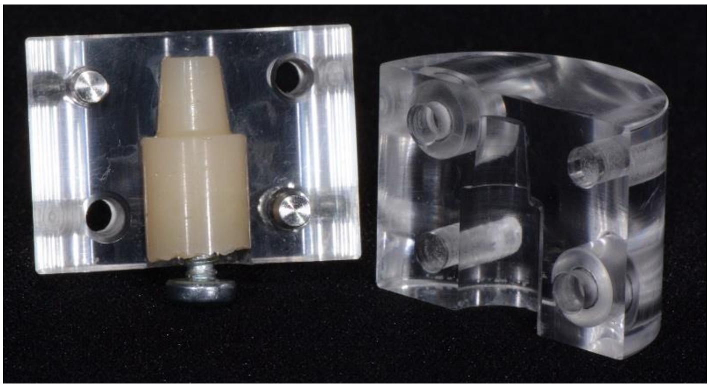
*Different lowercase letters in the same column indicate a significant difference.

** Different lowercase letters in the same column indicate a significant difference (no overlap of the confidence intervals)

*** 50S: more that 50% of cement adhered on the substratum;
50C: more that 50% of cement adhered on the crown.
COE: cohesive failure: composite die fracture.

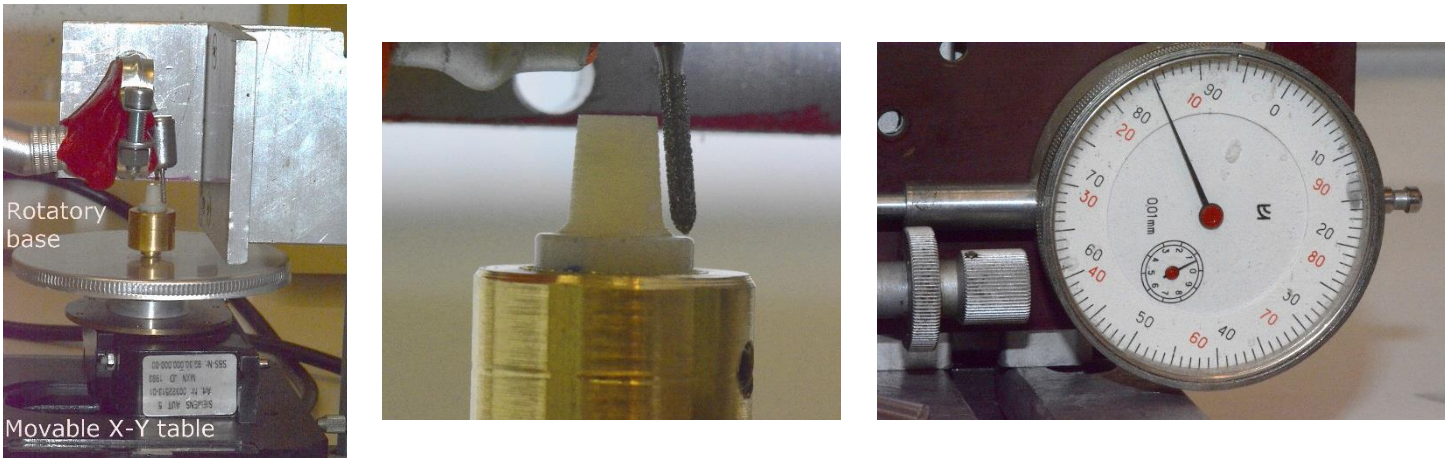
3.8. FIGURES





A **B**

Figure 1. A. Schematic drawing of the composite preparations (ø: diameter and R: radius) referent to trunked cone. B. Split transparent templates used to produce the composite cores (note the screw on the composite base center).



A **B** **C**

Figure 2. A. Special device for grinding the composite preparations. B. Composite core after grinding (bur positioned parallel to the composite surface). C. Micrometer installed onto a movable X-Y table to standardize the grinding pressure.

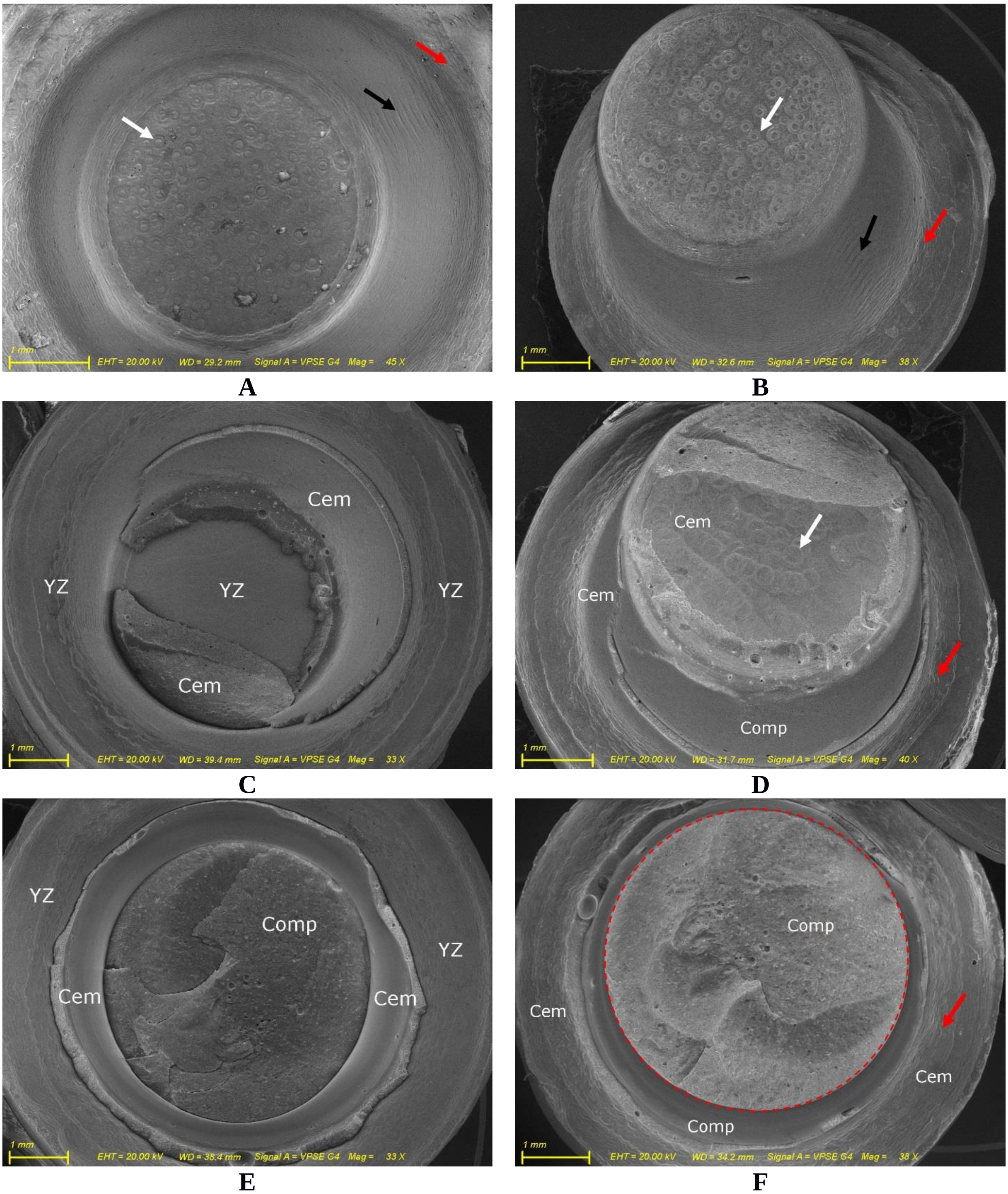


Figure 3. Representative scanning electric microscopies. A. Zirconia crown of the Coarse bur group (white arrow: circular machining marks on the internal occlusal surface; black arrow: axial machining marks on the axial internal surface; red arrow: semicircular machining marks on crown shoulder). B. Composite core of the Coarse bur group (it is possible to note that the resin cement layer is adhered totally on the core and the machining marks are reproduced on the cement layer) – 50S failure. C. Zirconia crown of the CTRL group (YZ: zirconia and Cem: cement) – cement partially adhered on crown. D. Composite core of the CTRL group (major part of cement adhered on composite surface) and machining marks on the layer cement – 50S failure. E. Zirconia crown of the Extra fine bur group – Cohesive failure (part of the core fracture into crown. F. Composite core of the Extra fine group (red circle: fractured occlusal third; cement adhered on the composite shoulder with semicircular machining marks).

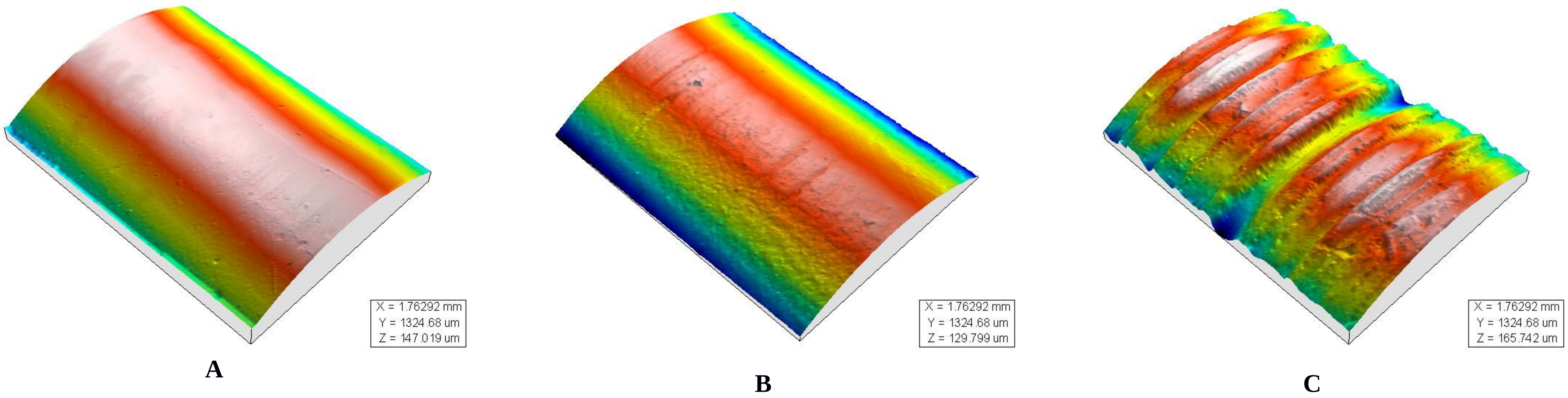


Figure 4. 3D surface topographies of experimental groups. A. Surface and roughness parameters to CTRL group, B. Surface and roughness parameters to extra fine bur group and C. Surface and roughness parameters to coarse bur group.

4 DISCUSSÃO

Vários ensaios clínicos relatam perda de retenção ou descimentação de próteses fixas de cerâmicas à base de zircônia (BEUER et al., 2009; BÖMICKE et al., 2016; GÜNCÜ et al., 2015; SAILER et al., 2007; SOLÁ-RUÍZ et al., 2015; TARTAGLIA; SIDOTI; SFORZA, 2015; TINSCHERT et al., 2008), indicando que estudos deveriam ser conduzidos para otimizar a retenção das restaurações. Em revisão sistemática sobre sobrevivência e complicações de próteses fixas, três estudos prospectivos foram incluídos, os quais avaliaram próteses parciais fixas totalmente cerâmicas com infraestrutura de zircônia e relataram a perda de retenção (SAILER et al., 2007). Outras duas revisões sistemáticas mostraram que a incidência de perda de retenção é significativamente maior para coroas unitárias e pontes fixas de zircônia densamente sinterizada em relação a próteses totalmente cerâmicas confeccionadas com outros materiais cerâmicos e metalo-cerâmicas (PJETURSSON et al., 2015; SAILER et al., 2015).

Em relação aos nossos achados, o primeiro artigo observou que os tratamentos das superfícies internas de coroas em zircônia com o jateamento com partículas de alumina revestidas por sílica (30 µm) ou com deposição de nanofilmes de sílica (5 nm) via plasma, ambos seguidos de silanização, promoveram os mais altos valores de retenção das coroas.

O jateamento com partículas de alumina revestidas por sílica tem sido amplamente estudado na literatura, mostrando altos valores de resistência de união (ZHANG et al., 2004; VALANDRO et al., 2006; THOMPSON et al., 2011; DRUCK et al., 2015). Por meio de alta pressão, as partículas de alumina revestidas por sílica atingem a superfície de zircônia promovendo um aumento da temperatura da superfície e transferindo a sílica para a superfície cerâmica (triboplasma). Assim, a superfície permanece rica em sílica e adequada para receber um agente de união silano (Thompson et al., 2011).

No que diz respeito a deposição de nanofilme de sílica como promotor de adesão, nós encontramos resultados promissores, à medida que esse método proporcionou retenção de coroas de zircônia similar ao método de silicatização. O processo de deposição consiste em depositar um filme de sílica muito fino sobre a superfície da zircônia, tornando esta camada superficial rica em sílica e adequada para receber um agente de união silano (DRUCK et al., 2015). Entretanto, entendemos que esses achados promissores devem ser vistos com relativa cautela, à medida que se trata de um método recente na Odontologia (DRUCK et al., 2015; QUEIROZ et al., 2011). Alguns aspectos precisam ser melhor elucidados: avaliar os parâmetros e a sistematização da deposição de sílica, assim como, a durabilidade da adesão, seja pela longa armazenagem em água, seja pela realização de testes em fadiga.

Comparativamente, deve-se ressaltar que a deposição de nanofilme evita que danos sejam incorporados na superfície da zircônia; ao contrário, a abrasão por partículas de alumina/sílica gera inclusão de danos e transformação de fase de tetragonal para monoclínica, muito embora recentes estudos tenham demonstrado que o jateamento com partículas não altera negativamente o comportamento sob carga estática (AURELIO et al., 2016) e em fadiga desse material (SCHERRER et al., 2011; ANAMI et al., 2016; CAMPOS et al., 2016). Nesse sentido, é importante acompanhar novos achados, para que os pesquisadores possam produzir observações mais conclusivas sobre o efeito longitudinal de superfícies com danos provocados por tratamentos de superfície interna de coroas, especialmente achados provenientes de análises fractográficas de superfícies fraturadas para a determinação da origem da falhas e possível relação causa-efeito (THOMPSON et al., 1994; QUINN et al., 2005; SCHERRER et al., 2006; QUINN et al., 2007; SCHERRER et al., 2007; SCHERRER et al., 2008; TASKONAK et al., 2008; LOHBAUER et al., 2010).

O segundo artigo encontrou que os desgastes com pontas diamantadas do núcleo em resina composta (acabamento dos preparos) não afetaram a retenção de coroas em zircônia cimentadas com um cimento resinoso autoadesivo. Além disso, o grupo sem acabamento apresentou um módulo de Weibull estatisticamente superior aos demais grupos (brocas grossa e extrafina), demonstrando que a confiabilidade nos valores de retenção foi maior quando nenhum processo de acabamento do preparo foi realizado. A inclusão de defeitos por desgaste com broca da superfície da resina composta pode ser considerada uma importante fonte de variação dos dados de retenção. Nota-se que o desvio-padrão dos dados dos grupos em que as brocas foram utilizadas foi maior. Isso pode estar associado ao fato do cimento autoadesivo não preencher adequadamente as irregularidades criadas no preparo. Esses resultados poderiam ser diferentes caso um agente intermediário fosse usado.

Alguns estudos mostram que quando um acabamento da superfície de um compósito com brocas diamantadas é realizado e um agente adesivo intermediário é utilizado, a resistência adesiva a um novo compósito é maior (COSTA et al., 2010; VALENTE et al., 2015). O uso de adesivos promove a adesão química entre a camada de compósito já polimerizada/acabada e o novo compósito (SWIFT, CLOE, BOYER, 1994; COSTA et al., 2010). Agentes de união silano ou adesivos aumentam a molhabilidade da superfície ao novo incremento de compósito (HISAMATSU, ATSUTA, MATSUMURA, 2002). Alguns estudos sugerem que primordialmente o clínico deva conhecer a composição da resina utilizada a ponto de selecionar o adequado agente intermediário (LOOMANS et al., 2011a; LOOMANS et al., 2011b; LOOMANS, OZCAN, 2016). Sendo assim, se um cimento resinoso convencional fosse usado associado a um agente intermediário, os resultados poderiam ter sido diferentes.

Outras opções de tratamento poderiam ter sido usadas como: jateamento com partículas de óxido de alumínio ou óxido de alumínio revestido por sílica, ou ainda o condicionamento com ácido fluorídrico ou fosfórico. Em relação ao jateamento, os resultados da literatura mostram resultados conflitantes com (BOUSCHLICHER, REINHARDT, VARGAS, 1997; RODRIQUES et al., 2009; RINASTITI et al., 2010) ou nenhum efeito (BONSTEIN et al., 2005). O mesmo se repete quando o ácido fluorídrico foi utilizado, apresentando efeito positivo (TRAJTENBERG; POWERS, 2004; YESILYURT et al., 2009) ou não (LUCENA-MARTÍN; GONZÁLEZ-LÓPEZ; NAVAJAS-RODRIGUES DE MONDELO, 2001; OZCAN et al., 2005). Em relação a esses métodos, grandes desvantagens são apresentadas, como por exemplo o risco de danos ao periodonto e inalação das partículas no caso de jateamento (COTES et al., 2015) e no caso de ácido fluorídrico, o risco de corrosão dos tecidos devido a sua toxicidade. Outra alternativa muito comum e com bom custo-benefício é o ácido fosfórico, o qual promove uma limpeza superficial (LOOMANS et al., 2011), porém nenhuma alteração topográfica superficial (CESAR et al., 2001).

A aplicação de lazer também apresenta-se como alternativa (BURNETT, SHINKAI, EDUARDO, 2004; POLAT et al., 2015; KIMYAI et al., 2010;), entretanto é de alto custo, tanto operacional ao operador quanto ao paciente.

É importante salientar que todos esses métodos de acabamento foram testados por meio de testes de adesão simplificados. Novos estudos são sugeridos utilizando geometrias complexas, como testes de retenção de coroas, assim como estudos de fadiga em longo prazo.

Em relação ao acabamento do preparo protético ou então a rugosidade do núcleo, a literatura apresenta alguns estudos, porém utilizando coroas fundidas, estrutura dental como substrato e cimentos diferenciados. Um estudo encontrou aumento da retenção de coroas metálicas com acabamento com brocas diamantadas em relação a brocas carbide usando cimento de fosfato de zinco (FELTON; KANOY; WHITE, 1987). Outro estudo mostrou diferença estatisticamente superior na retenção de coroas metálicas cimentadas a dentes humanos preparados com brocas grossas em relação a brocas finas usando cimento resinoso, de ionômero de vidro e fosfato de zinco (TUNTIPRAWON, 1999). Ambos os estudos mostram achados diferentes aos do segundo artigo, possivelmente por terem usado coroas metálicas e estrutura dental como substrato, sendo que nesses casos a rugosidade apresenta impacto para a retenção. Além disso, cimentos resinosos e resinas compostas apresentam composição semelhante (TEZVERGIL; LASSILA; VALLITTU, 2003), o que pode estar relacionado à ausência de diferença estatística no segundo artigo. No entanto, é possível que se outros cimentos (ionômero de vidro e fosfato de zinco) tivessem sido utilizados, os resultados do segundo artigo desta tese poderiam ser diferentes.

Clinicamente, quando um núcleo protético em resina composta é construído, desgastes e acabamentos serão em geral necessários, assim diferentes alterações topográficas das superfícies de resina podem ser alcançadas, à medida que diversos tipos de instrumentos rotatórios podem ser utilizados. Nesse aspecto, o segundo artigo mostrou que o acabamento com pontas diamantadas do preparo em resina não tem efeito sobre a retenção das coroas de zircônia, quando estas são cimentadas com um cimento resinoso autoadesivo. Esse achado parece demonstrar que as características do preparo protético se configuram como um fator relevante influenciando a retenção, mais que as alterações de superfície do preparo.

Heintze, (2010) em uma revisão sistemática avaliou a influência de fatores que afetam testes laboratoriais que investigam a efetividade de agentes de cimentação na retenção de coroas. Segundo o autor, um estudo *in vitro* racional deve incluir: ao menos 20 espécimes por grupo, altura de preparo de 3 mm, termociclagem dos espécimes (5000 ciclos), evitar forças de cisalhamento durante o deslocamento e executar uma estatística de probabilidade de falha (Weibull). Dentro disso, entendemos que os dois estudos apresentados foram executados considerando os parâmetros descritos.

Por fim, deve-se ponderar que no ambiente oral as restaurações são submetidas a cargas mecânicas mastigatórias na presença de umidade (GONZAGA et al, 2011), levando ao acúmulo de tensões e crescimento de trincas tanto em cerâmicas quanto em materiais resinosos (SURESH, 1998). Considerando-se que os ensaios de fadiga simulam de forma mais próxima o que ocorre clinicamente, são necessários estudos avaliando os fatores estudados em ambos os trabalhos com testes de fadiga. Ensaios clínicos também deveriam ser iniciados avaliando as hipóteses testadas nesses estudos *in vitro*.

5 CONCLUSÃO

- Artigo 1: o jateamento com partículas de alumina revestidas por sílica e a deposição de nanofilme de sílica como tratamentos da superfície interna de coroas em zirconia representam alternativas favoráveis para aumentar a retenção de coroas de zircônia.

- Artigo 2: o acabamento de núcleos de resina composta com brocas diamantadas não afetam a retenção de coroas de zircônia.

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ANEXO A – IMAGEM DO PROCESSO DE UNIÃO ENTRE UMA SUPERFÍCIE RICA EM SÍLICA E UM CIMENTO RESINOSO

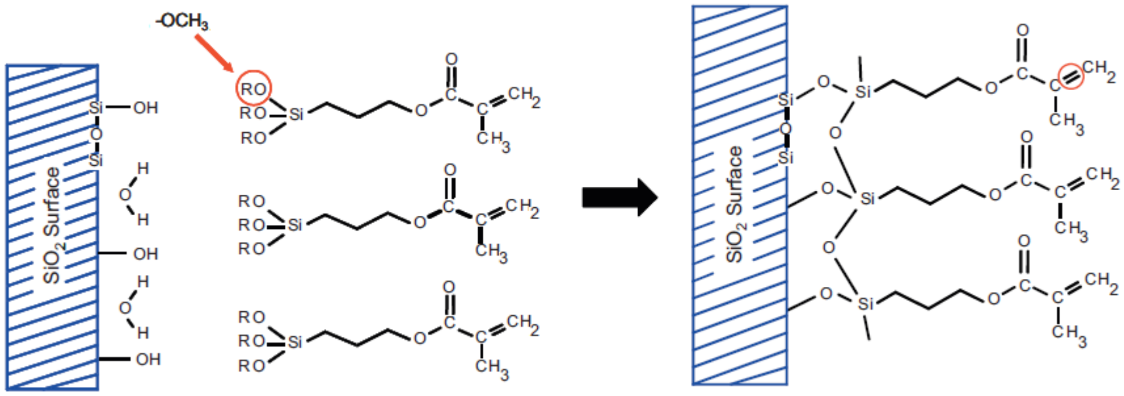

Ilustração idealizada de como ocorre a união entre a sílica inorgânica da superfície com a sílica presente na molécula de silano (lado esquerdo da figura) e a polimerização com a matriz orgânica (lado direito da figura – a ligação dupla circulada em vermelho seria quebrada durante um processo de polimerização conduzida pelo radical livre, facilitando a união covalente com a matriz orgânica do agente resinoso) (THOMPSON et al., 2011).

ANEXO B – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *OPERATIVE DENTISTRY*

GUIDE FOR AUTHORS

Manuscript submission

General Requirements

Operative Dentistry requires electronic submission of all manuscripts. All submissions must be sent to Operative Dentistry using the Allen Track upload site. A mandatory and nonrefundable $25.00 fee is required at submission. Your manuscript will only be considered officially submitted after it has been approved through our initial quality control check, and any quality problems have been resolved. You will have 6 days from when you start the process to submit and approve the manuscript. After the 6 day limit, if you have not finished the submission, your submission may be removed from the server. You are still able to submit the manuscript, but you must start from the beginning. Be prepared to submit the following manuscript files in your upload:

• A Laboratory or Clinical Research Manuscript file must include:

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- a running (short) title
- a clinical relevance statement o a concise summary (abstract) 14 Current as of: 3-Sep-14
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- references (see Below)

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- Author identifying information such as:
  - Authors names or titles
  - Acknowledgements
  - Correspondence information
  - Response to reviewer files should also NOT include any author identifying information, such as a signature at the end, etc.
- Figures
- Graphs
- Tables

• An acknowledgement, disclaimer and/or recognition of support (if applicable) must in a separate file and uploaded as supplemental material.

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All Manuscripts

CORRESPONDING AUTHOR must provide a WORKING / VALID e-mail address which will be used for all communication with the journal. NOTE: Corresponding authors MUST update their profile if their e-mail or postal address changes. If we cannot contact authors within seven days, their manuscript will be removed from our publication queue.

AUTHOR INFORMATION must include:

• full name of all authors

• complete mailing address for each author

• valid email address for each author • degrees (e.g. DDS, DMD, PhD)

• affiliation (e.g. Department of Dental Materials, School of Dentistry, University of Michigan)

MENTION OF COMMERCIAL PRODUCTS/EQUIPMENT must include:

• full name of product • full name of manufacturer

• city, state and country of manufacturer

MANUSCRIPTS must be provided as Word for Windows files. Files with the .doc and .docx extensions are accepted.

TABLES may be submitted as either Word (.doc and .docx) or Excel (.xls and .xlsx) files. All tables must be legible, with fonts being no smaller than 7 points. Tables have the following size limitations: In profile view a table must be no larger than 7 x 9 inches; landscape tables should be no wider than 7 inches. It is the Editor’s preference that tables not need to be rotated in order to be printed, as it interrupts the reader’s flow.

ILLUSTRATIONS, GRAPHS AND FIGURES must be provided as TIFF or high resolution JPEG files with the following parameters:

• line art (and tables that are submitted as a graphic) must be sized with the short edge being no shorter than 5 inches. It should have a minimum resolution of 600 dpi and a maximum resolution of 17 Current as of: 3-Sep-14 1200 dpi. This means the shortest side should be no smaller than 3000 pixels.

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CLINICAL TECHNIQUE/CASE STUDY MANUSCRIPTS must include as part of the narrative:

- a running (short) title
- purpose
- description of technique
- list of materials used
- potential problems
- summary of advantages and disadvantages
- references (see below)

LITERATURE AND BOOK REVIEW MANUSCRIPTS must include as part of the narrative:

- a running (short) title
- a clinical relevance statement based on the conclusions of the review
- conclusions based on the literature review…without this, the review is just an exercise and will not be published
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References

REFERENCES must be numbered (superscripted numbers) consecutively as they appear in the text and, where applicable, they should appear after punctuation. The reference list should be arranged in numeric sequence at the end of the manuscript and should include:

1. Author(s) last name(s) and initial (ALL AUTHORS must be listed) followed by the date of publication in parentheses.

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3. Full journal name in italics (no abbreviations), volume and issue numbers and first and last page numbers complete (i.e. 163-168 NOT attenuated 163-68).

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8. DO NOT include unpublished data or personal communications in the reference list. Cite such references parenthetically in the text and include a date.

9. References that contain Crossref.org's DOIs (Digital Object Identifiers) should always be displayed at the end of the reference as permanent URLs. The prefix http://dx.doi.org/ can be appended to the listed DOI to create this URL. i.e. http://dx.doi.org/10.1006/jmbi.1995.0238

Reference Style Guide

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

- Journal article-multiple authors: Eick JD, Gwinnett AJ, Pashley DH & 19 Current as of: 3-Sep-14 Robinson SJ (1997) Current concepts on adhesion to dentin Critical Review of Oral and Biological Medicine 8(3) 306-335.

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

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• Journal Article with DOI: SA Feierabend, J Matt & B Klaiber (2011) A Comparison of Conventional and New Rubber Dam Systems in Dental Practice. Operative Dentistry 36(3) 243-250, http://dx.doi.org/10.2341/09-283-C

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Reviewers and the Reviewer Board

The list of current Reviewer Board Members will be printed in issue 6 of each volume in a manner that will allow the reviewer to remove the pages for use in professional folders. Reviewer Board members serve as the primary source for peer review of submitted manuscripts, and are invaluable to us. In order to be as efficient as possible for everyone, Reviewers are required to update the online review system with current email address, areas of interest, and dates when unavailable for review. Every effort is made to limit review requests of new manuscripts. It will be assumed that members who repeatedly fail to respond with acceptance or regrets to requests for review will be removed from the Reviewer Board. Should a reviewer’s circumstance change to where they are no longer able or willing to review, we request that a notice be sent to our offices at editor@jopdent.org. Reviewer Board Members can expect to be asked to review to completion no more than 6 (original) manuscripts a year, and to participate in the annual Reviewer 21 Current as of: 3-Sep-14 Board Meeting, whether in person, or by proxy. The following items apply to all reviewers for Operative Dentistry:

• Jopdent must have a CV and current email address on file – the CV is due by the last day of September in the year in which the reviewer completed a review (in order to be recognized in issue 6). It should be updated by the reviewer upon any significant change.

• To be considered for the RB, a reviewer must have 3 or more published articles in internationally recognized journals in which the reviewer was either a corresponding author or 1st author on at least one article.

• A reviewer with “no response” for every request made in a calendar year will be dropped from the RB.

• A reviewer who completed 0 reviews in a calendar year citing, “time constraints” will be removed from the Reviewer Board. Inopportune requests can be prevented by having reviewer availability dates current.

• A reviewer who cites, “conflict of interest” to either decline or withdraw from a review will not be charged for a declined review.

Conflicts of Interest

OpDent believes in the free market and that it is in the best interest of the profession for the market to give back generously to those groups who promote continuing education of those professionals. There must be clear guidelines and expectations however, so that the goodwill and generosity of the Market do not taint the educational activities with bias, real or imagined. To this end we have adopted the following policies and guidelines.

Commercialism

To those who advertise in any medium at any activity where Operative Dentistry, Inc. is acting as the administrative authority for continuing education, whether as sole authority, or in joint sponsorship, the following guidelines must be observed:

1. Program topic selection will be based on perceived needs for professional information and not for the purpose of endorsing specific commercial drugs, materials, products, treatments, or services.

2. Funds received from commercial sources in support of any educational programs shall be unrestricted and the planning committee of said program shall retain exclusive rights regarding selection of presenters, instructional materials, program content and format, etc.

3. Promotional material or other sales activities are not allowed in the area of instruction, neither in the lecture hall/operatory nor in close proximity to the doors of said areas.

Commercial Support

To those who provide monetary support for any activity where Operative Dentistry, Inc. is acting as the administrative authority for continuing education, whether as sole authority, or in joint sponsorship, the following guidelines must be observed:

1. Program topic selection will be based on perceived needs for professional information and not for the purpose of endorsing specific commercial drugs, materials, products, treatments, or services.

2. Funds received from commercial sources in support of any educational programs shall be unrestricted and the planning committee of said program shall retain exclusive rights regarding selection of presenters, instructional materials, program content and format, etc.

3. Any and all commercial support received shall be acknowledged in program announcements, brochures, and in the on-site program book. This announcement may not be located on any page, or facing page, of the book announcing program speakers, or program evaluations.

4. Commercial support shall be limited to:

  a. The payment of reasonable honoraria;

  b. Reimbursement of presenters’ out-of-pocket expenses; and

  c. The payment of the cost of modest meals or social events held as part of an educational activity.

5. When the Provider supports presenters, support shall be limited to:

  a. The payment of reasonable honoraria; and

  b. Reimbursement of presenters’ out-of-pocket expenses.

Full Disclosure

To those who present at any activity where Operative Dentistry, Inc. is acting as the administrative authority for continuing education, whether as sole authority, or in joint sponsorship, the following guidelines must be observed:

1. All presentations should promote improvements in oral healthcare and not specific drugs, devices, services, or techniques.

2. Any media shown to the participants should be free from advertising, trade names, or product messages (except as applies in guideline #3).

3. Presenters shall avoid recommending or mentioning any specific product by its trade name, using generic terms

whenever possible. When reference is made to a specific product by its trade name, reference shall also be made to competitive products.

Conflict of Interest

A Conflict of interest may be considered to exist if a presenter, author or reviewer for an OpDent CDE activity is directly affiliated with or has a direct financial interest in any organization(s) that may be co-supporting a course/manuscript, or may have a direct interest in the subject matter of the presentation/manuscript. The intent of this policy is not to prevent a speaker with an affiliation or financial interest from making a presentation, or submitting a manuscript. It is intended that any potential conflict be identified openly so that the participants in the CDE have the full disclosure of the facts so that they may form their own judgments about the presentation/manuscript. To those who participate at any activity where Operative Dentistry, Inc. is acting as the administrative authority for continuing education, whether as sole authority, or in joint sponsorship, the following guidelines should be understood:

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Speakers/presenters at any CE activity will be required to disclose any potential bias towards commercial supporters, or any other commercial entity that will be mentioned in their presentation.

Author

Authors of every accepted manuscript will be required to disclose any potential bias towards commercial supporters, or any other commercial entity that will be mentioned in their manuscript.

Reviewer

Reviewers of manuscripts will be required to disclose any potential bias towards commercial supporters, or any other commercial entity that is mentioned in the manuscripts they are asked to review. Should a conflict arise, the reviewer is obligated to withdraw themselves as reviewers of the manuscript, and OpDent will select a new reviewer.

Faculty Posting:

Faculty postings are available from OpDent for a $175.00USD flat fee which covers up to 250 words and free logo placement if one is provided. Each additional 50 words is charged at $50.00USD per unit, and each additional issue for which you would like the posting to run is charged at $50.00USD as well. OpDent reserves the right to refuse any posting.

ANEXO C – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *JOURNAL OF PROSTHODONTICS.*

Author Guidelines

Instructions to contributors

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Please note: the Journal of Prosthodontics will no longer review the following manuscripts:

1) Those testing groups with sample sizes less than 10 per group, unless the manuscript also includes a power calculation to determine the small group's statistical validity, or if the manuscript includes a justification for the smaller sample size (i.e., citations to similar studies also using small sample sizes).
2) 2D FEA studies, unless a strong case can be made that the study cannot be conducted via 3D FEA.

Title page - The title page should contain the following information in the order given: 1) Full title of manuscript. 2) Authors' full names. 3) Authors' institutional affiliations including city and country. 4) A running title, not exceeding 60 letters and spaces. 5) The name and address of the author responsible for correspondence about the manuscript.

If the work has previously been presented, the name, place, and date of meeting(s) must be given. If any financial support was received, the grant/contract number, sponsor name, and city, state, and country location must be supplied.

Abstract page – An abstract is required for all manuscripts and must precede the body of the manuscript. Abbreviations and references should not appear in the abstract.

Research manuscripts must conform to the Structured Abstract format. Structured Abstracts should not exceed 350 words and must contain the following information: (1) Purpose (2) Materials and Methods (3) Results (4) Conclusions

Clinical reports and Techniques and Technology manuscripts do not need a structured abstract.

Following the abstract and on the same page, there should be several words not appearing in the title of the manuscript to be titled: KEYWORDS.

Text – Research manuscripts should include the following sections: Introduction, Materials and Methods, Results, Discussion, Conclusion, Acknowledgements, and References. Experimental design should be clearly described (eg, randomized clinical trial, cohort study, case-control study, case series).

Other manuscripts should begin with an introductory paragraph of at least two to five sentences. The remainder of the manuscript should be divided into sections preceded by appropriate headings.

The Introduction will include the following: a description of the problem that inspired the study; a brief discussion of relevant published material that addressed the same problem or that documents methodology used in the study; and the goal of the study, the purpose statement or hypothesis.

The Materials and Methods section describes materials or subjects used and the methods selected to evaluate them, including information about the overall design, the nature of the sample studied, the type of interventions (or treatments) applied to the individual elements in the sample, and the principal outcome measure. Statistical methodology should be included in this section.

Please note: All human subject research (including surveys) must include a statement of ethical or institutional review board approval.

Please note: For research reports, we require a minimum of ten (10) specimens per experimental group UNLESS a power calculation has been performed by a statistician to demonstrate that the sample size is capable of providing statistical significance. Or UNLESS the manuscript includes a justification for the smaller sample size (i.e., citations to similar studies also using small sample sizes).

The Results section will be a clear statement of the findings and an evaluation of their validity based on the outcome of statistical tests.

The Discussion section presents the research in its broader context, describes its clinical implications, identifies limitations or problems that emerged during the course of the study, characterizes the larger significance of the findings, and articulates any further questions remaining to be answered on the subject.

The Conclusion section includes only a brief and succinct summary of the findings.

References - Number references consecutively in the order in which they are first mentioned in the text. Identify references in texts, tables, and legends by superscript Arabic numerals. Use the style of the examples below, which are based on the format used by the US National Library of Medicine in Index Medicus. For abbreviations of journals, consult the 'List of the Journals Indexed' printed annually in the January issue of Index Medicus.

For standard journal articles list all authors when three or fewer; when three or more, list first three authors and add et al.

Example:
Raghoebar GM, Brouwer TJ, Reintesma H, et al: Augmentation of the maxillary sinus floor of autogenous bone for the placement of endosseous implants: A preliminary report. J Oral Maxillofac Surg 1993;51:1198-1203

Chapter in book
Phoenix, RD: Denture base resins: Technical considerations and processing techniques, in Anusavice KJ (ed): Phillips' Science of Dental Materials, vol 1 (ed 10). Philadelphia, PA, Saunders, 1996, pp 237-271

Tables – Tables should be positioned following the references, not in the body of the manuscript. The tables should be numbered consecutively with Arabic numerals. Each table should be typed on a separate sheet. Include any necessary legends on the same page with the associated table.

Illustrations – All graphs, drawings, and photographs are considered figures and should be numbered in sequence with Arabic numerals. Each figure should have a legend and all legends should be typed together on a separate sheet and numbered correspondingly.

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Figures submitted to the Journal of Prosthodontics

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Upon approval for publication, we will publish the letter in the next available print issue of the Journal of Prosthodontics. When written in response to an article published in the Journal, we will also give the author of the original article the opportunity to respond. If they choose to do so, we will attempt to publish the letter and response in the same issue.

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