

UNIVERSIDADE DE TAUBATÉ
Frederico dos Reis Goyatá

**AVALIAÇÃO DE PARÂMETROS CLÍNICOS
RELACIONADOS COM A LONGEVIDADE DE
RESTAURAÇÕES CERÂMICAS LIVRES DE METAL**

Taubaté - SP
2010

UNIVERSIDADE DE TAUBATÉ
Frederico dos Reis Goyatá

**AVALIAÇÃO DE PARÂMETROS CLÍNICOS
RELACIONADOS COM A LONGEVIDADE DE
RESTAURAÇÕES CERÂMICAS LIVRES DE METAL**

Tese apresentada para a obtenção do Título de Doutor pelo Programa de Pós-graduação em Odontologia do Departamento de Odontologia da Universidade de Taubaté.
Orientador: Prof. Dr. Leonardo Gonçalves Cunha

Taubaté - SP
2010

**Ficha catalográfica elaborada pelo
SIBi – Sistema Integrado de Bibliotecas / UNITAU**

G724r Goyatá, Frederico dos Reis
Avaliação de parâmetros clínicos relacionados com a longevidade de restaurações cerâmicas livres de metal / Frederico dos Reis Goyatá. - 2010.
96f.il.

Tese (doutorado) - Universidade de Taubaté, Programa de Pós-graduação em Odontologia, 2010.
Orientação: Prof. Dr. Leonardo Gonçalves Cunha, Departamento de Odontologia.

1. Cerâmica. 2. Cimentação. 3. Rugosidade. 4. Prótese dentária.
I. Título.

FREDERICO DOS REIS GOYATÁ

Data: _____

Resultado: _____

BANCA EXAMINADORA

Prof.Dr. Leonardo Gonçalves Cunha – Universidade de Taubaté

Assinatura _____

Prof.Dra. Ana Christina Claro Neves – Universidade de Taubaté

Assinatura _____

Prof.Dr. Sigmar de Mello Rode – Universidade de Taubaté

Assinatura _____

Prof.Dra. Fabíola Pessoa Pereira Leite – Universidade Federal de Juiz de Fora

Assinatura _____

Prof.Dr. Leonardo César Costa – Universidade Federal de Juiz de Fora

Assinatura _____

Dedico este trabalho às pessoas mais importantes e maravilhosas da minha vida, meus amores, minha esposa Fernanda e meu filho Enzo. Sem vocês não sou absolutamente nada.

Faço uma dedicatória especial à minha querida mãe Luzia, a você agradeço a minha vida, minha educação e formação como homem e profissional, obrigado pelo apoio nos momentos mais difíceis.

Aos meus irmãos, cunhados e cunhadas, sobrinhos, amigos que estiveram comigo e me apoiaram constantemente.

AGRADECIMENTOS

Agradeço ao meu orientador Prof. Dr. Leonardo Gonçalves Cunha, que pessoa espetacular. Obrigado por tudo, você me apresentou a pesquisa e me ajudou muito a crescer como professor e pesquisador.

Obrigado a todos os professores do Programa de Pós-graduação em Odontologia da UNITAU. À Profa. Dra. Ana Christina Claro Neves, quanta dedicação e preocupação com o andamento e qualidade do curso. Aos Professores Doutores: Laís Regiane, Sigmar, Vanessa, Gilson, Davi, João Marcelo obrigado pela convivência e experiência adquirida com vocês. Não esquecendo o Biba por ter me apresentado à Profa Ana Christina e a UNITAU.

Aos meus colegas e amigos do Doutorado, Júlio Brigolini, Rafael Pazinato e Daher Queiroz, obrigado pela presença, pelo apoio, pela amizade e companheirismo.

Agradeço ao Laboratório de Testes Mecânicos da Faculdade de Engenharia - UNITAU, ao laboratório de Testes Mecânicos da Faculdade de Engenharia - UNESP, Guaratinguetá na pessoa do Neto, ao LNLS na pessoa do Vinícius.

Agradeço à Universidade Severino Sombra e aos meus colegas professores do Curso de Odontologia pelo apoio incondicional.

Agradeço à Faculdade de Odontologia da UFJF, aos professores Doutores Fabíola e Leonardo Costa por terem aceitado meu convite para a Banca e pela amizade e convivência profissional, aprendo muito com vocês.

Um agradecimento especial à minha família em Taubaté, Telma, Toninho, Isabel e Felipe, obrigado pela acolhida e pelo apoio de vocês, sempre me senti em casa.

Goyatá FR. Avaliação de parâmetros clínicos relacionados com a longevidade de restaurações cerâmicas livres de metal (Tese de doutorado). Taubaté: Universidade de Taubaté, Departamento de Odontologia, 2010. 96p.

RESUMO

Proporcionar longevidade clínica às restaurações cerâmicas deve ser um dos objetivos principais dos cirurgiões-dentistas ao realizar um tratamento protético.

Objetivo: Correlacionar a influência do tratamento interno do substrato cerâmico e do tipo de polimento da superfície cerâmica externa com a efetividade clínica de restaurações com sistemas cerâmicos livres de metal. **Métodos:** Foram elaborados quatro artigos científicos. No Capítulo 1, apresentou-se um estudo de revisão da literatura a respeito dos métodos de tratamento de superfície para as cerâmicas reforçadas por óxido de zircônia e a implicação clínica deste procedimento na resistência da união deste sistema cerâmico quando realizada cimentação adesiva. Os Capítulos de 2 a 4 são representativos de trabalhos in vitro. No Capítulo 2, foi delineado um experimento com o objetivo de avaliar a influência do tratamento da superfície interna da cerâmica e do tipo de cimento (resinoso dual ou autoadesivo) sobre a resistência da união da interface cerâmica-cimento por meio de ensaio de microcisalhamento. Os tratamentos de superfície avaliados foram: condicionamento com ácido fluorídrico a 10%, jateamento com óxido de alumínio 110µm, silicatização e junção de jateamento com óxido de alumínio 110µm e aplicação de um agente silano específicos. O teste foi realizado após o período de 24 horas e os resultados expressos em MPa. Adicionalmente, foi avaliada quantitativamente e qualitativamente a modificação promovida na superfície interna do substrato cerâmico por cada um dos tratamentos de superfície avaliados. No Capítulo 3, o mesmo delineamento executado no Capítulo 2 foi realizado, porém avaliando-se a resistência à degradação hidrolítica promovida pelos mesmos métodos de tratamento da superfície interna da cerâmica, com a realização do ensaio de resistência da união ao microcisalhamento após 24 horas e seis meses de armazenagem em água. No Capítulo 4, foi avaliada a eficácia de diferentes métodos de polimento da superfície cerâmica externa por meio de um ensaio de rugosidade de superfície e microscopia de força atômica, sendo ainda realizada a comparação entre os sistemas cerâmicos pelo ensaio de resistência à tração diametral. **Conclusões:** Com este trabalho foi possível determinar a importância do tratamento da superfície interna das cerâmicas previamente à cimentação adesiva, objetivando aprimorar a resistência da união da interface cerâmica-cimento, conferindo longevidade às restaurações cerâmicas. Com este propósito, para sistemas cerâmicos reforçados por zircônia, tratamentos de superfície que promovam modificações mecânicas e químicas da superfície cerâmica devem ser utilizados. Também foi possível observar que as restaurações cerâmicas depois de cimentadas e ajustadas clinicamente, devem ser submetidas a técnicas de polimento, sendo que a efetividade desta etapa é dependente da técnica de polimento executada.

Palavras-chave: Cerâmica; Cimentação; Rugosidade; Prótese dentária.

FR Goyatá. Evaluation of clinical parameters to associated with longevity in metal free ceramic restorations (Tese de doutorado). Taubaté: Universidade de Taubaté, Departamento de Odontologia, 2010. 96p.

ABSTRACT

Make the clinical longevity to ceramic restorations has must been a principal objective in prosthetic treatment realized for a dentist.

Purpose: Correlation the influence of internal treatment in ceramic substrate and polishing method to ceramic external surface with clinical effectiveness in the metal free ceramic systems. **Methods:** Was elaborated four scientific articles. In the chapter one, present a literature review study with describes the treatment surface methods to ceramics reinforced to zirconium oxide and the clinical importance this procedure to the bond strength when realized adhesive cementation with ceramic system. The chapter two at four represented in vitro studies. In the chapter two, was delineated a experiment with the objective to evaluated the influence internal treatment in ceramic and the type of cement (dual cure resin and self adhesive) on the bond strength in interface ceramic-cement with microshear test. The surface treatments was: hydrofluoric acid conditioned, sandblasting using aluminum oxide 110µm, silica coating and sandblasting using aluminum oxide 110µm associated with silane agent specified. The test was performed after 24 hours and results presents in Mpa. In addition, was a quantitative and qualitatively evaluation the modification to improved in internal surface in ceramic substrate for the each surface treatment evaluated. In the chapter three, was realized the study assemble the chapter two, therefore evaluated the hydrophilic degradation improved to surface treatment internal ceramic with the microshear test after 24 hours and six months in water storage. In the chapter four, the efficacy in different polishing methods of external surface ceramic was evaluated by the surface roughness test and atomic force microscope, being even performed comparison between ceramic systems and the experiment of the diametral tensile strength. **Conclusions:** In this study, was possible to determinate the importance of internal surface treatment in ceramics previously adhesive cementation with objective the best bond strength in ceramic-cement interface to obtained longevity in ceramic restorations. With this objective, to ceramics reinforced to zirconium oxide, treatments surface that improve mechanical and chemistry modification to ceramic surface must be realized. Was possible too observed that ceramic restorations after cementations and clinical adjustments must be submitted the polishing techniques where the effectiveness is depended to polishing technique realized.

Keywords: Ceramic; Cementation; Roughness; Prosthetic dentistry.

SUMÁRIO

1 INTRODUÇÃO	8
2 CAPITULO 1	
2.1 A IMPORTÂNCIA DO TRATAMENTO DE SUPERFÍCIE DAS CERÂMICAS REFORÇADAS POR ÓXIDO DE ZIRCÔNIA PREVIAMENTE À CIMENTAÇÃO ADESIVA	14
3 CAPITULO 2	
3.1 CHARACTERIZATION AND INFLUENCE OF SURFACE TREATMENTS ON TOPOGRAPHY AND BOND STRENGTH OF LUTING AGENTS TO A DENSELY-SINTERED ZIRCONIUM-OXIDE CERAMIC	30
4 CAPITULO 3	
4.1 DURABILITY OF RESIN BOND STRENGTH TO A DENSELY-SINTERED ZIRCONIUM-OXIDE CERAMIC AFTER SURFACE TREATMENT METHODS	51
5 CAPITULO 4	
5.1 SURFACE TOPOGRAPHY AND MECHANICAL PROPERTIES OF CERAMIC SYSTEMS AFTER DIFFERENT POLISHING METHODS: PERFILOMETER AND AFM ANALYSIS	70
6 CONSIDERAÇÕES FINAIS	89
REFERÊNCIAS	91

1 INTRODUÇÃO

As cerâmicas odontológicas são também denominadas como porcelanas, cerâmicas vítreas ou estruturas altamente alcalinas. Possuem propriedades químicas, mecânicas, físicas e térmicas que as distinguem de outros materiais, tais como metais e resinas compostas. As suas propriedades são desenvolvidas especialmente para aplicação na odontologia por meio de um controle rígido do tipo e quantidade de componentes usados na sua produção, o que determinará de forma contundente a sua indicação clínica mais precisa. As cerâmicas odontológicas exibem uma resistência à flexão e tenacidade à fratura de moderada à excelente. Embora as cerâmicas sejam resistentes às altas temperaturas, elas são friáveis e podem apresentar fraturas quando flexionadas ou expostas seguidamente ao calor e ao frio (Anusavice, 2003).

Inicialmente, as cerâmicas foram introduzidas na Odontologia para as próteses fixas do tipo coroa total metalocerâmica, constituindo-se numa estrutura para cobertura estética de uma infraestrutura metálica. Estas cerâmicas são denominadas cerâmicas feldspáticas, compostas por uma variedade de óxidos constituídos em uma matriz vítrea com uma ou mais fases cristalinas. Diversos fatores contribuíram para a ampla indicação e utilização clínica desta restauração protética na Odontologia. Fatores como longevidade comprovada por estudos clínicos de acompanhamento por dez anos, resistência mecânica, facilidade técnica e custo foram determinantes para a sedimentação das próteses metalocerâmicas na Odontologia (Fradeani & Redemagni, 2002).

Embora as próteses metalocerâmicas venham sendo utilizadas com índices de sucesso superior a 90% após dez anos de acompanhamento clínico, apresentam diferentes desvantagens se relacionadas às próteses livres de metal. A ausência de biocompatibilidade com o periodonto de algumas ligas metálicas utilizadas como infraestrutura é umas das principais desvantagens, podendo provocar um manchamento da região cervical em contato com o metal pode proporcionar reações inflamatórias crônicas em pacientes alérgicos (Mehulic et al., 2005). Adicionalmente, mesmo que a cerâmica feldspática apresente características ópticas semelhantes à estrutura dental, a sua utilização com as ligas metálicas reduz essa característica e limita significativamente o resultado estético (Kramer & Frankenberger, 2005).

Dessa forma, materiais cerâmicos que eliminassem a necessidade de utilização das ligas metálicas como opção nas infraestruturas de próteses fixas começaram a ser desenvolvidos (Luthy et al., 2005; Mc Laren & Giordano, 2005). Diferentes sistemas cerâmicos livres de metal são encontrados comercialmente, utilizando adições de partículas de reforço e métodos de fabricação. Em relação às partículas de reforço, pode ser destacado o uso de dissilicato de lítio (IPS e.max/Ivoclar-Vivadent), alumina e zircônia infiltrada por vidro (InCeram, Vita) e os sistemas reforçados por alumina ou zircônia (Procera/Nobel Biocare; Cercon/Degudent; Lava/3M-ESPE). As propriedades ópticas e físico-mecânicas destas cerâmicas estão diretamente relacionadas ao tipo, quantidade, tamanho, e características físico-químicas das partículas de reforço (Atsu et al., 2006; Griggs, 2007; Manicone et al., 2007; Della Bona et al., 2008; Hilgert et al., 2009a; Hilgert et al., 2009b; Hilgert et al., 2010).

Apesar da alta resistência e estética favorável dos sistemas reforçados por zircônia, diversos trabalhos na literatura enfatizam que as possibilidades

convencionais de tratamento de superfície previamente à cimentação não promovem uma união efetiva entre a cerâmica e o cimento resinoso (Valandro et al., 2005; Kiyon et al., 2007).

Diversas pesquisas associaram como uma das principais causas de insucesso em reabilitações cerâmicas falhas na cimentação e atribuíram ao tratamento interno da superfície cerâmica insuficiente (Borges et al., 2003; Bottino et al., 2005). Dentre as opções de tratamento de superfície, destacam-se, como as mais utilizadas, o condicionamento com ácido fluorídrico em diferentes concentrações, o jateamento interno da superfície das restaurações com partículas de óxido de alumínio em diferentes tamanhos, e a aplicação de agentes anfóteros (silano) (Spohr et al., 2003; Guler et al., 2005; Valandro et al., 2005).

O condicionamento com ácido fluorídrico é baseado na modificação estrutural da superfície e na capacidade de molhamento da cerâmica, aumentando a energia de superfície e, conseqüentemente, melhorando a capacidade de união da cerâmica com o cimento resinoso. Entretanto, a microestrutura dos sistemas cerâmicos reforçados por zircônia evidencia uma superfície ácido-resistente, não sendo observadas alterações topográficas significativas após o condicionamento com este ácido (Borges et al., 2003; Della Bona et al., 2007a; Spohr et al., 2008; Chaiyabutr et al., 2008).

O jateamento com partículas de óxido de alumínio representa uma opção de tratamento de superfície em que irregularidades superficiais são criadas na cerâmica, aumentando a possibilidade de retenção micromecânica com o cimento resinoso. Alguns estudos verificaram que o jateamento em cerâmicas reforçadas por zircônia foram efetivos somente em um momento inicial, porém não estável, com redução significativa da resistência da união após diferentes períodos de

armazenagem e termociclagem (Guazzato et al., 2006; Akgungor et al., 2008).

A aplicação de uma camada de sílica, denominado silicatização, tem sido muito utilizada como tratamento de superfície para cerâmicas reforçadas por zircônia. Foi inicialmente desenvolvido para metais (Peutzfeldt & Asmussen, 1988) objetivando o aumento da resistência da união com os cimentos resinosos. As apresentações comerciais incluem o sistema Rocatec e Cojet (3M-ESPE) e o sistema Silicoater MD (Heraeus). A técnica consiste no jateamento da superfície interna da cerâmica com partículas de óxido de alumínio modificadas por sílica (Özcan, 2002). A pressão do jateamento promove a formação de uma camada de sílica na cerâmica com efetiva capacidade de união ao agente silano, tornando essa superfície quimicamente reativa com o cimento resinoso. Este método tem mostrado resultados satisfatórios de resistência da união em diferentes estudos encontrados na literatura (Özcan & Vallitu, 2003; Della Bona et al., 2007b; Blatz et al., 2007; Re et al., 2008; May, 2010).

A cimentação representa uma etapa de significativa importância nas restaurações com sistemas cerâmicos livres de metal (Blatz et al., 2003). A resistência e a durabilidade da união entre a cerâmica, o cimento e o substrato dental possui papel importante na longevidade clínica das restaurações (Piwowarczyk et al., 2007; Hilgert et al., 2009c).

Atualmente, diversas opções de cimentos estão disponíveis e com uma especificidade de acordo com o sistema cerâmico utilizado pelo cirurgião-dentista. Cimentos resinosos de presa dual e fotoativado, cimentos autoadesivos em que não há necessidade de pré-tratamento do substrato dental, simplificando dessa forma o procedimento clínico de cimentação e diminuindo a sensibilidade da técnica adesiva (Radovic et al., 2008). Diversos estudos associaram o cimento autoadesivo à

promoção de satisfatória adaptação marginal de restaurações com sistemas cerâmicos livres de metal (Behr et al., 2004; Fabianelli et al., 2005; Ibarra et al., 2007).

Posteriormente à cimentação adesiva das restaurações cerâmicas, na maioria dos casos, é necessário realizar um ajuste oclusal a fim de proporcionar uma oclusão funcional ao paciente e saúde ao sistema estomatognático (Okeson, 2010).

Em função desta etapa clínica, as restaurações cerâmicas poderão adquirir uma rugosidade de superfície comprometendo diretamente a sua longevidade na cavidade bucal em função da possível propagação de trincas na sua estrutura assim como um desgaste superficial conforme o contato oclusal com o dente antagonista ou outro material dentário (Fischer et al., 2003; Loubaher et al., 2008). Também poderá ocorrer um comprometimento da saúde do periodonto em função do acúmulo de microrganismos na superfície das restaurações (Kawai et al., 2000; Auschill et al., 2002).

Os diferentes métodos de polimento para as cerâmicas odontológicas têm como objetivo principal estabelecer uma lisura de superfície semelhante ou próxima ao glaze aplicado ao final da confecção laboratorial das restaurações protéticas (Raimondo et al., 1990; Al-Wahadani & Martin, 1998; Sarykaya & Guler 2010; Ylmaz & Özcan, 2010).

Diversos métodos de polimento são descritos na literatura com certa especificidade para os diferentes sistemas cerâmicos. Torna-se muito importante o cirurgião-dentista estabelecer um critério clínico de utilização destes métodos de acordo com o tipo de cerâmica que será utilizada seja nas restaurações parciais do tipo *inlay* e *onlay* até as próteses do tipo coroa total e as próteses parciais fixas (Wright et al., 2004; Sarac et al., 2006; Sarac et al., 2007).

Este trabalho visa apresentar uma relação entre a resistência adesiva e a rugosidade de superfície das cerâmicas odontológicas com a longevidade clínica das restaurações pela descrição de quatro artigos científicos, um de revisão de literatura e três artigos de pesquisa “in vitro”.

2 CAPÍTULO 1

2.1 A Importância do Tratamento de Superfície das Cerâmicas Reforçadas por Óxido de Zircônia Previamente à Cimentação Adesiva

The Importance of the Surface Treatment in Zirconium Oxide Reinforced Ceramic Previously to Adhesive Cementation

AUTORES:

Frederico dos Reis Goyatá *

Especialista, Mestre e Doutorando em Prótese UNITAU – Taubaté-SP.

Professor Assistente II de Prótese do Curso de Odontologia da USS – Vassouras – RJ.

*Av. Rui Barbosa 310/802 - CEP: 27521-190 - Resende - RJ.

Email: fredgoyata@oi.com.br

Leonardo Gonçalves Cunha

Professor Doutor do Programa de Pós-Graduação Mestrado e Doutorado em Odontologia da UNITAU- Taubaté-SP.

José Guilherme da Rocha Gilson

Cirurgião-dentista graduado pelo Curso de Odontologia da USS– Vassouras – RJ.

Fabiano Vieira de Landa

Cirurgião-dentista graduado pelo Curso de Odontologia da USS.

Especialista em Prótese Dentária pela SUPREMA – Juiz de Fora-MG

RESUMO

As próteses livres de metal têm sido muito utilizadas na Odontologia restauradora estética. Diferentes sistemas cerâmicos surgiram nos últimos anos com tecnologia de fabricação apurada e excelente qualidade estética. As cerâmicas reforçadas por óxido de zircônia têm sido indicadas na confecção de coroas totais e próteses fixas em dentes anteriores e posteriores. Em função da necessidade de se realizar uma cimentação adesiva eficiente, este trabalho tem por objetivo realizar uma revisão da literatura dos métodos de tratamento de superfície empregados para as cerâmicas reforçadas por zircônia previamente à cimentação adesiva.

PALAVRAS - CHAVE

Prótese Dentária. Cerâmica. Tratamento de Superfície. Cimentação.

SIGNIFICÂNCIA CLÍNICA

Estabelecer um protocolo de cimentação para as próteses livres de metal com cerâmica reforçada por óxido de zircônia é importante para obter um prognóstico favorável e conferir longevidade clínica ao tratamento reabilitador. Dentro deste protocolo, o tratamento de superfície irá determinar uma alta resistência de união da cerâmica com o cimento e deste com o substrato dental.

ABSTRACT

Metal-free prosthetic has great use in esthetic restorative dentistry. Different ceramic systems appeared in the last few years with accurate manufacture technology and high aesthetic quality. Zirconia- reinforced ceramics have been recommended for the fixed prosthesis and full crown making in anterior and posterior teeth. In order to accomplish an efficient adhesive

cementation, this work focuses on literature review concerning the methods of surface treatment employed to the reinforced ceramics by Zirconia before adhesive cementation.

KEYWORDS

Prosthetic Dentistry. Ceramic. Surface Treatment. Cementation

INTRODUÇÃO

Embora as próteses fixas metalocerâmicas venham sendo utilizadas com altos índices de sucesso ao longo dos anos na Odontologia, alguns aspectos desvantajosos em relação a esta opção reabilitadora são evidenciados ¹. Uma das desvantagens está relacionada à biocompatibilidade com o tecido gengival de algumas ligas metálicas utilizadas como infraestruturas. Esta situação acarreta o manchamento da região gengival devido ao contato com óxidos metálicos e pode ocasionar reações inflamatórias crônicas em pacientes alérgicos ².

Outra desvantagem é a estética inadequada e mesmo que a cerâmica feldspática apresente características ópticas semelhantes à estrutura dental, a sua utilização com as ligas metálicas reduz essa característica e limita significativamente o resultado estético alcançado por esse material ³.

Dessa forma, materiais cerâmicos para infraestruturas de próteses fixas começaram a ser desenvolvidos ⁴⁻⁶. Diferentes sistemas cerâmicos livres de metal são encontrados comercialmente e caracterizam-se, principalmente, pela incorporação de partículas de reforço e modificações nos métodos laboratoriais de confecção das próteses. Com relação às partículas de reforço, pode ser destacado o uso do dissilicato de lítio, óxido de alumina e zircônia infiltrado por vidro e sistemas altamente reforçados por óxido de alumina ou

zircônia. As propriedades ópticas e físico-mecânicas destas cerâmicas estão diretamente relacionadas ao tipo, quantidade, tamanho, e características físico-químicas das partículas de reforço utilizadas⁷⁻⁹.

Apesar da alta resistência e estética favorável dos sistemas cerâmicos reforçados por óxido de zircônia, alguns trabalhos na literatura enfatizam que o tratamento de superfície convencional previamente ao procedimento clínico de cimentação não promove união efetiva entre cerâmica e o cimento resinoso¹⁰⁻¹².

Estudos “in vitro” associaram como uma das principais causas de insucesso em reabilitações cerâmicas falhas na cimentação e atribuíram a um tratamento da superfície cerâmica insuficiente¹³⁻¹⁴. Dentre as opções de tratamento de superfície existentes, destacam-se como as mais utilizadas o condicionamento com ácido fluorídrico em diferentes concentrações, o jateamento interno da superfície das restaurações com partículas de óxido de alumínio em diferentes tamanhos, e a aplicação de agentes anfóteros (silano)¹⁵⁻¹⁶.

O condicionamento com ácido fluorídrico a 10% é baseado na modificação estrutural da superfície e na capacidade de molhamento da cerâmica, aumentando a energia de superfície e, conseqüentemente, melhorando a capacidade de união da cerâmica ao cimento resinoso. Entretanto, a microestrutura dos sistemas cerâmicos reforçados por óxido de zircônia evidencia uma superfície ácido-resistente, não sendo observadas alterações topográficas significativas após o condicionamento com este tipo de ácido^{12,17-19}.

O jateamento com partículas de óxido de alumínio representa uma opção de tratamento de superfície em que irregularidades superficiais são criadas na cerâmica, aumentando a possibilidade de retenção micromecânica com o cimento resinoso. Entretanto, alguns estudos verificaram que o jateamento em sistemas cerâmicos reforçados por óxido de zircônia torna-se efetivo somente em um momento inicial, porém não estável, com redução

significativa da resistência da união após diferentes períodos de armazenagem e termociclagem²⁰⁻²¹.

A aplicação de uma camada de sílica, processo intitulado silicatização, tem sido muito utilizado para estas cerâmicas. Este tipo de tratamento foi inicialmente desenvolvido para metais objetivando o aumento da resistência da união com os cimentos resinosos²². A técnica consiste no jateamento da superfície interna da cerâmica com partículas de óxido de alumínio modificadas por sílica²³. A pressão do jateamento promove a formação de uma camada de sílica, agora com efetiva capacidade de união ao agente silano, tornando essa superfície quimicamente reativa com o cimento resinoso. Este método tem mostrado resultados satisfatórios de resistência da união²⁴⁻²⁹.

Em função da necessidade de se realizar uma cimentação adesiva eficiente para garantir longevidade clínica às próteses confeccionadas em cerâmica³⁰⁻³⁶, este trabalho tem por objetivo realizar uma revisão da literatura dos métodos de tratamento de superfície empregados para as cerâmicas reforçadas por zircônia previamente à cimentação adesiva.

REVISÃO DA LITERATURA

Cerâmicas Odontológicas

Com o advento da Odontologia estética, houve uma necessidade de mudança nas condutas clínicas pelos cirurgiões-dentistas. Os insucessos estéticos com coroas e restaurações indiretas parciais metálicas impulsionou estas mudanças. Novas possibilidades de tratamento restaurador protético com as cerâmicas foram introduzidas clinicamente, tais como: facetas estéticas e restaurações parciais (*inlay* e *onlay*) em dentes posteriores sempre enfatizando o resultado estético do tratamento².

Alguns estudos têm sido descritos na literatura, principalmente na última década, abordando o desenvolvimento de novos sistemas cerâmicos e novas tecnologias para a confecção de próteses livres de metal. Diferentes tipos de cerâmica foram produzidos com o objetivo de conferir mais resistência ao material e garantir estética superior às próteses livres de metal. Surgiram as porcelanas aluminizadas, os vidros ceramizados fundidos e usinados e os vidros ceramizados prensados. Na medida em que estes novos sistemas cerâmicos foram desenvolvidos, estudos clínicos foram realizados com o objetivo de estabelecer uma relação de biocompatibilidade e longevidade da cerâmica com meio bucal determinando indicações e contra-indicações das próteses livres de metal ³⁻⁴.

Com o surgimento de novos sistemas cerâmicos como o sistema In Ceram (VITA, Alemanha) a base de óxido de alumina, os sistemas IPS Empress, IPS Empress 2 (Ivoclar Vivadent, Liechtenstein) com cerâmicas a base de dissilicato de lítio, as indicações clínicas para próteses livres de metal passaram a ter uma abrangência maior dentro da Odontologia reabilitadora com possibilidades desde restaurações parciais como restaurados *inlay*, *onlay* e *overlay* e facetas estéticas até coroas totais e próteses fixas de três elementos em dentes anteriores ^{5,7,36}.

Estudos científicos e clínicos passaram a apontar para um cuidado maior na indicação de próteses fixas livres de metal em dentes posteriores, devido ao grande estresse e cargas oclusais geradas nesta região da cavidade bucal. A opção pelo desenvolvimento de infraestruturas cerâmicas à base de óxido de zircônia teve como principal objetivo conferir maior resistência mecânica às próteses e desta forma também ampliar a indicação destas reabilitações na substituição de um ou mais dentes posteriores ausentes ⁸⁻⁹.

Recentemente, houve a incorporação de partículas de óxido de ítrio às infraestruturas de óxido de zircônia com o objetivo de elevar a resistência mecânica e assim ratificar a

indicação das próteses fixas livres de metal em dentes posteriores. Desta forma, a possibilidade de microfraturas na infraestrutura cerâmica será minimizada pela adição do óxido de ítrio que irá contribuir para formação de uma estrutura mais homogênea e resistente. Aliado a isto tem sido observado um grande avanço nos métodos de fabricação e confecção das infraestruturas. A grande maioria dos sistemas atuais utilizam o método CAD/CAM para elaboração das infraestruturas tais como: (Cercon, Degudent, Alemanha); (Cerec, Sirona, Alemanha); (Lava, 3M-ESPE, USA); (Procera, Nobel Biocare, Suécia). Em países desenvolvidos, este método é uma realidade, como por exemplo, na Alemanha, onde, em 2007, foram produzidas 2,5 milhões de restaurações cerâmicas, das quais 72% tiveram em sua confecção o envolvimento de técnicas CAD/CAM³⁷.

O termo CAD/CAM deriva de “Computer Aided Design/ Computer Aided Manufacturing” em que todos os métodos de planejamento e produção de uma prótese são realizados com auxílio do computador. Inicialmente, uma ferramenta irá digitalizar o preparo protético criando um modelo virtual no computador e um programa (*software*) irá planejar a infraestrutura virtualmente, sendo esta etapa do trabalho executada pelo componente CAD do sistema. A partir daí, uma tecnologia de produção (componente CAM) processará a infraestrutura a partir de um bloco cerâmico selecionado. O principal objetivo deste sistema de confecção de infraestruturas cerâmicas para próteses fixas livres de metal é ampliar as indicações clínicas e proporcionar outra possibilidade clínica de tratamento reabilitador³⁸⁻³⁹.

Tratamento de Superfície

Na cimentação das restaurações cerâmicas é fundamental que se estabeleça uma boa adesão do material cerâmico ao substrato dental proporcionando segurança e longevidade no tratamento. Diferentes métodos para o tratamento da superfície cerâmica, previamente à cimentação, tem sido descritos. Estes métodos tem como principal objetivo à obtenção de uma

superfície mais rugosa e propensa à adesão, pelo aumento da energia livre de superfície ^{12,14,22-24}.

O tratamento de superfície das cerâmicas também visa melhorar o resultado clínico dos procedimentos de cimentação. A norma ISO 6872 classificou as cerâmicas de acordo com sua indicação clínica e filosofia de cimentação empregada. Estabeleceu-se dois grupos de cerâmicas: as cimentadas de forma adesiva e as cerâmicas cimentadas de forma não adesiva. Seguindo este raciocínio, alguns autores classificam as cerâmicas odontológicas em cerâmicas ácido sensíveis e cerâmicas ácido resistentes, criterizando o material pela modificação de sua estrutura superficial devido ação do ácido fluorídrico a 10% ^{10,13,15,36}.

As cerâmicas feldspáticas e as cerâmicas a base de dissilicato de lítio podem ser classificadas como cerâmicas ácido sensíveis e as cerâmicas reforçadas por óxido de alumina e óxido de zircônia como cerâmicas ácido resistentes. Para o primeiro grupo de cerâmicas, o condicionamento com ácido fluorídrico já está bastante sedimentado pela prática clínica e estudos científicos, sendo amplamente aceito pela comunidade científica odontológica. Já as cerâmicas reforçadas por óxido de alumina ou zircônia, por apresentarem uma microestrutura físico-química muito densa, dificilmente a ação do ácido fluorídrico promoverá qualquer alteração significativa em sua superfície. Estas cerâmicas necessitam de um tratamento com utilização de jatos abrasivos com partículas de óxido de alumínio revestidas por sílica a fim de promover uma alteração na rugosidade da superfície aumentando a energia livre de superfície. Dentre os métodos utilizados destacam-se os sistemas Rocatec (indicados para laboratórios de prótese) e Cojet (indicados para aplicação em consultório) desenvolvidos e comercializados pela empresa 3M-ESPE (USA) ^{25,27-28}.

Cimentação Adesiva

Dentre as etapas da cimentação de uma prótese ou restauração cerâmica, a seleção correta do agente cimentante e sua relação com o tipo de cerâmica utilizada tem grande importância no sucesso do tratamento. Observar as características físico-químicas dos cimentos, adesividade, solubilidade em meio bucal são fatores que irão determinar a escolha do cimento ideal para cada restauração cerâmica. O principal objetivo desta etapa clínica é estabelecer uma forte união micro-mecânica e adesiva entre a restauração cerâmica, o cimento e o substrato dental, sendo este o objetivo dos cirurgiões-dentistas e um fator determinante para conferir longevidade às próteses ³¹⁻³².

De acordo com a classificação determinada pela ISO 6872 que esquematizou as cerâmicas de acordo com sua indicação clínica e filosofia de cimentação empregada: cimentação adesiva e cimentação não adesiva (ou convencional). Basicamente na filosofia de cimentação convencional a união se dará por embricamento micromecânico e a opção clínica fundamenta-se pelos cimentos convencionais do tipo fosfato de zinco e cimento de ionômero de vidro. Na filosofia adesiva, a opção são os cimentos resinosos, fotoativados, de polimerização química ou dual estabelecendo-se forte união adesiva da cerâmica com o cimento e substrato dental ²⁹.

Nas cerâmicas ácido sensíveis o método de tratamento de superfície com ácido fluorídrico a 10% associados a silanização e aplicação de um agente cimentante resinoso tem proporcionado resultados clínicos bastante satisfatórios ao longo dos anos, sendo sedimentados na literatura, por trabalhos que conferem longevidade clínica à cimentação evidenciando valores elevados de resistência adesiva e baixa solubilidade em meio bucal mesmo após longo tempo de armazenamento ³⁰.

Nas cerâmicas reforçadas por óxido de zircônia, classificadas como cerâmicas ácido resistentes, o objetivo dos estudos científicos tem sido promover uma união micromecânica e adesiva mais estável entre cerâmica, cimento resinoso e substrato dental. Dentro deste aspecto, o tratamento de superfície com o jateamento com partículas de óxido de alumínio e sílica associados a silanização tem proporcionado resultados de resistência adesiva melhores e como consequência uma cimentação mais efetiva ^{11,16,18-20}.

Aliado a isto, o desenvolvimento de uma ampla gama de cimentos resinosos de polimerização dual e cimentos autoadesivos assim como os cimentos resinosos autopolimerizáveis com monômeros fosfatados tem possibilitado ao cirurgião-dentista ampliar as opções quanto à seleção dos cimentos resinosos para a cimentação de restaurações cerâmicas e obter melhores resultados clínicos com as próteses livres de metal ³²⁻³⁵.

Nos últimos anos tem sido observado uma grande utilização dos cimentos resinosos autoadesivos na prática clínica odontológica, principalmente em função da praticidade de uso. Diversos estudos têm sido realizados com o objetivo de qualificar algumas características clínicas destes cimentos, tais como: espessura de película de cimentação, microinfiltração marginal e características mecânicas, principalmente a resistência adesiva. Os estudos tem avaliado a resistência adesiva por teste de microtração e microcisalhamento com resultados que indicam uma predominância na seleção destes dois tipos de testes, em que se utilizam amostras com dimensões reduzidas, tornando os resultados das pesquisas mais fidedignos com a realidade clínica ^{6,17,26,30}.

Tem sido utilizado nos estudos com testes de resistência adesiva a armazenagem dos espécimes (cerâmica – cimento resinoso) em ambiente úmido, seja com métodos de termociclagem ou até mesmo a estocagem em água armazenados em estufa a fim de aproximar os testes in vitro de uma situação mais próxima da realidade clínica ^{21,37-38}.

DISCUSSÃO

Novos materiais dentários livres de metal tem sido desenvolvidos como opção no planejamento das infraestruturas para as prótese fixas. Dentro deste contexto, destacam-se o grande avanço das cerâmicas odontológicas e suas possibilidades clínicas ¹⁻³.

Diversos sistemas cerâmicos foram desenvolvidos, porcelanas aluminizadas, os vidros ceramizados fundidos e usinados e os vidros ceramizados prensados, todos objetivando biocompatibilidade e principalmente estética, o que tem sido observado nos resultados clínicos com as próteses livres de metal ^{4-7, 37-39}.

Porém, as indicações restritas para trabalhos protéticos em dentes anteriores fez com que mais estudos fossem desenvolvidos, ao longo dos anos, buscando ampliar as indicações das próteses livres de metal, principalmente na região posterior em que se exige maior esforço mastigatório. A partir daí, surgiram às cerâmicas a base de dissilicato de lítio e infraestruturas à base de óxido de zircônia e mais recentemente com a incorporação de partículas de óxido de ítrio ⁸⁻⁹.

Para o dentista clínico a escolha de qual sistema cerâmico utilizar não se constitui como o único fator para garantir longevidade e resistência às próteses. A cimentação representa uma etapa clínica de significativa importância nas restaurações com sistemas cerâmicos livres de metal ²⁹⁻³³.

O tratamento de superfície previamente à cimentação adesiva varia com relação ao sistema cerâmico utilizado. Alguns autores classificam as cerâmicas em ácido-resistente e as ácido-sensíveis em função do tratamento de superfície com ácido fluorídrico ¹⁰⁻¹⁴.

Os métodos de tratamento de superfície mais utilizados ao longo dos anos foram o jateamento das restaurações com partículas de óxido de alumínio em diferentes tamanhos, o

condicionamento com ácido fluorídrico em diferentes concentrações e posteriormente a aplicação do agente silano ¹⁴⁻¹⁹.

As restaurações cerâmicas à base de óxido de zircônia são denominadas cerâmicas ácido-resistentes, não sendo passíveis de tratamento com ácido fluorídrico. Desta forma, o jateamento com partículas de óxido de alumínio tem demonstrado bons resultados em pesquisas, proporcionando uma retenção micromecânica com os cimentos resinosos ²⁰⁻²¹.

Em contra partida alguns trabalhos demonstram que em diferentes períodos de armazenagem e termociclagem o jateamento com partículas de óxido de alumínio não tem sido eficaz ^{30,34-35}. Com o objetivo de conferir maior longevidade e aperfeiçoar os resultados clínicos da cimentação adesiva, tem sido utilizado o jateamento com partículas de óxido de alumínio modificadas por sílica, denominado de silicatização ²²⁻²⁵. Este tratamento de superfície aumenta a capacidade de união com o silano, buscando não só retenção micromecânica como química com o cimento resinoso ²⁶⁻³⁰.

Desta forma, é importante o cirurgião dentista elencar no seu planejamento reabilitador as etapas de tratamento da superfície das cerâmicas e a sua cimentação. Estabelecer uma relação entre sistema cerâmico utilizado, método de tratamento de superfície e filosofia de cimentação e seleção do cimento irão promover longevidade ao tratamento protético e satisfação ao paciente.

CONSIDERAÇÕES FINAIS

O sucesso de um tratamento com próteses livres de metal utilizando-se as cerâmicas odontológicas depende da seleção do sistema cerâmico de acordo com suas propriedades físico-químicas e resistência mecânica.

Realizar um protocolo clínico de cimentação bem elaborado, observando-se o tratamento de superfície da cerâmica, o preparo do substrato dental e eleição de um cimento resinoso corretamente irão proporcionar mais longevidade às restaurações e próteses cerâmicas.

REFERÊNCIAS

- 1- Walton TR. A 10-year longitudinal study of fixed prosthodontics: clinical characteristics and outcome of single unit metal-ceramic crowns. **Int J Prosthodont.**1999;12(6):519-26.
- 2- Mehulic M, Mehulic K, Kos P, Komar D, Katunaric M. Expression of contact allergy in undergoing prosthodontic therapy patients with oral diseases. **Minerva Stomatol.**2005;54(5):303-9.
- 3- Fradeani M, Redemagni M. An 11-year clinical evaluation of leucite reinforced glass-ceramic crowns: a retrospective study. **Quintessence Int.**2002;33(7):503-10.
- 4- Luthy H, Filser F, Loeffel O, Schumacher M, Gaucker LJ, Hammerle CHF. Strength and reliability of four-unit all-ceramic posterior bridges. **Dent Mater.**2005;21(10):930-7.
- 5- Mc Laren EA, Giordano RA. Zirconia-based ceramics: Material properties, esthetics, and layering techniques of new veneering porcelain, VM9. **QTD.** 2005: 99-111.
- 6- Atsu SS, Kilicarlsan MA, Kucukesmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. **J Prosthet Dent.**2006;95(6):430-36.
- 7- Griggs JA. Recent advances in materials for all- ceramic restorations. **Dent Clin North Am.**2007;51(3):713-27.
- 8- Manicone PF, Rossi Iommetti P, Raffaelli L. An overview of Zirconia ceramics: Basic properties and clinical applications. **J Dent.**2007; 35(11):819-26.
- 9- Della Bona A, Melcholsky JJ Jr, Barret AA, Griggs JA. Characterization of glass-infiltrated alumina-based ceramics. **Dent Mater.** 2008; 24(11):1568-74.
- 10- Valandro LF, Della Bona A, Bottino MA, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. **J Prosthet Dent.** 2005;93(3):253-9.
- 11- Kiyan VH, Saraceni CH, da Silveira BL, Aranha AC, Eduardo Cda P. The influence of internal surface treatments on tensile bond strength for two ceramic systems. **Oper Dent.**2007;32(5):457-65.
- 12- Borges GA, Sophr AM, de Goes MF, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. **J Prosthet Dent.**2003;89(5):479-88.
- 13- Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. **Int J Prosthodont.**2005;18(1):60-5.

- 14- Kamada K, Yoashida K, Atsuta M. Effect of ceramic surface treatments on the bond of four resin luting agents to a ceramic material. **J Prosthet Dent.**1998;79(5):508-13.
- 15- Spohr AM, Sobrinho LC, Consani S, Sinhoreti MAC, Knowles JC. Influence of surface conditions and silane agent on the bond of resin to IPS Empress 2 ceramic. **Int J Prosthodont.** 2003;16(3):277-82.
- 16- Guler AU, Yilmaz F, Ural Ç, Guler E. Evaluation of 24-hour shear bond strength of resin composite to porcelain according to surface treatment. **Int J Prosthodont.**2005;18(2):156-60.
- 17- Della Bona A, Donassollo TA, Demarco FF, Barrett AA, Mecholsky JJ Jr. Characterization and surface treatment effects on topography of a glass-infiltrated alumina/zirconia-reinforced ceramic. **Dent Mater.**2007; 23(6):769-75.
- 18- Spohr AM, Borges GA, Júnior LH, Mota EG, Oshima HM. Surface modification of In-Ceram Zirconia ceramic by Nd: YAG laser, Rocate System, or aluminium oxide sandblasting and its bond strength to a resin cement. **Photomed Laser Surg.**2008;26(3):203-8.
- 19- Chaiyabutr Y, Mc Gowan S, Phillips KM, Kois JC, Giordano RA. The effect of hydrofluoric acid surface treatment and bond strength of a zirconia veneering ceramic. **J Prosthet Dent.**2008;100(3):194-202.
- 20- Guazzato M, Albarkry M, Quach L, Swain MV. Influence of surface and heat treatments on the flexural strength of a glass-infiltrated alumina-zirconia-reinforced dental ceramic. **Dent Mater.**2006;21(5):454-63.
- 21- Akgungor G, Sen D, Aydin M. Influence of different surface treatments on the short-term bond strength and durability between a zirconia post and composite resin core material. **J Prosthet Dent.**2008;99(5):388-99.
- 22- Peutzfeldt A, Asmussen E. Silicoating. Evaluation of a new method of bonding composite resin to metal. **Scand J Dent Res.**1988;96(2):171-6.
- 23- Özcan M. The use of chairside silica coating for different dental applications. **J Prosthet Dent.**2002;87(5):469-72.
- 24- Özcan M, Vallitu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramic. **Dent Mater.**2003;19(8):725-31.
- 25- Della Bona A, Borba M, Beneti P, Ceccheti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. **Braz Oral Res.**2007;21(1):10-15.
- 26- Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. **Quintessence Int.**2007;38(9):745-53.

- 27- de Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to densely sintered zirconium-oxide ceramic. **Dent Mater.**2009;25(2):172-9.
- 28- Re D, Augusti D, Sailer I, Spreafico D, Ceruti A. The effect of surface treatment on the adhesion of resin cements to Y-TZP. **Eur J Esthet Dent.**2008;3(2):186-96.
- 29- Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. **J Prosthet Dent.**2003;89(3):268-74.
- 30- Piwowarczyk A, Bender R, Ottl P, Lauer HC. Long-term bond between dual-polymerizing cementing agents and human hard dental tissue. **Dent Mater.**2007;23(2):211-17.
- 31- Hilgert LA, Monteiro Jr S, Vieira LCC, Gerney W, Edelhoff D. A escolha do agente cimentante para restaurações cerâmicas. **Clínica- Int J Braz Dent.**2009;5(2):194-205.
- 32- Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. **J Adhes Dent.** 2008;10(4):251-8.
- 33- Behr M, Rosentritt M, Regnet T, Lang R, Handel G. Marginal adaptation in dentin of self-adhesive universal resin cement compared with well-tried systems. **Dent Mater.**2004;20(2):191-7.
- 34- Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning method. **Dent Mater.**2007;23(1):45-50.
- 35- Kumbuloglu O, Lassila LV, User A, Vallittu PK. Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. **Oper Dent.**2006;31(2):248-55.
- 36- Della Bona A. **Adesão às Cerâmicas.** Artes Médicas, São Paulo, 2009.
- 37- Hilgert LA, Schweiger J, Beuer F, Andrada MAC, Araújo E, Edelhoff D. Odontologia Restauradora com Sistemas CAD/CAM: O Estado Atual da Arte Parte 1: Princípios de Utilização. **Clínica – Int J Braz Dent.** 2009;5(3):294-303.
- 38- Hilgert LA, Schweiger J, Beuer F, Andrada MAC, Araújo E, Edelhoff D. Odontologia Restauradora com Sistemas CAD/CAM: O Estado Atual da Arte Parte 2: Possibilidades Restauradoras e Sistemas CAD/CAM. **Clínica – Int J Braz Dent.** 2009;5(4):424-35.
- 39- Hilgert LA, Schweiger J, Beuer F, Eichberger M, Andrada MAC, Araújo E, Edelhoff D. Odontologia Restauradora com Sistemas CAD/CAM: O Estado Atual da Arte Parte 3: Materiais Restauradores para Sistemas CAD/CAM. **Clínica – Int J Braz Dent.** 2010;6(1):86-96.

3 CAPÍTULO 2

3.1 Characterization and influence of surface treatments on topography and bond strength of luting agents to a densely-sintered zirconium-oxide ceramic

Leonardo Gonçalves Cunha

Frederico dos Reis Goyatá

Jack Liborio Ferracane

Lais Regiane da Silva-Concilio

Ana Christina Claro Neves

Abstract

Objectives: the aim of the present study was to evaluate the effect of surface treatments on the roughness and bond strength of cements to zirconium-reinforced ceramic substrates. **Methods:** Forty square-shaped zirconium-oxide ceramic blocks (Lava Zirconia, 3M-ESPE) were treated as follows: (C) polished, with no surface treatment (control); (HF) etching using 9.5% hydrofluoric acid for 1 minute; (SB) sandblasting using 110 μ m aluminum oxide particles, (SC) blasting using 110 μ m aluminum oxide particles modified by silica (silica coating), and (AP) sandblasting using 110 μ m aluminum oxide particles and one coat of an alloy primer. Two luting agents were individually applied on the treated ceramic surface: self-adhesive (RelyX U100 - U100 - 3M-ESPE) and dual cure (RelyX ARC - ARC - 3M-ESPE). After 24 hours, each sample was submitted to microshear using speed of 1.0mm/min, and the failure mode of the ceramic-cement interface was evaluated in a stereomicroscope. Roughness was measured using a profilometer (Ra). Two additional samples of each surface treatment were confectioned to atomic force microscope (AFM) for qualitative analyses. Results were submitted to ANOVA and Tukey test ($p=0.05$). **Results:** to the bond strength, results ranged from 10.82 (HF) to 25.66 MPa (AP) for ARC. AP and SC were both statistically superior to the other treatments, and HF was associated with a statistically lower mean value compared to the other groups. For U100, results ranged between 11.45 (HF) and 25.87 MPa (SC). SC and HF were related to mean values of bond strength statistically higher and lower compared to the other methods, respectively. Roughness mean values varied from 0.07 (C) to 0.85 μ m (SC). In a general way, statistical differences of roughness were observed among all surface treatments, as also confirmed qualitatively on AFM images. **Conclusion:** ceramic surface treatments presented significant influence on

the bond strength of the cement-ceramic interface. In this sense, silica coating and sandblasting plus alloy primer were associated with the best treatments of the ceramic surface.

INTRODUCTION

The introduction of computer-aided design/computer-aided manufacture (CAD/CAM) systems in Dentistry promoted a significant increase of all-ceramic restorations. This technology enabled the dental laboratory to control the fabrication process, allowing the production of high quality rehabilitations in a known production schedule, and also reducing technicians working time.

Zirconium-oxide represents one of the main particles used to reinforce ceramic substrates submitted to the CAD/CAM process. Such popularity is related to superior physical properties, such as high flexural strength, biocompatibility, and esthetics.¹⁻² In addition, short-term clinical trials and lifetime predictions reveal favorable success rates for zirconia restorations,³ with an acceptable marginal fit of zirconia rehabilitations confectioned by this milling system.^{4,5} However, many doubts remain about the cementation process between the luting agent and the ceramic substrate.

The achievement of reliable adhesion to ceramics requires surface pre-treatments. Bonding to glass-ceramics normally is obtained by etching with hydrofluoric acid. This step promotes dissolution of the glassy phase, creating a rough surface, which favors adherence that relies on mechanical interlocking.⁶ However, the zirconia surface, due to its chemical properties, cannot be modified by hydrofluoric acid etching.⁷ As a consequence, alternative conditioning methods have

been proposed. Some previous studies reported that airborne abrasion may increase the surface area, resulting in acceptable micrometer scale roughness facilitating resin/ceramic micromechanical interlocks formation.⁸⁻⁹ However, no chemical alteration is achieved in the ceramic surface using this treatment.

In addition, the use of silane coupling agents to enhance the bond of resin composites to silica-based ceramics is well accepted in the dental literature.¹⁰ However, due to the low content of silica (below 1%) in the chemical structure of zirconia, this kind of treatment does not affect adhesion to zirconia.¹¹ In this sense, silica coating the ceramic surface seems to be a promising method to promote the bonding of acid-resistant ceramics to resin. It has been suggested that this surface treatment can increase the silica content on ceramics and metals enhancing the bond to resins via silane agent.¹²⁻¹⁴

Alternative surface treatments have also been proposed. Among them, the current evidence suggests surface treatment by airborne-particle abrasion with aluminum oxide particles and modified priming that contains special adhesive monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) to achieve a satisfactory and long-term durable resin bond to high-strength ceramic materials, such as zirconia ceramics.

Therefore, the aim of the present study was to evaluate the effect of surface treatments on the roughness and bond strength of cements to zirconium-reinforced ceramic substrates. The tested hypothesis was that the surface treatment can influence the topography of the ceramic surface and the bond strength of luting agents to the ceramic substrate.

MATERIALS AND METHODS

A densely sintered zirconia ceramic (Lava, 3M-ESPE, St. Paul, MN, USA) samples were used in the present study. Forty blocks were confectioned following manufacturer's instructions, with dimensions of 5 x 5 x 2 mm.

Ceramic blocks were individually embedded in acrylic resin (Vipi Flash, Vipi, São Paulo, Brazil) using a standard cylindrical silicon matrix (Arotec Ind e Com, São Paulo, Brazil). After curing, the surface of the ceramic blocks was wet-ground with aluminum oxide sandpaper granulation 600, 1200, and 1500 grit in a horizontal Polishing Machine (Aropol, Arotec Ind e Com, São Paulo, Brazil) and all blocks were cleaned in ultrasonic equipment for 5 minutes.

After that, ceramic blocks were randomly divided into five groups (n=4), according to the following surface treatments:

Group CT: no surface treatment (control).

Group HF: 9,5% hydrofluoric acid (Ultradent porcelain etch, Ultradent Dental Products, South Jordan, UT, USA) applied for 1 min, and ultrasonic cleaning for 5 minutes.

Group SB: sandblasting using 110 μ m aluminum oxide particles for 10 s, applied perpendicularly to the surface at a 10-mm distance and pressure of 30psi, and ultrasonic cleaning for 5 minutes.

Group SC: silica coating using particles of 110 μ m (Rocatec, 3M-ESPE, St Paul, USA). The Rocatec-Sand was blasted for 10s applied perpendicularly to the surface at a 10-mm distance and pressure of 30psi

Group AP: sandblasting using 110 μ m aluminum oxide particles for 10s using the same parameters as samples in the SB groups. After sandblasting, samples were ultrasonic cleaned for 5 minutes, and one coat of a metallic primer (Clearfil Alloy Primer, Kuraray Ind., Tokyo, Japan) was applied for 1 min.

After surface treatments, one coat of a silane agent (RelyX Ceramic Primer, 3M-ESPE, St Paul, USA) was applied to all ceramic blocks, except to the blocks of Group AP, in which the specific silane agent was used, as described above. After silane application, a period of 60 seconds was waited.

In the next step, one coat of adhesive agent (Scotchbond Multi Purpose, 3M-ESPE, St Paul, USA) was applied, gently dried and light cured using a LED curing unit (Elipar FreeLight 2, 3M-ESPE, St Paul, USA) for 10 s at irradiance of 1200 mW/cm².

Acrylic plastic tubes (1,6mm internal diameter and 4,0mm in height) were used to the confection of the luting agent samples. Four tubes were positioned in each treated ceramic surface, and then filled with one of two luting agents tested: an adhesive resin cement (Rely X ARC, 3M-ESPE, St Paul, USA), or a self-adhesive cement (Rely X U100, 3M-ESPE, St Paul, USA). Each cement was used following the manufacturer's instructions.

Each cement sample was cured for 40 seconds using the same curing unit (Elipar FreeLight 2) at irradiance of 1200 mW/cm², constantly checked using a radiometer (RD7, Ecel, Ribeirão Preto, Brazil), with a total of 16 samples to each group.

After that, samples were stored in distilled water (37⁰C) for 24 hours. At the end of this period, a microshear bond strength test was performed at a crosshead speed of 1mm/min in a universal testing machine (Versat 2000, Panambra, São Paulo, Brazil). Results were obtained in Kgf and converted to MPa by dividing the failure load by the cross-sectional area of each sample.

Results were submitted to normality test, and after that to ANOVA and post-hoc Tukey test with significance level of 5%.

Failure mode evaluation

The analysis of the debonded surfaces of all samples was performed by two blinded examiners over all bonding area using an Optical Stereomicroscope (VEB Leipzig, Germany) under magnification of 100x. Images were analyzed using specific software (VEB Jena, Germany) and classified as to the characteristic of failure, as follows: cohesive failure in ceramic, cohesive failure in the luting agent, adhesive failure, or mixed (adhesive and cohesive in cement). When there was disagreement during an evaluation, the two examiners made the decision by consensus.

Surface Roughness

To each surface treatment evaluated, a total of eight samples were confectioned, with dimensions of 4x2 mm.

Initially, all ceramic samples were submitted to selective worn using aluminum oxide sandpaper 600, 1200 e 1500 in a horizontal Polishing Machine (Aropol, Arotec, São Paulo, SP, Brazil) to standardize the surface roughness. After

that, the same surface treatments described above were performed in the surface of the ceramic. To these samples, no adhesive procedure was performed. After that, samples were individually positioned in a SurfTest SV-600 profilometer (Mitutoyo, São Paulo, SP, Brazil) to the measurement of surface roughness (Ra) values. Three readings in different parts of the sample were performed on each surface using a stylus tip (2 μ m in diameter). The extension of each reading was 1.25mm, using a cut-off of 0.8 μ m.

Results were submitted to normality test, and after that to ANOVA and post-hoc Tukey test with level significance 5%.

Atomic Force Microscope (AFM)

Furthermore, two additional samples of each surface treatment were qualitatively examined using Atomic Force Microscope (AFM).

A Multimode Atomic Force Microscope (AFM Nanoscope IIIa- Veeco Instruments, Santa Barbara, CA, USA) was used in contact mode. The force between the AFM tip and the sample surface was kept constant, and the vertical piezoelectric ceramic movement was recorded. Images of 512 X 512 pixels were acquired with a scan size of 10 μ m X 10 μ m and scan rate of 2.03 Hz. An NP-type V-shape Si₃N₄ cantilever (Veeco Instruments, Santa Barbara, CA, USA) with normal bending constant of K=0.075 N/m and a tip radius approximately 100nm was used. During imaging, the set point was chosen to be 2.0 volts higher than the top-bottom laser photo detector output obtained when the tip was out of surface contact.

RESULTS

Bond Strength

Bond strength results and failure mode are show in Table 1.

To the adhesive resin cement, mean values ranged from 10.82 MPa (HF) to 25.66 MPa (AP). HF was associated with a statistically lower mean value of bond strength, followed by CT (18.82 MPa) and SB (18.72 MPa), in which no statistical difference was observed. SC (25.28 MPa) and AP were related to statistically higher mean values of bond strength, compared to the other groups.

To the self-adhesive cement, mean values ranged between 11.45 MPa (HF) and 25.87 MPa (SC). The same order was observed, being HF associated with the lowest mean value of bond strength, followed by CT (18.80 MPa) and SB (19.90 MPa), in which no statistical difference was observed. However, AP (22.12 MPa) showed a statistically lower mean value compared to SC, the latter related to the highest mean value of bond strength, compared to the other groups.

Comparing both luting agents, no difference was observed between them to all surface treatments evaluated, exception to the AP group, in which the adhesive resin cement (25.66 MPa) was associated with statistically higher mean values of bond strength, compared to the self-adhesive cement (22.12 MPa).

No cohesive failure in the ceramic block or in the luting agent was observed. Adhesive failure mode was the most common type to all groups in study, being the only mode of failure to CT, to both luting agents. AP (6) and SC (5) presented a higher prevalence of mixed failure with the adhesive resin cement, compared to the other groups.

Surface Roughness and AFMs

The surface roughness results are show in Table 2.

Mean values ranged between 0.07 μm (CT) and 0.85 μm (AP). CT (0.07 μm) was associated with a statistically lower mean value of roughness, followed by HF (0.25 μm). AP (0.41 μm) and SB (0.46 μm) presented intermediate mean values, with no statistical differences between themselves. SC (0.85 μm) was related to a statistically higher mean value of surface roughness, compared to the other groups.

AFM images (Figure 1) confirmed the mean values obtained of surface roughness (Table 2). CT promoted the smoothest surface, and SC a more regular pattern of roughness, in comparison to the others.

Discussion

Although zirconium oxide ceramics are able to withstand fracture loads showing optimum mechanical properties, the clinical success is also dependent on a reliable bond formation within the luting agent.⁷ It has been suggested that, even if the clinical success of a restoration does not rely on the resin bond to the tooth, successful resin bonding can improve retention,¹⁵ marginal adaptation,¹⁶ and fracture resistance of the restoration.¹⁷ Therefore, it can be concluded that adhesive cementation procedures are necessary to support all-ceramic materials. In this sense, creating an effective micromechanical ceramic surface is crucial for an adequate adhesive bond and/or repair of ceramic restorations.¹⁸ Resin penetration and polymerization into this treated surface produce the most important adhesion mechanism of the resin–ceramic systems.¹⁸⁻¹⁹

The clinical protocol used to bond glassy ceramics using resin luting agents is well established in the literature based on the silane coupling agent interaction with the silica present in this kind of ceramic. However, unlike conventional dental ceramics, polycrystalline material zirconia ceramics contains almost no glass. In this sense, traditional surface treatments, such as the acid etching technique, are not effective to the zirconia ceramics.

From the results of the present study, it was observed that acid etching technique, instead of has no effect on the bond strength of the ceramic-luting agent interface, as observed in previous studies⁷, promoted a reduction of this value. Compared to the control group, in which the ceramic surface was polished and submitted to no surface treatment, acid etching caused a reduction of almost 40% in the mean bond strength value, from 18.8 MPa (control group) to 11.45 MPa (acid etching group). A possible explanation for this reduction is related to a preference of the HF to etch the glassy phase of the ceramic, usually silica-based phases, creating a retentive surface.²⁰⁻²¹ However, zirconia ceramic contains a small amount of a lanthanum oxide-based glassy matrix, which hinders the HF etching action.¹⁴ This situation could be also seen in the AFM images (Figure 1). Compared to the control group (letter A), in which a smooth and regular surface is observed, etching using hydrofluoric acid (letter B) caused a degradation of the ceramic surface, compromising the bonding between the resin luting agent and the ceramic, thus, decreasing the bond strength values. This situation could be also observed in the roughness measurement, in which HF etching caused an increase of the mean Ra value of four times, from 0.07 μm (control) to 0.25 μm (HF). Therefore, this type of surface treatment should be avoided to zirconia ceramics.

Laboratory and in-office airborne particle abrasion systems have also been tested and applied clinically. These systems generally use two types of abrasives: aluminum oxide particles and silica modified alumina particles (silica coating systems). In the present study, aiming to compare the effectiveness of these systems, the same particle size (110 μm) was used to both blasting.

Sandblasting using aluminum oxide particles was effective to change the surface of the ceramic substrate, as observed by the increase of the mean roughness value associated with this surface treatment, 0.46 μm , a mean value similar to 650% higher to the one found to the control group (0.07 μm). This surface alteration could be also qualitatively observed in the AFM images (Figure 1), showing a rough surface to the ceramics substrates submitted to this treatment (letter C). However, instead of increasing quantitatively and qualitatively the roughness of the ceramic surface, this type of treatment was ineffective to improve bond strength. No difference was observed between control (18.82 MPa) and sandblasting groups (18.72 MPa). It has to be pointed out that control group samples were submitted to polishing before cementation procedures, so all steps related to the cementation were performed in a very smooth surface in this group. Similar results were also observed by previous studies.^{7,17,20,29,34} A possible explanation to the low effectiveness of sandblasting is that this technique produces an irregular pattern of roughness and, consequently, ceramic wettability occurs in a diffuse mode.

It has also been suggested that the association of mechanical (via micro-retention using sandblasting) and chemical (via silica coating or MDP-containing silane) adhesive mechanisms produce better bond strengths of high crystalline content ceramics bonded to resins.^{12,24-25}

In this sense, the goal of the silica coating systems is to promote a retentive surface and to deposit a silica layer on ceramic and metal surfaces.^{7,12} The tribochemical reaction associated with this treatment produces a high temperature contact area that can hold the blasted particles and/or the silica layer on the ceramic surface,²² promoting resin bonding via silane agents, enhancing the mechanical and chemical bonding,^{12,23} as observed in the present study. From the analyses of roughness, blasting using Rocatec was associated with the highest mean value (0.85 μm), statistically superior to all others treatments. Besides that, the pattern of roughness created was very regular, as can be seen by the AFM images (Figure 1E), significantly different from the others. In addition to these advantages, the silica layer created on the ceramic surface allowed a chemical bond between the ceramic and the silane agent.^{11,23} A previous study¹⁴ concluded that the silica coated ceramic surface showed a significant increase (76%) in the concentration of surface silicon. This superficial silica content contributes to the bond strength by promoting a chemical bond to resinous materials via cross-linkages with methacrylate groups, also increasing the substrate surface energy and improving the surface wettability to resin.^{11,24} Indeed, bond strength was found to be significantly higher in the Rocatec group (25.28 MPa and 28.87 MPa to ARC and U100, respectively), when compared to the other groups, except AP to ARC, which indicates that silica coating system would improve the bonding of zirconium-oxide ceramic restorations.

In the present study, treatment with a MDP-containing silane coupling agent increased the bond strength of zirconium ceramic when applied over surface that had been airborne-particle abraded. This type of treatment reached mean bond strength values of 25.66 MPa and 22.12 MPa, to ARC and U100, respectively, superior to all tested methods, except to the Rocatec groups. These results can be explained by the

improved surface wettability, also forming cross-linkages with methacrylate groups as well as siloxane bonds with the OH groups of the ceramic substrate.²⁶ Such reaction may be promoted and sustained by the acidity of the substrate treated with the coupling solution. A relatively strong poly-molecular layer may be responsible for the ceramic–resin cement bond.²⁶

The results of shear bond strength test of the present study are consisted with the failure modes observed: groups C, HF and SB showed lower bond strength mean values and adhesive type failures as the most common type of failure mode, whereas the groups with higher bond strength mean values (Roc and AP) showed an increased occurrence of mixed type of failure in the interface ceramic-resin luting agent. No cohesive failure was observed, to all groups, showing the effectiveness of the microshear test performed to evaluate the bond strength of the adhesive interface.

In addition, luting cement selection seems to be a relevant factor when bonding to zirconium oxide ceramics. In the present study, two types of luting agents were tested: conventional dual cure resin cement and self-adhesive cement. Both luting agents reached very similar results to all surface treatment groups, proving the effectiveness of the self-adhesive cement to reach satisfactory results of bond strength, when compared to well-established dual cure resin cement. The only exception was observed in the AP group, in which U100 (22.12 MPa) was associated with a statistically lower mean value of bond strength compared to the ARC (25.66 MPa). A possible explanation for this situation is that the MDP-containing silane coupling agent may have acted as a prejudicial material to the U100 cement, decreasing the interaction between the self-adhesive cement to the ceramic surface.

In conclusion, the tested hypothesis that the surface treatment can influence the topography of the ceramic surface and the bond strength of luting agents to the ceramic substrate was accepted by the results. It was possible to see that, considering high crystalline content ceramics, mechanical retention is not enough to reach a satisfactory bond strength. The association of mechanical and chemical procedures is essential to the effectiveness of the luting procedure. However, the longevity of the interfaces created by these surface treatments should be further evaluated.

References

1. Sadan A, Blatz MB, Lang B. Clinical considerations for densely sintered alumina and zirconia restorations: Part 1. *Int J Periodontics Restorative Dent* 2005; 25:213-219.
2. Sadan A, Blatz MB, Lang B. Clinical considerations for densely sintered alumina and zirconia restorations: Part 2. *Int J Periodontics Restorative Dent* 2005; 25:343-349.
3. Fischer H, Weber M, Marx R. Lifetime prediction of all-ceramic bridges by computational methods. *J Dent Res* 2003; 82:238-242.
4. Reich S, Wichmann M, Nkenke E, Proeschel P. Clinical fit of all-ceramic three-unit fixed partial dentures, generated with three different CAD/CAM systems. *Eur J Oral Sci* 2005; 113:174-179.
5. Tinschert J, Natt G, Mautsch W, Spiekermann H, Anusavice KJ. Marginal fit of alumina and zirconia-based fixed partial dentures produced by a CAD/CAM system. *Oper Dent* 2001;26:367-374.
6. Edris A, Jabr A, Cooley RL, Barghi N. SEM evaluation of etches patterns by three etchants on three porcelains. *J Prosthet Dent* 1990;64:734-739.
7. Derand P, Derand T. Bond strength of luting cements to zirconium oxide ceramics. *Int J Prosthodont* 2000;13:131-135.
8. Kumbuloglu O, Lassila LVJ, User A, Vallitu PK. Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. *Oper Dent* 2006;31:248-255.
9. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dent Mater* 2007;23:45-50.

10. Hayakawa T, Horie K, Aida M, Kanaya H, Kobayashi T, Murata Y. The influence of surface conditions and silane agents on the bond of resin to dental porcelain. *Dent Mater* 1992;8:238-240.
11. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003;89:268-274.
12. Valandro LF, Della Bona A, Bottino MA, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. *J Prothet Dent* 2005;93:253-9.
13. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater* 1998;14:64-71.
14. Della Bona A, Donassolo TA, Demarco FF, Barret AA, Mecholsky Jr JJ. Characterization and surface treatment effects on topography of a glass-infiltrated alumina/zirconia-reinforced ceramic. *Dent Mater* 2007;23:769-775.
15. Burke FJ, Fleming GJ, Nathanson D, Marquis PM. Are adhesive technologies needed to support ceramics? An assessment of the current evidence. *J Adhes dent* 2002;4:7-22.
16. Rosenstiel SF, Land MF, Crispin BJ. Dental luting agents: a review of the current literature. *J Prosth Dent* 1998;80:280-301.
17. Burke FJ. Fracture resistance of teeth restored with dentin-bonded crowns: the effect of increased tooth preparation. *Quintessence Int* 1996;27:115-121.
18. Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. *J Adhes Dent* 2000;2:305-13.
19. Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. *Int J Prosthodont* 2005;18:60-5.

20. Della Bona A, Anusavice KJ, Mecholsky Jr JJ. Failure analysis of resin composite bonded to ceramic. *Dent Mater* 2003;19:693–9.
21. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithium disilicate-based ceramic. *Dent Mater* 2004;20:338–44.
22. Haselton DR, Diaz-Arnold AM, Dunne JT Jr. Shear bond strengths of 2 intraoral porcelain repair systems to porcelain or metal substrates. *J Prosthet Dent*. 2001;86(5):526-31.
23. Özcan M, Vallitu PK. Effect of surface conditioning methods on the bond strength of luting cements to ceramics. *Dent Mater* 2003;19:725-731.
24. Sun R, Suansuwan N, Kilpatrick N, Swain M. Characterization of tribochemically assisted bonding of composite resin to porcelain and metal. *J Dent* 2000;28:441-445.
25. Özcan M, Alkumru HN, Gemalmaz D. The effect of surface treatment on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic. *Int J Prosthodont* 2001;14:335-9.
26. Yoshida K, Tsuo Y, Astuta M. Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *J Biomed Mater Res* 2006;77B:28–33.

Table 1. Mean bond strength values (MPa) and failure mode to each surface treatment and luting agent

Surface Treatment	ARC		U100		ARC		U100	
	Failure Mode							
	Adhesive		Mixed		Adhesive		Mixed	
Control	18.82 (2.22) A, b	18.80 (1.65) A, c	16	0	16	0		
Hydrofluoric Acid	10.82 (1.82) A, c	11.45 (1.93) A, d	15	1	15	1		
Sandblasting	18.72 (1.95) A, b	19.90 (3.30) A, c	15	1	14	2		
Sandblasting + Alloy Primer	25.66 (1.46) A, a	22.12 (1.89) B, b	10	6	13	3		
Silica Coating	25.28 (2.86) A, a	25.87 (2.04) A, a	11	5	13	3		

Mean values followed by different small letters in the same column and different capital letters in the same row differ statistically among themselves for the Tukey test at the level of 5%. () – Standard Deviation

Table 2. Mean roughness values (Ra, μm) to each surface treatment

Surface Treatment	Roughness
Control	0.07 (0.03) d
Hydrofluoric Acid	0.25 (0.05) c
Sandblasting	0.46 (0.08) b
Sandblasting + Alloy Primer	0.41 (0.07) b
Silica Coating	0.85 (0.19) a

Mean values followed by different small letters differ statistically among themselves for the Tukey test at the level of 5%. () – Standard Deviation

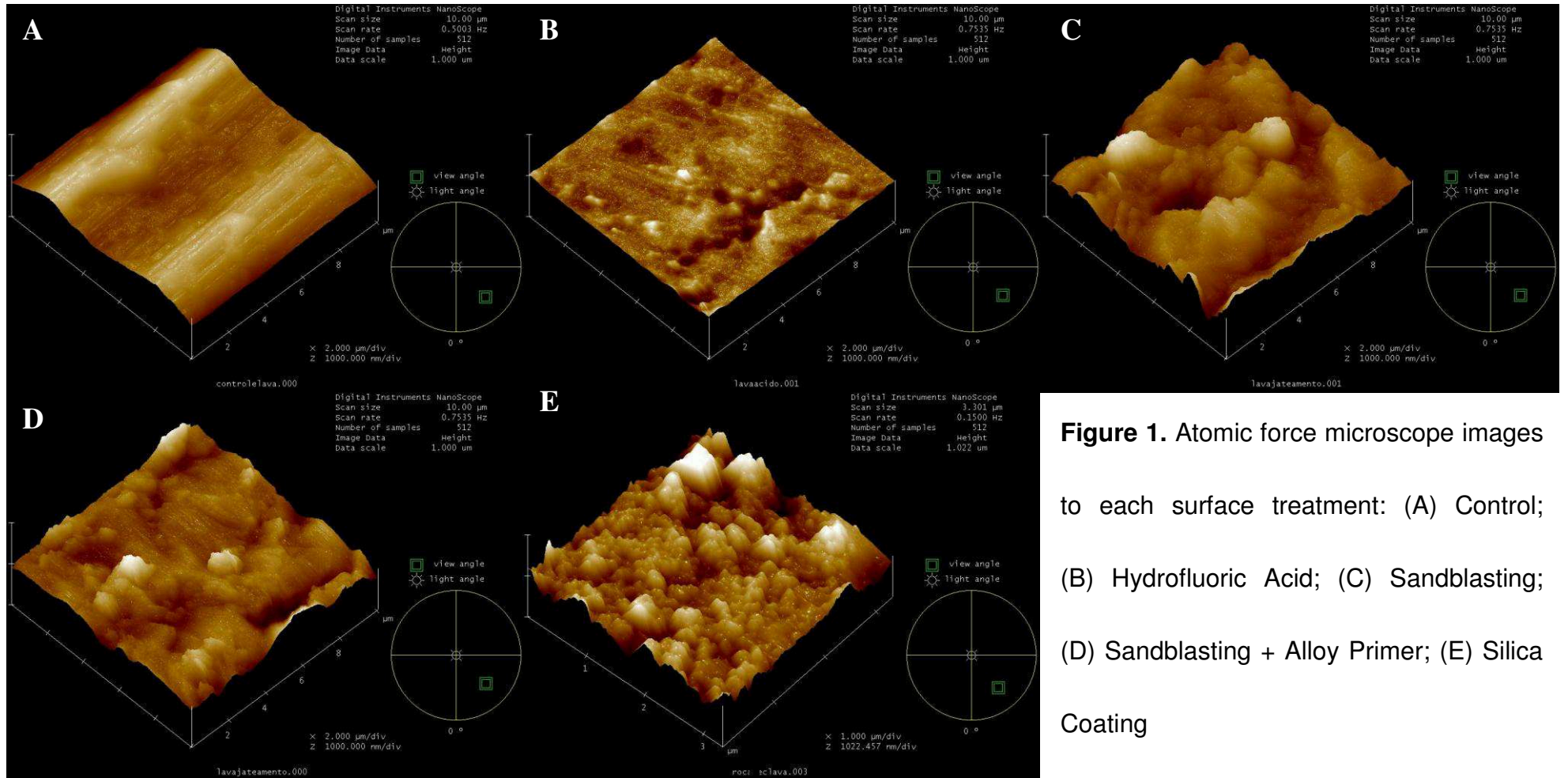


Figure 1. Atomic force microscope images to each surface treatment: (A) Control; (B) Hydrofluoric Acid; (C) Sandblasting; (D) Sandblasting + Alloy Primer; (E) Silica Coating

4 CAPÍTULO 3

4.1 Influence of the surface treatment on the bond strength of resin cements to a densely-sintered zirconium-oxide ceramic after different storage periods

Leonardo Gonçalves Cunha

Frederico dos Reis Goyata

Jack Liborio Ferracane

Lais Regiane da Silva-Concilio

Ana Christina Claro Neves

Abstract

Objectives: the present study aimed to evaluate the influence of surface treatments on the bond strength of two luting agents to a zirconium-reinforced ceramic after different storage periods. **Methods:** Forty square-shaped zirconium-oxide ceramic blocks (Lava Zirconia, 3M-ESPE) were treated as follows: (C) polished, with no surface treatment (control); (HF) etching using 9.5% hydrofluoric acid for 1 minute; (SB) sandblasting using 110 μ m aluminum oxide particles, (SC) blasting using 110 μ m aluminum oxide particles modified by silica (silica coating), and (AP) sandblasting using 110 μ m aluminum oxide particles and one coat of an alloy primer. Two luting agents were individually applied on the treated ceramic surface: self-adhesive (RelyX U100 - U100 - 3M-ESPE) and dual cure (RelyX ARC - ARC - 3M-ESPE). After 24 hours and 180 days of water storage, each sample was submitted to a microshear bond strength test using speed of 1.0mm/min, and the failure mode of the ceramic-cement interface was evaluated in a stereomicroscope. Results were submitted to ANOVA and Tukey test ($p=0.05$). **Results:** after 24 hours, results ranged from 10.82 (HF) to 25.66 MPa (AP) for ARC. AP and SC were both statistically superior to the other treatments, and HF was associated with a statistically lower mean value compared to the other groups. After 180 days, results were between 2.10 (HF) and 23.68 MPa (SC), AP and SC was statistically superior to the other treatments. For U100, results ranged between 11.45 (HF) and 25.87 MPa (SC) after 24 hours. SC and HF were related to mean values of bond strength statistically higher and lower compared to the other methods, respectively. After 180 days, results ranged from 0.59 (HF) and 21.13 MPa (SC). **Conclusion:** ceramic surface treatments presented significant influence on the bond strength of the cement-ceramic interface. In this sense, silica coating and sandblasting plus alloy primer were associated with the best

treatments of the ceramic surface. In a general way, storage promoted a significant reduction of the bond strength values, to both resin luting agents.

INTRODUCTION

In recent years, the increasing demand for all ceramic restorations led to the development of ceramic systems with excellent mechanical properties and superior aesthetic characteristics. In this sense, aluminum-oxide and zirconium-oxide ceramics have been used to the confection of prosthetic restorations with excellent results.

The zirconium-oxide represents one of the main particles used to reinforce ceramic substrates submitted to the CAD/CAM process. This ceramic system is associated with improved characteristics such as superior physical properties, high flexural strength, biocompatibility and esthetics.¹⁻² In addition, short-term clinical trials and lifetime predictions reveal favorable success rates for zirconia restorations,³ with an acceptable marginal fit of this kind of rehabilitation.^{4,5}

Considering a clinical situation of compromised retention or for minimally invasive treatment options that rely on resin bonding, a strong and a long-term durable adhesive bond between the tooth and ceramic material is very important to the clinical survival of the restoration.

In this sense, previous studies investigated the bond strength and the durability of different bonding methods to zirconium-oxide ceramics.⁶⁻⁹ It was concluded that, aiming to obtain a satisfactory adhesion between ceramic and luting cement, a ceramic surface treatment is necessary.

Adhesion to glass-ceramics has been extensively tested, and normally it is achieved by etching the ceramic substrate using hydrofluoric acid.⁶ However, the zirconia surface, due to its chemical properties, cannot be modified by hydrofluoric acid etching.⁷ Airborne abrasion may increase the surface area facilitating cement/ceramic micromechanical interlocks formation because the roughness surface.⁸⁻⁹ However, no chemical alteration is achieved in the ceramic surface using this treatment.

Aiming to modify chemically the ceramic surface, silica coating seems to be a promising method to promote the bonding of acid-resistant ceramics to resin cement.^{6,10} In addition, this type of treatment creates a silica covered surface, which is essential for using a silane coupling agent, aiming to enhance the bond of resin composites to ceramics, as well accepted in the dental literature.¹¹

Other alternative surface treatments have also been proposed. The airborne-particle abrasion with aluminum oxide particles and modified priming that contains special adhesive monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP)¹²⁻¹³ and alloy primers to achieve a satisfactory and long-term durable resin bond to high-strength ceramic materials, such as zirconium-oxide ceramics¹⁴⁻¹⁶.

In addition, different studies evaluated the stability of the adhesive interface after long-term water storage and/or thermal cycling¹⁸⁻²⁰. This is a very important factor, because the longevity of the restorative procedure is directly related to maintenance of the bonded interface.

Therefore, the aim of the present study was to evaluate the effect of ceramic surface treatments and storage on the bond strength of two luting cements to a zirconium-reinforced ceramic. The tested hypotheses were that the surface

treatment and storage can influence the bond strength of luting agents to the ceramic substrate.

MATERIALS AND METHODS

Densely sintered zirconia ceramic (Lava, 3M-ESPE, St. Paul, MN, USA) samples were used in the present study. Forty blocks were confectioned following manufacturer's instructions, with dimensions of 5 x 5 x 2 mm.

Ceramic blocks were individually embedded in acrylic resin (Vipi Flash, Vipi, São Paulo, Brazil) using a standard cylindrical silicon matrix (Arotec Ind e Com, São Paulo, Brazil). After acrylic resin curing, the surface of the ceramic blocks was wet-ground with aluminum oxide sandpaper granulation 600, 1200, and 1500 grit in a horizontal Polishing Machine (Aropol, Arotec Ind e Com, São Paulo, Brazil) and all blocks were cleaned in ultrasonic equipment for 5 minutes.

After that, ceramic blocks were randomly divided into five groups (n=4), according to the following surface treatments:

Group CT: no surface treatment (control).

Group HF: 9,5% hydrofluoric acid (Ultradent porcelain etch, Ultradent Dental Products, South Jordan, UT, USA) applied for 1 min, and ultrasonic cleaning for 5 minutes.

Group SB: sandblasting using 110µm aluminum oxide particles for 10 s, applied perpendicularly to the surface at a 10-mm distance and pressure of 30psi, and ultrasonic cleaning for 5 minutes.

Group SC: silica coating using particles of 110 μ m (Rocatec, 3M-ESPE, St Paul, USA). The Rocatec-Sand was blasted for 10s applied perpendicularly to the surface at a 10-mm distance and pressure of 30psi.

Group AP: sandblasting using 110 μ m aluminum oxide particles for 10s using the same parameters as samples in the SB groups. After sandblasting, samples were ultrasonic cleaned for 5 minutes, and one coat of a metallic primer (Clearfil Alloy Primer, Kuraray Ind., Tokyo, Japan) was applied for 1 min.

After surface treatments, one coat of a silane agent (RelyX Ceramic Primer, 3M-ESPE, St Paul, USA) was applied to all ceramic blocks, except to the blocks of Group AP, in which the specific silane agent was used, as described above. After silane application, a period of 60 seconds was waited.

In the next step, one coat of adhesive agent (Scotchbond Multi Purpose, 3M-ESPE, St Paul, USA) was applied, gently dried and light cured using a LED curing unit (Elipar FreeLight 2, 3M-ESPE, St Paul, USA) for 10 s at irradiance of 1200 mW/cm².

Acrylic plastic tubes (1,6mm internal diameter and 4,0mm in height) were used to the confection of the luting agent samples. Four tubes were positioned in each treated ceramic surface, and then filled with one of two luting agents tested: an adhesive resin cement (Rely X ARC, 3M-ESPE, St Paul, USA), and a self-adhesive cement (Rely X U100, 3M-ESPE, St Paul, USA). To the each cement was following the manufacturer's instructions in the manipulation techniques.

Each cement sample was cured for 40 seconds using the same curing unit (Elipar FreeLight 2) at irradiance of 1200 mW/cm², constantly checked using a

radiometer (RD7, Ecel, Ribeirão Preto, Brazil), with a total of 16 samples to each group.

After that, samples were stored in distilled water (37⁰C) for 24 hours and 180 days. At the end of each period, a microshear bond strength test was performed at a crosshead speed of 1 mm/min in a universal testing machine (Versat 2000, Panambra, São Paulo, Brazil). Results were obtained in Kgf and converted to MPa by dividing the failure load by the cross-sectional area of each sample.

Results were submitted to normality test and after that to ANOVA and post-hoc Tukey test with level significance 5%.

Failure mode evaluation

The analysis of the debonded surfaces of all samples was performed by two blinded examiners over all the area of the bond using an Optical Stereomicroscope (VEB Leipzig, Germany) under magnification of 100x. Images were analyzed using specific software (VEB Jena, Germany) and classified as to the characteristic of failure, as follows: cohesive failure in ceramic, cohesive failure in the luting agent, adhesive failure, or mixed (adhesive and cohesive in cement). When there was disagreement during an evaluation, the two examiners made the decision by consensus.

RESULTS

Bond Strength

Bond strength results are show in Table 1.

After 24 hours storage to the adhesive resin cement, mean values ranged from 10.82 MPa (HF) to 25.66 MPa (AP). HF was associated with a statistically lower mean value of bond strength, followed by CT (18.82 MPa) and SB (18.72 MPa), in which no statistical difference was observed. SC (25.28 MPa) and AP were related to statistically higher mean values of bond strength, compared to the other groups.

After storage of 180 days, to the adhesive resin cement, mean values ranged from 2.10 MPa (HF) to 23.68 MPa (SC). HF presented a statistically lower mean value, followed by CT (13.89 MPa) and SB (18.67 MPa), in which statistical differences were also observed. SC and AP (22.62 MPa) presented the highest mean values, statistically higher to the other groups.

To the self-adhesive cement, mean values ranged between 11.45 MPa (HF) and 25.87 MPa (SC) after 24 hours storage. The same order was observed, being HF associated with the lowest mean value of bond strength, followed by CT (18.80 MPa) and SB (19.90 MPa), in which no statistical difference was observed. However, AP (22.12 MPa) showed a statistically lower mean value compared to SC, the latter related to the highest mean value of bond strength, compared to the other groups.

After storage of 180 days, to the self-adhesive cement mean values ranged between 0.59 MPa (HF) and 21.13 MPa (SC). A statistically lower mean value was observed to HF compared to the others, followed by CT (11.56 MPa). AP (17.37 MPa) and SB (18.51 MPa) showed intermediate mean values, in which no statistical

difference was observed. SC was associated with statistically higher mean values compared to the others.

Comparing both luting agents after 24 hours storage, no difference was observed between them to all surface treatments evaluated, exception to the AP group, in which the adhesive resin cement (25.66 MPa) was associated with statistically higher mean values of bond strength, compared to the self-adhesive cement (22.12 MPa). After 180 days storage, the same scenario was found. AP group in the adhesive resin cement (22.62 MPa) presented a statistically higher mean value compared to the self-adhesive cement (17.37 MPa).

Failure mode results are show in Table 2. No cohesive failure in the ceramic block or in the luting agent was observed. Adhesive failure mode was the most common type to all groups in study, being the only mode of failure to CT, to both luting agents. AP (6) and SC (5) presented a higher prevalence of mixed failure with the adhesive resin cement, compared to the other groups.

After storage of 180 days, adhesive failure was again the most common but the mixed failure has increased in the all groups with prevalence in the self adhesive cement (11 to 25) about the adhesive cement (13 to 20).

Discussion

Zirconium-oxide ceramic restoratives can be luted by conventional techniques using zinc phosphate or glass ionomer cement. However, when mechanical retention and stability of dental preparation requires adhesive

cementation with resin cements, the internal surface of the ceramic should be treated to provide satisfactory retention.⁶⁻⁸

Adhesive cementation procedures are necessary to support all-ceramic materials, and creating an effective micromechanical ceramic surface is important for an adequate adhesive bond of ceramic restorations.⁹⁻¹¹ Resin penetration and polymerization into this treated surface produce the most important adhesion mechanism of the resin–ceramic systems.^{17,26}

Densely sintered ceramics are resistant to acids, therefore, hydrofluoric acid, which successfully etches feldspathic ceramics, have no effect on zirconia ceramic surfaces.^{6,24} Hydrofluoric acid etching, followed by silane application, is known to not provide a strong bond to zirconia ceramics, as demonstrated in this study because the ceramic tested was acid resistant.^{12-14,22}

From the results of the present study was possible to observe a significant influence of the surface treatments and storage conditions on the bond strength values. In the hydrofluoric acid etching groups, storage for six months caused a mean reduction of 520% (from 10.82 MPa after 24 hours to 2.10 MPa after six months) in the bond strength values to ARC and a surprisingly reduction of almost 1950% in the bond strength values to U100 (from 11.45 MPa after 24 hours to 0.59 MPa after six months). Similar results were observed for previous studies.^{9-10,23}

It has to be pointed out that the reduction after the storage period observed for the HF groups was even higher than the one from the control group, in which no surface treatment was performed. The mean reduction of the CT group was 26% to ARC (from 18.8 MPa after 24 hours to 13.89 MPa after six months) and 39% to U100 (from 18.8 MPa after 24 hours to 11.56 MPa after six months). A possible explanation for such results is related to the preference of the HF to etch the glassy phase of the

ceramic, usually silica-based phases, creating a retentive surface.²¹ However, zirconia ceramic contains a small amount of a lanthanum oxide-based glassy matrix, which hinders the HF etching action.²⁵

Laboratory and in-office airborne particle abrasion systems have also been tested and applied clinically. These systems generally use two types of abrasives: aluminum oxide particles and silica modified alumina particles (silica coating systems). In the present study, aiming to compare the effectiveness of these systems, the same particle size (110 μm) was used to both blasting.

Sandblasting using aluminum oxide particles was effective to change the surface of the ceramic substrate. However, instead of increasing the roughness of the ceramic surface, this type of treatment was ineffective to improve bond strength. No difference was observed between control (18.82 MPa) (18.80 MPa) and sandblasting groups (18.72 MPa) (19.90 MPa) to ARC and U110 cements respectively.

After storage of 180 days, sandblasting groups presented no significant reduction of the bond strength values. Similar results were also observed by previous studies.^{9,13,20} A possible explanation to the results of sandblasting after storage is that this technique produces an irregular surface and facilitating cement/ceramic micromechanical interlocks formation because the roughness surface.

It has also been suggested that the association of mechanical (via micro-retention using sandblasting) and chemical (via silica coating or MDP-containing silane) adhesive mechanisms produce better bond strengths of high crystalline content ceramics bonded to resins.^{10,18-20}

The silica coating systems is to promote a retentive surface and to deposit a silica layer on ceramic and metal surfaces.^{7,12} The tribochemical reaction associated

with this treatment produces a high temperature contact area that can hold the blasted particles and/or the silica layer on the ceramic surface,²⁷ promoting resin bonding via silane agents, enhancing the mechanical and chemical bonding²⁶ and created on the ceramic surface allowed a chemical bond between the ceramic and the silane agent.^{11,29} This superficial silica contributes to the bond strength by promoting a chemical bond to resinous materials via cross-linkages with methacrylate groups, also increasing the substrate surface energy and improving the surface wettability to resin.^{11,27}

In the present study, to the SC group (25.28 MPa and 25.87 MPa to ARC and U100, respectively) the mean values was higher when compared to the other groups, except AP group with 24 hours mean values (25.66 MPa to ARC and 22.12 MPa to U100) which indicates that silica coating system would improve the bonding of zirconium-oxide ceramic restorations. After long-term storage, the mean values were a small reduce (23.68 MPa and 21.13 MPa to ARC and U100 respectively) that the confirmed a durability to improved the surface treatment to zirconia ceramic.¹⁷⁻¹⁸

The surface treatment with a MDP-containing silane coupling agent increased the bond strength of zirconium ceramic when applied over surface that had been airborne-particle abraded. This type of treatment reached mean bond strength values of 25.66 MPa and 22.12 MPa, to ARC and U100, respectively, superior to all tested methods, except to the Rocatec groups. However, in long term storage, the results were a small reduce in the mean values (22.62 MPa to ARC and 17.37 MPa to U100). These results can be explained by the improved surface wettability, also forming cross-linkages with methacrylate groups as well as siloxane bonds with the OH groups of the ceramic substrate.¹⁵⁻¹⁶

In the failure mode had a small decreased in the adhesive type failures after 180 days storage to both cements, especially in C, HF and SB groups that showed lower bond strength mean values before and after storage. In the groups with higher bond strength mean values (SC and AP) showed mixed type of failures in the interface ceramic-resin luting agent. After 180 days storage, the mixed failure to the ARC cement not altered, but to the U100 cements has increased to 50% in AP group and 75% SC group. No cohesive failure was observed, to all groups, showing the effectiveness of the microshear test performed to evaluate the bond strength of the adhesive interface.

In addition, luting cement selection seems to be a relevant factor when bonding to zirconium oxide ceramics. In the present study, two types of luting agents were tested: conventional dual cure resin cement and self-adhesive cement. The luting agents reached very similar results in both situations tested: surface treatment and storage for 180 days in distilled water. The effectiveness of the self-adhesive cement to reach satisfactory results of bond strength, when compared to dual cure resin cement.

In conclusion, the tested hypotheses that the surface treatment and storage for 180 days in distilled water can influence the bond strength of luting agents to the ceramic substrate was accepted by the results. The association of mechanical and chemical procedures is essential to the effectiveness and durability of the luting procedure.

References

1. Sadan A, Blatz MB, Lang B. Clinical considerations for densely sintered alumina and zirconia restorations: Part 1. *Int J Periodontics Restorative Dent* 2005; 25:213-19.
2. Sadan A, Blatz MB, Lang B. Clinical considerations for densely sintered alumina and zirconia restorations: Part 2. *Int J Periodontics Restorative Dent* 2005; 25:343-9.
3. Sorensen JA. The Lava system for CAD/CAM production of high-strength precision fixed prosthodontics. *Quintessence Dent Technol* 2004;26:57-67.
4. Reich S, Wichmann M, Nkenke E, Proeschel P. Clinical fit of all-ceramic three-unit fixed partial dentures, generated with three different CAD/CAM systems. *Eur J Oral Sci* 2005; 113:174-9.
5. Tinschert J, Natt G, Mautsch W, Spiekermann H, Anusavice KJ. Marginal fit of alumina and zirconia-based fixed partial dentures produced by a CAD/CAM system. *Oper Dent* 2001;26:367-74.
6. Kiyani VH, Saraceni CH, da Silveira BL, Aranha AC, Eduardo CP. The influence of internal surface treatments on tensile bond strength for two ceramic systems. *Oper Dent* 2007;32:457-65.
7. Valandro LF, Özcan M, Amaral R, Leite FP, Bottino MA. Microtensile bond strength of a resin cement to silica-coated and silanized In-Ceram Zirconia before and after aging. *Int J Prosthodont* 2007;89:268-74.

8. Kumbuloglu O, Lassila LVJ, User A, Vallitu PK. Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. *Oper Dent* 2006;31:248-55.
9. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dent Mater* 2007;23:45-50.
10. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003;89:268-74.
11. Hayakawa T, Horie K, Aida M, Kanaya H, Kobayashi T, Murata Y. The influence of surface conditions and silane agents on the bond of resin to dental porcelain. *Dent Mater* 1992;8:238-40.
12. Valandro LF, Della Bona A, Bottino MA, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. *J Prosthet Dent* 2005;93:253-9.
13. Della Bona A, Donassolo TA, Demarco FF, Barret AA, Mecholsky Jr JJ. Characterization and surface treatment effects on topography of a glass-infiltrated alumina/zirconia-reinforced ceramic. *Dent Mater* 2007;23:769-75.
14. Kitayama S, Nikaido T, Maruoka R, Zhu L, Ikeda M, Watanabe A et al. Effect of an internal technique on tensile bond strengths of resin cements to zirconia ceramics. *Dent Mat J* 2009;28:446-53.
15. Okuya N, Minami H, Kurashige H, Marahara S, Suzuki S, Tanaka T. Effects of metal primers on bonding of adhesive resin cement to noble alloys for porcelain fusing. *Dent Mat J* 2010;29:177-87.

16. Yun JY, Ha SR, Lee JB, Kim SH. Effect of sandblasting and various metal primers on the shear strength of resin cement to Y-TZP ceramic. *Dent Mater* 2010;26:650-8.
17. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, Van Meerbeek B. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005;84:118-32.
18. Lindgren J, Smeds J, Sjogren G. Effects of surface treatments and aging water on bond strength to zirconia. *Oper Dent* 2008;33:675-81.
19. Luthy H, Loeffel O, Hammerle CHF. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. *Dent Mater* 2006;22:195-200.
20. Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. *J Prosthet Dent* 2004;91:356-62.
21. Haselton DR, Diaz-Arnold AM, Dunne JT Jr. Shear bond strengths of 2 intraoral porcelain repair systems to porcelain or metal substrates. *J Prosthet Dent*. 2001;86(5):526-31.
22. Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. *Int J Prosthodont* 2005;18:60-5.
23. Della Bona A, Anusavice KJ, Mecholsky Jr JJ. Failure analysis of resin composite bonded to ceramic. *Dent Mater* 2003;19:693-9.
24. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithium disilicate-based ceramic. *Dent Mater* 2004;20:338-44.

25. Piwowarczyk A, Lauer HC, Sorensen JA. The shear bond strengths between luting cements and zirconia ceramics after two pre-treatments. *Oper Dent.* 2005;30:382-88.
26. Özcan M, Vallitu PK. Effect of surface conditioning methods on the bond strength of luting cements to ceramics. *Dent Mater* 2003;19:725-31.
27. Sun R, Suansuwan N, Kilpatrick N, Swain M. Characterization of tribochemically assisted bonding of composite resin to porcelain and metal. *J Dent* 2000;28:441-5.
28. Özcan M, Alkumru HN, Gemalmaz D. The effect of surface treatment on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic. *Int J Prosthodont* 2001;14:335-9.
29. Yoshida K, Tsuo Y, Astuta M. Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *J Biomed Mater Res* 2006;77B:28-33.

Table 1. Mean bond strength values (MPa) to each surface treatment and luting agent after 24 hours and 6 months.

Surface Treatment	ARC – 24 hours	ARC – 180 days	U100 – 24 hours	U100 – 180 days
Control	18.82 (2.22) A, b	13.89 (2.50) B, c	18.80 (1.65) A, c	11.56 (4.94) B, c
Hydrofluoric Acid	10.82 (1.82) A, c	2.10 (0.55) B, d	11.45 (1.93) A, d	0.59 (0.29) B, d
Sandblasting	18.72 (1.95) A, b	18.67 (2.78) A, b	19.90 (3.30) A, c	18.51 (3.80) A, b
Sandblasting + Alloy Primer	25.66 (1.46) A, a	22.62 (2.26) B, a	22.12 (1.89) A, b	17.37 (2.69) B, b
Silica Coating	25.28 (2.86) A, a	23.68 (2.19) A, a	25.87 (2.04) A, a	21.13 (4.43) B, a

Mean values followed by different small letters in the same column and different capital letters in the same row differ statistically among themselves for the Tukey test at the level of 5%. () – Standard Deviation

Table 2. Failure mode to each surface treatment and luting agent after 24 hours and 6 months

Surface Treatment	ARC		U100		ARC		U100	
	Failure Mode – 24 hours				Failure Mode – 180 days			
	Adhesive	Mixed	Adhesive	Mixed	Adhesive	Mixed	Adhesive	Mixed
Control	16	0	16	0	14	2	14	2
Hydrofluoric Acid	15	1	15	1	16	0	16	0
Sandblasting _(SB)	15	1	12	4	12	4	8	8
SB + Alloy Primer	10	6	13	3	10	6	10	6
Silica Coating	11	5	13	3	10	6	7	9

5 CAPÍTULO 4

5.1 Surface topography and mechanical properties of ceramic systems after different polishing methods: perfilometer and AFM analysis

Short Title - Polishing methods on surface topography of ceramics

Frederico dos Reis Goyatá*

Alexandra Andrade**

Laís Regiane da Silva-Concílio***

Leonardo Gonçalves Cunha***

Ana Christina Claro Neves***

Vinícius do Lago Pimentel****

* *Doctoral Student - Department of Prosthodontics – School of Dentistry, University of Taubate - Taubaté, São Paulo, Brazil.*

** *Master of Science Student - Department of Prosthodontics – School of Dentistry, University of Taubate - Taubaté, São Paulo, Brazil.*

*** *PhD, associate professor - Department of Prosthodontics - School of Dentistry, University of Taubate - Taubaté, São Paulo, Brazil.*

**** *Scanning Probe Microscopy Group - Brazilian Synchrotron National Laboratory - Campinas, São Paulo, Brazil.*

Clinical Relevance

The final polishing after the adjustment procedures is necessary to improve surface smoothness and minimize wear or fracture of the ceramic restorations.

Summary

Evaluate a mechanical property and the effect of different polishing techniques on the surface topography (roughness and atomic force microscope - AFM) of two ceramic systems.

A lithium disilicate reinforced-ceramic (IPS e.max - Em) and a feldspathic ceramic (IPS Classic - Cl) were used. Disc shaped samples were confectioned for each ceramic and randomly divided into four groups, according to the polishing technique (n=10): JB: silicon carbide impregnated polyamide bristle brush; DPP: diamond polisher points; JRP: abrasive impregnated silicon rubber points; DP: diamond pastes. Three additional samples of each group were evaluated by AFM. Both ceramic systems were also submitted to a Diametral Tensile Strength test (DTS - n=10), using crosshead speed of 1 mm/min. Results were analyzed by one-way ANOVA and post-hoc tests ($\alpha = 0.05$).

To both ceramics, polishing using diamond paste (DP) was associated with the smoothest surface (0.17 and 0.14 μm to CL e Em respectively). No statistical difference was observed when JB and JRP were compared, to both ceramics. DPP was associated with the highest mean value of roughness (0.52 and 0.36 μm to CL e Em, respectively), statistically higher than DP e JRP, to both ceramics. The same pattern was observed qualitatively evaluating AFM images. Em (53.5 MPa) was related to statistically higher mean values of DTS when compared to Cl (11.2 MPa).

To both ceramics, polishing using diamond paste produced the smoothest surface, and the lithium disilicate reinforced-ceramic (IPS e.max) was associated with a better mechanical property.

Keywords: ceramic, roughness, diametral tensile strength, atomic force microscope.

Introduction

Throughout the years, different noble and non-noble metallic alloys have been used in Dentistry for rehabilitation of extremely damaged teeth.¹ However, it is well known that metals are susceptible to biodegradation in the oral environment due to a combination of dissolution in saliva, wear and corrosion, mainly when different metals and alloys are used.² In addition, such materials offer an evident esthetical limitation, and patients look for restorations that provide functional requirements as well as esthetical ones.

Ceramics have been used in dentistry since 1774 for a variety of dental restorations, including metal ceramic crowns, all ceramic restorations and fixed partial dentures, because of their esthetical properties, durability and biocompatibility.³

In addition, it is possible to perform a conservative teeth preparation design with an adhesive restoration technique when all-ceramic rehabilitations are used.⁴ In this situation, different ceramic systems, such as lithium disilicate reinforced-and zirconia-based ceramics, provide satisfactory strength and esthetics compared to feldspathic ceramics.⁵⁻⁷

Ceramic restorations can provide the most natural option for teeth replacement, also associated with significant surface smoothness, and both

characteristics can be maintained for a significant period of time.⁸⁻¹⁰ In addition, metal-free ceramic systems are related to satisfactory functional properties, such as good wear resistance and toughness, extremely important properties for long-term clinical results.^{11,12}

In the laboratory, dental ceramic rehabilitations are fired and glazed, what results in a surface texture with appearance resembling that one from the natural tooth.^{13,14} However, the modification of the ceramic surface in the clinical activity is usually necessary, aiming to correct occlusal interference and inadequate contours. These adjustments produce a rough surface, increasing the probability of plaque formation and maturation, and, consequently, producing gingival inflammation and adverse soft tissue reaction. In addition, the roughness of the ceramic surface worn may increase the wear of the opposing dentition or restorative material.^{15,16}

Thus, the final polishing after the adjustment procedures is necessary to improve surface smoothness and minimize wear or fracture of the ceramic restorations.¹⁷ Some previous studies have investigated and described different polishing techniques for dental ceramics and supported the use of polishing as an alternative for glazing.¹⁸⁻²⁰ Many techniques to ceramic polishing have been studied in the literature, such as diamond polisher points, rubber and brush points, and diamond pastes in different abrasiveness granulation, but most of them to feldspathic ceramics.^{21,22} In this sense, there is a lack of studies showing what is the most effective polishing technique to reinforced ceramic systems.

Therefore, the purpose of the present study was to evaluate the surface topography by roughness and atomic force microscope analyses of two ceramic systems subjected to four polishing techniques after simulation of occlusal adjustment, and to compare both ceramics by a mechanical test. The first hypothesis tested was

that polishing using diamond polisher points promotes the smoothest surface. The second hypothesis was that lithium disilicate reinforced-ceramic presents a better mechanical property when compared to the feldspathic ceramic.

Materials and Methods

A lithium disilicate reinforced-ceramic (IPS e.max, Ivoclar-Vivadent, Schaan, Liechtenstein) and a feldspathic ceramic (IPS Classic, Ivoclar-Vivadent, Schaan, Liechtenstein) were used in the present study.

Surface Roughness

To each ceramic tested, forty disc shaped samples were confectioned following manufacturer's instructions, with dimensions of 4x2 mm.

Initially, all ceramic samples were submitted to selective worn using aluminum oxide sandpaper #600 (3M, São Paulo, SP, Brazil) in a horizontal Polishing Machine (Aropol, Arotec, São Paulo, SP, Brazil) to simulate a standardized occlusal adjustment. After that, samples were individually positioned in a SurfTest SV-600 profilometer (Mitutoyo, São Paulo, SP, Brazil) to the measurement of the initial roughness (Ra) values. Three readings in different parts of the sample were performed on each surface using a stylus tip (2µm in diameter). The extension of each reading was 1.25mm, using a cut-off of 0.8 µm.

After this first analysis, the ceramic discs were randomly divided into four groups of ten samples each, according to the following polishing techniques:

JB – Polishing using silicon carbide impregnated polyamide bristle brush of a single granulation (Jiffy Polishing Brushes, Ultradent Corporation, South Jordan, UT, USA).

DPP – Polishing using diamond polisher points (Eve System, Ernest Vetter, GmbH Germany).

JRP – Polishing using abrasive impregnated rubber points in three-abrasiveness granulation: course, medium and fine (Jiffy Polisher, Ultradent Corporation, South Jordan, UT, USA).

DP – Polishing using diamond pastes in three abrasiveness granulations: course, medium and fine (Storm Fire, São Paulo, SP, Brazil) using an auxiliary disc.

Polishing was performed by a single operator with a low speed device at 15.000 rotations per minute, using uniform pressure for a time of 30 seconds to each step. All samples were then rinsed using air/water spray for 60 s and air-dried. After that, the measurement of the final roughness was performed, using the same parameters described for the initial analyses.

Atomic Force Microscope (AFM)

Three additional samples of each ceramic system and condition tested (no polishing and polishing techniques evaluated) were confectioned to the AFM test, aiming to obtain a qualitative evaluation of the samples before and after the polishing. A Multimode Atomic Force Microscope (AFM Nanoscope IIIa, Veeco Instruments, Santa Barbara, CA, USA) was used in contact mode. The force between the AFM tip and the specimen surface was kept constant, and the vertical piezoelectric ceramic movement was recorded. Images with 512 X 512 pixels were acquired with a scan size of 10 μm X 10 μm and scan rate of 2.03 Hz. An NP-type V-shape Si_3N_4 cantilever (Veeco Instruments, Santa Barbara, CA, USA) with normal bending constant of $K=0.075$ N/m and a tip radius approximately 100nm was used. During

imaging, the set point was chosen to be 2.0 volts higher than the top-bottom laser photo detector output obtained when the tip was out of surface contact.

Diametral Tensile Strength

To the mechanical test, ten disc shaped samples of each ceramic system were confectioned following manufacturer's instructions, with dimensions of 6x3 mm. A universal testing machine (VERSAT 2000, Pantec, São Paulo, SP, Brazil) was used to determine the load at break. A crosshead speed of 1 mm/min was used for all samples. The load at which break occurred was recorded and DTS was calculated using the equation:

$$DTS \text{ (MPa)} = 2P / \pi DL$$

Where D is diameter of specimen in mm, L is the height of the specimen in mm, and P is the tension value recorded in the test in Kgf. The mean and standard deviation to each ceramic system were then calculated.

Statistical Analysis

The results of roughness were analyzed by one-way analysis of variance (ANOVA). Tukey post-hoc test was performed using 0.05 significance level for comparison of means among the polishing methods.

Diametral Tensile Strength values were submitted to Student-t Test using 0.05 significance level.

Results

Influence of the polishing technique on the surface roughness

Mean values of roughness (Ra) before and after the polishing techniques are shown in Table 1.

Before polishing, mean values of roughness of the feldspathic ceramic ranged between 0.67 and 0.76 μm , and no statistical difference was observed among the groups. After polishing, DP was associated with the lowest mean value of roughness (0.17 μm), statistically lower when compared to the other polishing methods. In opposite, DPP presented the highest mean value of roughness (0.52 μm), statistically higher compared to JRP and DP.

To the lithium disilicate reinforced-ceramic, mean values of roughness ranged between 0.29 and 0.42 μm before polishing, with no statistical difference among the groups. After polishing, the same order of the feldspathic ceramic was observed. DP presented the lowest mean value of roughness (0.14 μm), statistically lower compared to DPP and JB, and DPP presented the highest mean value of roughness (0.36 μm), statistically higher compared to JRP and DP.

In almost all groups, polishing techniques were effective to reduce statistically the mean value of roughness, to both ceramic systems.

Influence of the polishing technique on the Atomic Force Microscope images

Figures 1 and 2 show AFM micrographs of the ceramic surface to each ceramic system. Polishing using DP was associated with the smoothest ceramic surface, corroborating with the roughness values.

In opposite, DPP polishing technique was related to the roughest ceramic surface. A similar pattern of rough surface was observed to JRP to IPS Classic.

Influence of the ceramic system on the Diametral Tensile Strength

Diametral tensile strength is shown in Table 2. Lithium disilicate reinforced ceramic (IPS e.max) presented a mean value of 53.5 MPa, statistically higher than the feldspathic ceramic (IPS Classic - 11.2 MPa).

Discussion

Ceramic restorations have been used significantly in Dentistry in last years.¹⁻² However, some low physical properties of this material, such as flexural and tensile strength, limited its application associated with metallic alloys.⁴ The reinforcement of some ceramic systems and the enhancement of the adhesive dentistry allowed the use of ceramics with no metal framing, improving mechanical strength and esthetics of these rehabilitations, for both anterior and posterior teeth.⁵⁻⁷

Independent of the ceramic system, occlusal adjustment are often required after cementation and much has been discussed regarding the negative impact of rough ceramic surfaces, such as bacterial plaque accumulation and wearing of opposite teeth.⁸ Therefore, one of the prime requisites for a satisfactory ceramic restoration is a highly smooth surface, which contributes to the patient's comfort, optimum esthetics, oral hygiene and low plaque retention.^{9,10}

In addition, an effective polishing prevents discoloration of rough areas and leads to a more natural appearance of dental rehabilitations.^{13,14} Besides that, rough or irregular ceramic surfaces after intraoral adjustment may concentrate stresses and initiate cracking propagation resulting in premature fracture of the restoration.¹⁵⁻¹⁷

However, there is a lack of information in the literature about satisfactory polishing techniques to the new ceramic systems available, such as lithium disilicate ceramics and heat pressed ceramics.^{18,19} Most of the previous studies are related to polishing methods performed in feldspathic ceramics associated with metallic alloy framing.^{20,21}

Aiming to compare polishing methods of ceramic systems, atomic force microscope is an alternative technique, with high resolution in nanometer scales.²² Although AFM verifies a small area, it is representative, because it goes point by point and covers the whole. In addition, AFM holds significant advantages, such as minimal sample preparation, high resolution and visualization of a 3-dimensional image of the surface, being considered a significant qualitative parameter in the study of biomaterials.²³

Based on the results of the present study, it may be stated that all polishing techniques were able to reduce surface roughness when compared to the initial situation after simulation of occlusal adjustment.⁸ The only exception was observed when diamond polisher points were used to polish e.max ceramic system, in which no difference in the mean roughness values were found before and after polishing.^{13,14} The mean reduction of roughness associated with the polishing was 41%, independent of the ceramic system. IPS Classic presented a higher mean percentage of reduction, 46%, when compared to the IPS e.max system (30.5%).

Individually, results have shown that the ceramic materials tested can be polished to different degrees of smoothness when submitted to different polishing systems.⁸ Indeed, polishing using diamond pastes was associated with the highest reduction of surface roughness, 77% to IPS Classic and 55% to IPS e.max. The same pattern of roughness reduction could be seen at the AFM micrographs,

presented in Figures 1 and 2. A smooth surface was reached after polishing using diamond pastes, to both ceramics, especially to IPS e.max. Such results could be related to a systematic reduction in diamond particle size of the paste, from course to fine particles. In addition, using diamond pastes, finishing is not totally dry, what was not observed to the other polishing techniques tested. The possibility of using a paste instead of dry instruments could be related to an increased probability of smoother surfaces.^{15,16}

In opposite, when diamond polisher points (DPP) were used, the roughest surface was observed. The reduction in roughness related to this polishing technique was 32% to IPS Classic and only 14% to IPS e.max. Indeed, as can be seen in AFMs micrographs, DPP created noticeable irregularities in the ceramic surface, caused by the abrasive potential of the diamond impregnated polisher points in different granulations.²¹⁻²³ Furthermore, an increase of temperature was observed on the ceramic surface when DPP was employed. This temperature rise was easily perceived by touch and it was higher than that recorded for the other polishing systems. This is due to thermal difference generated on the ceramic, which could cause microfissures on the ceramic surface, thus, increasing roughness values and the quality of the AFM obtained.^{3,15,26}

Jiffy brush (JB) and Jiffy Rubber points (JRP) were associated with intermediate results. JB promoted a roughness reduction of 33% and 30% to IPS Classic and e.max, respectively. JRP was related to improved results to IPS Classic, causing a roughness reduction of 43% to IPS Classic and 26% to IPS e.max.

Concerning mechanical properties, the continuous improvement of the ceramic systems still have not eliminated some limitations for the use of these esthetic restorative materials, mainly in posterior teeth.^{11,12,17,27} This rationale is

supported by the results of some studies, which reported that better mechanical properties improve the resistance to wear and to fracture, minimizing clinical problems involving loss of anatomic form and decreased marginal adaptation of ceramic rehabilitations.^{15,16} In this sense, diametral tensile strength (DTS) is an effective method to test and compare biomaterials in relation to their mechanical properties.²⁴⁻²⁶ From the results of the present study, it was observed a higher mean of DTS to the lithium disilicate glass ceramic (IPS e.max - 53.5 MPa), a mean value almost 480% higher than the one reached by the feldspathic ceramic (IPS Classic - 11.2 MPa). The superiority of the reinforced ceramic system confirmed the possibility of using IPS e.max to single crowns in posterior teeth rehabilitations, in agreement to manufacturer's indications.

Therefore, the first hypothesis that polishing using diamond polisher points – DPP would promote the smoothest surface was denied by the results. Actually, this technique was associated with the roughest surface after polishing when compared to the others. However, the second hypothesis was accepted, because lithium disilicate glass ceramic showed improved performance to the mechanical property evaluated, when compared to the feldspathic ceramic.

Conclusions

Considering the limitations of the present study and the reached results, the following conclusions were drawn:

1. Polishing using diamond paste created a smooth surface to the two ceramic systems evaluated.
2. Lithium disilicate glass ceramic (IPS e.max) was associated with an improved mechanical property when compared to the feldspathic ceramic.

References

1. Anusavice KJ (2003) *Phillips Science of Dental Materials* 11^a Ed, Elsevier, Florida.
2. Walton TR (1999) A 10-year longitudinal study of fixed prosthodontics: clinical characteristics and outcome of single unit metal-ceramic crowns *International Journal of Prosthodontics* **12(6)** 519-526.
3. Jason AG (2007) Recent Advances in Materials for All-Ceramic Restorations *Dental Clinics of North America* **51(3)** 713-727.
4. Roberson TM, Heymann HO & Swift EJ (2000) *Sturdevant's Art and Science of Operative Dentistry* 4^a Ed, Mosby, St. Louis.
5. Fradeani M & Redemagni M (2002) An 11-year clinical evaluation of leucite reinforced glass-ceramic crowns: a retrospective study *Quintessence International* **33(7)** 503-510.
6. Manicone PF, Rossi Iommetti P & Raffaelli L (2007) An overview of zirconia ceramics: basic properties and clinical applications *Journal of Dentistry* **35(11)** 819-826.
7. O'Brien WJ (2008) *Physical Properties in Dental materials and Their Selection* 4^a Ed, Quintessence Publishing Co, Illinois.
8. Al-Wahadani A & Martin DM (1998) Glazing and finishing dental porcelain: a literature review *Journal Canadian Dental Association* **64(8)** 580-583.
9. Kawai K, Urano M & Ebisu S (2000) Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans *The Journal of Prosthetic Dentistry* **83(6)** 664-667.
10. Auschill TM, Arweiler NB, Brex M, Reich E, Sculean A & Netuschil L (2002) The effect of dental restorative materials on dental biofilm *European Journal of Oral Sciences* **110(1)** 48-53.

11. Molin MK & Karlsson SL (2000) A randomized 5-year clinical evaluation of 3 ceramic inlay systems *The International Journal of Prosthodontics* **13(3)** 194-200.
12. Kramer N & Frankenberger R (2005) Clinical performance of bonded leucite-reinforced glass ceramic inlays and onlays after eight years *Dental Materials* **21(3)** 262-271.
13. Klausner LH, Cartwright CB & Charbeneau GT (1982) Polishing versus autoglazed porcelain surfaces *The Journal of Prosthetic Dentistry* **47(2)** 157-162.
14. Raimondo RL Jr, Richardson JT & Weidner B (1990) Polished versus autoglazed dental porcelain *The Journal of Prosthetic Dentistry* **64(5)** 553-557.
15. Fisher H, Schafer M & Marx R (2003) Effect of surface roughness on flexural strength of veneer ceramics *Journal of Dental Research* **82(12)** 972-975.
16. Lohbauer U, Muller FA & Petschelt A (2008) Influence of surface roughness on mechanical strength of resin composite versus glass ceramic materials *Dental Materials* **24(2)** 250-256.
17. Stappert CF, Guess PC, Chitmongkolsuk S, Gerds T & Strub JR (2006) Partial coverage restoration systems on molars-comparison of failure load after exposure to a mastication simulator *Journal of Oral Rehabilitation* **33(9)** 698-705.
18. Jagger DC & Harrison A (1994) A vivo investigation into the wear effects of unglazed, glazed, and polished porcelain on human enamel *The Journal of Prosthetic Dentistry* **72(3)** 320-323.
19. Wright MD, Masri R, Driscoll CF, Romberg E, Thompson GA & Runyan DA (2004) Comparison of three systems for the polishing of ultra-low fusing dental porcelain *The Journal of Prosthetic Dentistry* **92(5)** 486-490.

20. Sarac D, Sarac YS, Yusbasioglu E & Bal S (2006) The effect of porcelain polishing systems on the color and surface texture of feldspathic ceramic. *The Journal of Prosthetic Dentistry* **96(2)** 122-128.
21. Sarac YS, Turk SE, Sarac D & Turk T (2007) Surface conditioning methods and polishing techniques effect on surface roughness of feldspar ceramic *The Angle Orthodontist* **77(4)** 723-728.
22. Tholt de Vasconcellos B, Miranda-Júnior WG, Prioli R, Thompson J & Oda M (2006) Surface roughness in ceramics with different finishing techniques using atomic force microscope and profilometer *Operative Dentistry* **31(4)** 442-449.
23. Hegedus C, Bistey T, Flora-Nagy E, Keszthelyi G & Jenei A (1999) An atomic force microscope study on the effect of bleaching agents on enamel surface *Journal of Dentistry* **27(7)** 509-515.
24. Aguiar FHB, Braceiro ATB, Ambrosano GMB & Lovadino JR (2007) Hardness and diametral tensile strength of a hybrid composite resin polymerized with different modes and immersed in ethanol or distilled water media *Dental Materials* **21(12)** 1098-1103.
25. Della Bona A, Benetti P, Borba M & Ceccheti D (2008) Flexural and diametral strength of composite resins *Brazilian Oral Research* **22(1)** 84-89.
26. Guazzato M, Albarkry M, Quach L & Swain MV (2005) Influence of surface and heat treatments on the flexural strength of a glass-infiltrated alumina-zirconia-reinforced dental ceramic *Dental Materials* **21(5)** 454-463.
27. Denry I & Kelly JR (2008) State of the art of Zirconia for dental applications. *Dental Materials* **24(3)** 299-307.

Table 1 – Mean values of roughness before and after different polishing methods by each ceramic system evaluated.

Polishing Systems	IPS Classic		IPS E.max	
	Before	After	Before	After
Jiffy brush (JB)	0.73(0.11) A, a	0.49(0.12) B, ab	0.40 (0.15) A, a	0.28 (0.09) B, ab
Diamond polisher points (DPP)	0.76(0.09) A, a	0.52 (0.10) B, a	0.42 (0.18) A, a	0.36 (0.10) A, a
Jiff rubber points (JRP)	0.67(0.15) A, a	0.38 (0.14) B, b	0.29 (0.09) A, a	0.21 (0.08) B, bc
Diamond paste (DP)	0.73(0.07) A, a	0.17 (0.03) B, c	0.31 (0.05) A, a	0.14 (0.04) B, c

Mean values followed by different small letters (a–c) in the same column and different capital letters (A-B) comparing each ceramic in row differ statistically among themselves for the Tukey test at the level of 5%. Values present within the parentheses denote standard deviation.

Table 2 – Mean values of diametral tensile strength (MPa) to the ceramic systems evaluated

Ceramic	Diametral Tensile Strength (MPa)
IPS Classic	11.2 (2.8) b
IPS e.max	53.5 (5.0) a

Mean values followed by different small letters differ statistically for the Student t-Test at the level of 5%. () Standard Deviation.

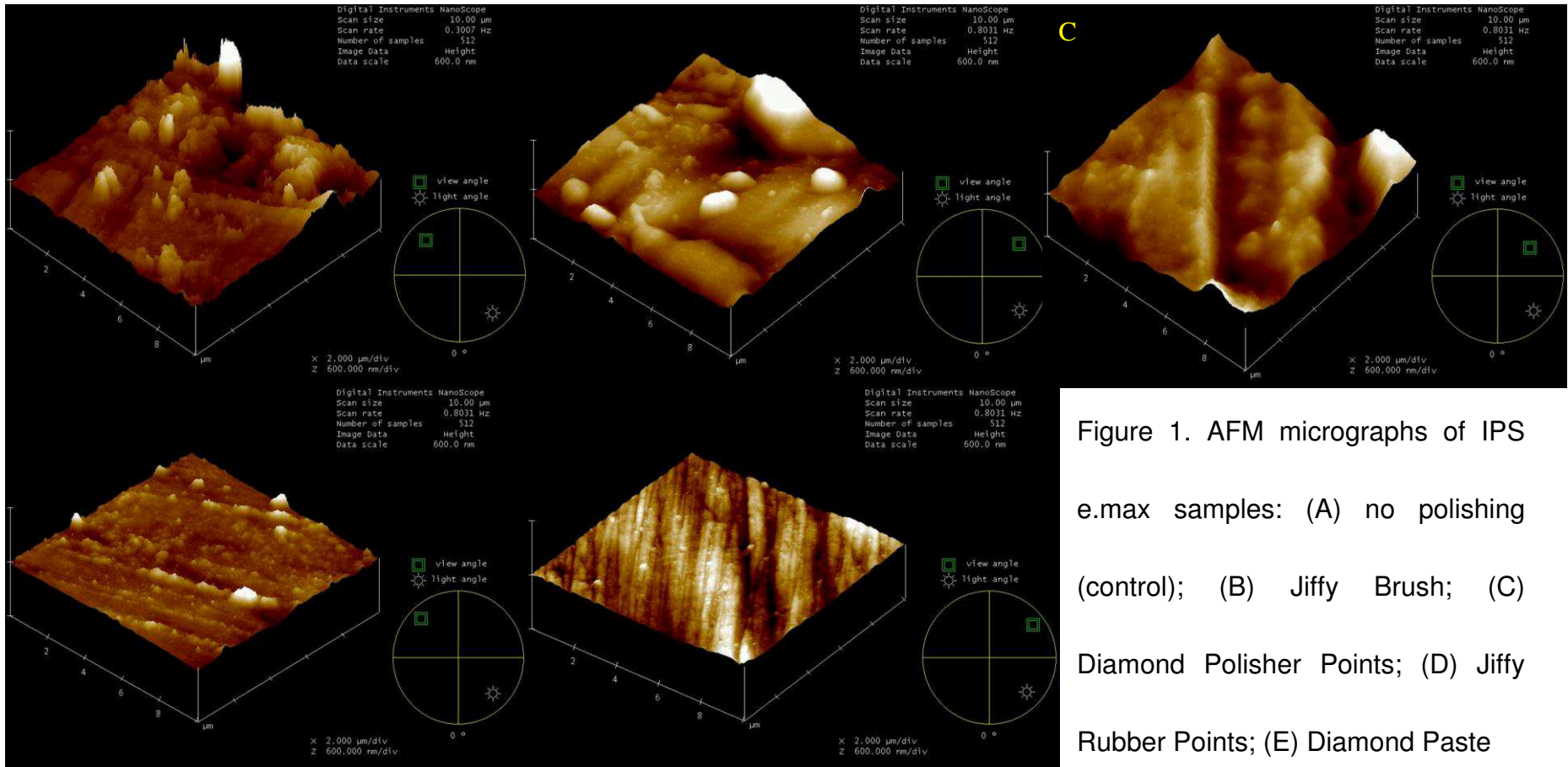
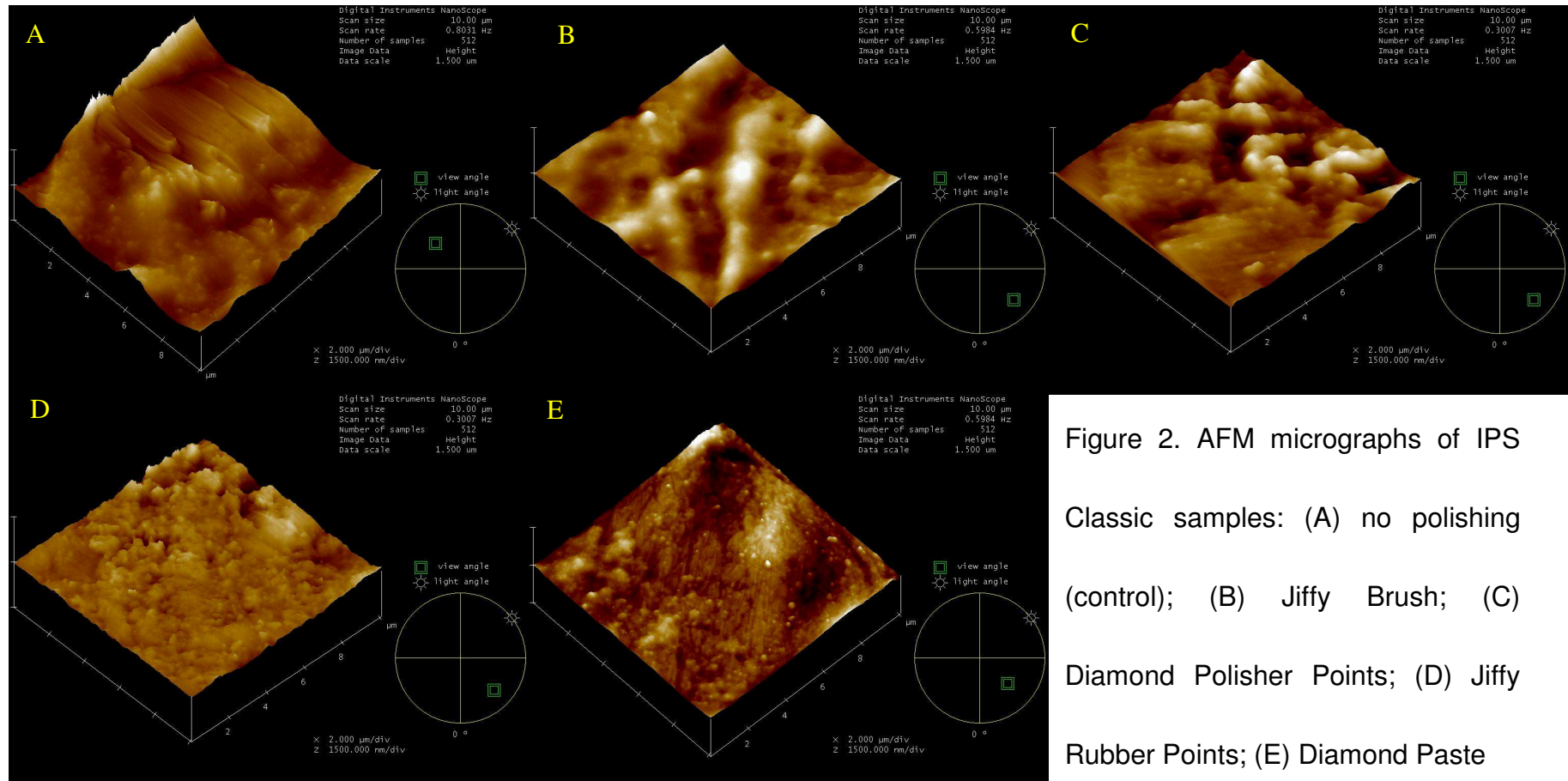


Figure 1. AFM micrographs of IPS e.max samples: (A) no polishing (control); (B) Jiffy Brush; (C) Diamond Polisher Points; (D) Jiffy Rubber Points; (E) Diamond Paste



5 CONSIDERAÇÕES FINAIS

Realizar um protocolo de cimentação criterioso para as restaurações e próteses em cerâmica é fundamental para se obter sucesso e longevidade nos procedimentos clínicos. Desta forma, um tratamento de superfície das cerâmicas bem executado, previamente à cimentação adesiva, irá proporcionar resistência adesiva mais eficiente com o substrato dental.

Da mesma maneira, é importante identificar o sistema cerâmico que será utilizado e planejar qual o melhor método de tratamento de superfície. Conforme descrito no capítulo 1 deste trabalho, diferentes métodos são aplicados, sendo que existe uma indicação específica para cada sistema cerâmico em função de suas propriedades físico-químicas.

Nas cerâmicas feldspáticas assim como nas cerâmicas a base de dissilicato de lítio, o condicionamento com ácido fluorídrico em concentrações de 9 a 10% seguido da aplicação do silano é bastante efetivo e tem proporcionado valores elevados de resistência adesiva em diversos relatos na literatura. Em contrapartida, para as cerâmicas reforçadas por óxido de zircônia, o jateamento com partículas de óxido de alumínio modificadas por sílica, denominado de silicatização, tem demonstrado os melhores resultados de resistência adesiva, conforme demonstrado em diversos trabalhos na literatura e apresentado nos capítulos 2 e 3 deste trabalho.

Da mesma forma, é importante considerar e eleger um método de tratamento de superfície para as cerâmicas reforçadas por óxido de zircônia de modo que se possam obter resultados clínicos de resistência adesiva com certa longevidade, o que pode ser observado nos resultados obtidos no capítulo 3, em que a

armazenagem em água por 180 dias alterou significativamente alguns métodos de tratamento realizados.

Após a cimentação, os procedimentos de ajuste oclusal devem ser realizados para proporcionar uma oclusão funcional ao paciente.

Desta forma, a superfície da cerâmica se tornará rugosa o que pode levar a microtrincas na restauração e comprometer a longevidade do tratamento. Da mesma maneira, poderá influenciar na saúde do periodonto, favorecendo o acúmulo de microrganismos próximo à restauração.

Sendo assim, realizar um acabamento e um polimento irá contribuir para a longevidade do tratamento impedindo possíveis fraturas da cerâmica ao longo do tempo. Também proporcionará ao paciente uma lisura de superfície maior e possibilidade de uma higienização mais eficiente. Diferentes métodos de acabamento e polimento para as cerâmicas são descritos na literatura. O capítulo 4 deste trabalho propõe uma correlação entre os métodos de acabamento para uma cerâmica feldspática e uma cerâmica a base de dissilicato de lítio e a obtenção de uma lisura de superfície. É importante observar que todos os métodos testados proporcionaram uma lisura superficial, conforme demonstrou os valores de rugosidade e as imagens em MFA. Da mesma forma, os métodos testados não alteraram as propriedades mecânicas das cerâmicas, demonstrado pelo teste de tração diametral.

O cirurgião-dentista deve instituir uma metodologia de trabalho criteriosa com as cerâmicas odontológicas embasado em trabalhos científicos a fim de que as restaurações protéticas possam ter longevidade clínica e atender aos anseios dos pacientes.

REFERÊNCIAS

1. Anusavice KJ. Phillips' science dental materials. 11a ed. Florida: Elsevier; 2003.
2. Fradeani M, Redemagni M. An 11-year clinical evaluation of leucite reinforced glass-ceramic crowns: a retrospective study. *Quintessence Int* 2002; 33(7):503-10.
3. Mehulic M, Mehulic K, Kos P, Komar D, Katunaric M. Expression of contact allergy in undergoing prosthodontic therapy patients with oral diseases. *Minerva Stomatol* 2005; 54(5):303-9.
4. Kramer N, Frankenberger R. Clinical performance of bonded leucite-reinforced glass ceramic inlays and onlays after eight years. *Dent Mater* 2005; 21(3):262-71.
5. Luthy H, Filser F, Loeffel O, Schumacher M, Gaucker LJ, Hammerle CHF. Strength and reliability of four-unit all-ceramic posterior bridges. *Dent Mater* 2005; 21(10):930-7.
6. Mc Laren EA, Giordano RA. Zirconia-based ceramics: Material properties, esthetics, and layering techniques of new veneering porcelain, VM9. *QTD* 2005; 99-111.
7. Atsu SS, Kilicarlsan MA, Kucukesmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. *J Prosthet Dent* 2006; 95(6):430-36.
8. Griggs JA. Recent advances in materials for all- ceramic restorations. *Dent Clin North Am* 2007; 51(3):713-27.
9. Manicone PF, Rossi IP, Raffaelli L. An overview of zirconia ceramics: Basic properties and clinical applications. *J Dent* 2007; 35(11):819-26.
10. Della Bona A, Melcholsky JJ Jr, Barret AA, Griggs JA. Characterization of glass-infiltrated alumina-based ceramics. *Dent Mater* 2008; 24(11):1568-74.

11. Hilgert LA, Schweiger J, Beuer F, Andrada MAC, Araújo E, Edelhoff D. Odontologia restauradora com sistemas CAD/CAM: o estado atual da arte. Parte 1: princípios de utilização. *Clínica-Int J Braz Dent* 2009a; 5(3):294-303.
12. Hilgert LA, Schweiger J, Beuer F, Andrada MAC, Araújo E, Edelhoff D. Odontologia restauradora com sistemas CAD/CAM: o estado atual da arte. Parte 2: possibilidades restauradoras e sistemas CAD/CAM. *Clínica-Int J Braz Dent* 2009b; 5(4):424-35.
13. Hilgert LA, Schweiger J, Beuer F, Eichberger M, Andrada MAC, Araújo E, et al. Odontologia restauradora com sistemas CAD/CAM: O estado atual da arte Parte 3: materiais restauradores para sistemas CAD/CAM. *Clínica-Int J Braz Dent* 2010; 6(1):86-96.
14. Valandro LF, Della Bona A, Bottino MA, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. *J Prosthet Dent* 2005; 93(3):253-9.
15. Kiyani VH, Saraceni CH, Silveira BL, Aranha AC, Eduardo CP. The influence of internal surface treatments on tensile bond Strength for two ceramic systems. *Oper Dent* 2007; 32(5):457-65.
16. Borges GA, Sophr AM, Goes MF, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 2003; 89(5):479-88.
17. Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. *Int J Prosthodont* 2005; 18(1):60-5.
18. Spohr AM, Sobrinho LC, Consani S, Sinhoreti MAC, Knowles JC. Influence of surface conditions and silane agent on the bond of resin to IPS Empress 2 ceramic. *Int J Prosthodont* 2003; 16(3):277-82.
19. Guler AU, Yilmaz F, Ural Ç, Guler E. Evaluation of 24-hour shear bond strength of resin composite to porcelain according to surface treatment. *Int J Prosthodont* 2005; 18(2):156-60.

20. Della Bona A, Donassollo TA, Demarco FF, Barrett AA, Mecholsky JJ Jr. Characterization and surface treatment effects on topography of a glass-infiltrated alumina/zirconia-reinforced ceramic. *Dent Mater* 2007a; 23(6):769-75.
21. Spohr AM, Borges GA, Júnior LH, Mota EG, Oshima HM. Surface modification of In-Ceram Zirconia ceramic by Nd: YAG laser, Rocatec System, or aluminium oxide sandblasting and its bond strength to resin cement. *Photomed Laser Surg* 2008; 26(3):203-8.
22. Chaiyabutr Y, Mc Gowan S, Phillips KM, Kois JC, Giordano RA. The effect of hydrofluoric acid surface treatment and bond strength of a zirconia veneering ceramic. *J Prosthet Dent* 2008; 100(3):194-202.
23. Guazzato M, Albarkry M, Quach L, Swain MV. Influence of surface and heat treatments on the flexural strength of a glass-infiltrated alumina-zirconia-reinforced dental ceramic. *Dent Mater* 2006; 21(5):454-63.
24. Akgungor G, Sen D, Aydin M. Influence of different surface treatments on the short-term bond strength and durability between a zirconia post and composite resin core material. *J Prosthet Dent* 2008; 99(5):388-99.
25. Peutzfeldt A, Asmussen E. Silicoating. Evaluation of a new method of bonding composite resin to metal. *Scand J Dent Res* 1988; 96(2):171-6.
26. Özcan M. The use of chairside silica coating for different dental applications. *J Prosthet Dent* 2002; 87(5):469-72.
27. Özcan M, Vallitu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramic. *Dent Mater* 2003; 19(8):725-31.
28. Della Bona A, Borba M, Beneti P, Ceccheti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. *Braz Oral Res* 2007b; 21(1):10-15.
29. Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. *Quintessence Int* 2007; 38(9):745-53.
30. Re D, Augusti D, Sailer I, Spreafico D, Ceruti A. The effect of surface treatment on the adhesion of resin cements to Y-TZP. *Eur J Esthet Dent* 2008; 3(2):186-96.

31. May LG, Passos SP, Capelli DB, Ozcan M, Bottino MA, Valandro LF. Effect of silica coating combined to an MDP-based primer on the resin bond to Y-TZP ceramic. *J Biomed Mater Res B Appl Biomater*. In press 2010.
32. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003; 89(3):268-74.
33. Piwowarczyk A, Bender R, Ottl P, Lauer HC. Long-term bond between dual-polymerizing cementing agents and human hard dental tissue. *Dent Mater* 2007; 23(2):211-17.
34. Hilgert LA, Monteiro Júnior S, Vieira LCC, Gerney W, Edelhoff D. A escolha do agente cimentante para restaurações cerâmicas. *Clínica-Int J Braz Dent* 2009c; 5(2):194-205.
35. Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. *J Adhes Dent* 2008; 10(4):251-8.
36. Behr M, Rosentritt M, Regnet T, Lang R, Handel G. Marginal adaptation in dentin of self-adhesive universal resin cement compared with well-tried systems. *Dent Mater* 2004; 20(2):191-7.
37. Fabianelli A, Goracci C, Bertelli E, Monicelli F, Grandini S, Ferrari M. In vitro evaluation of wall-to-wall adaptation of self-adhesive resin cement used for luting gold and ceramic inlays. *J Adhes Dent* 2005; 7(1):33-40.
38. Ibarra G, Johnson GH, Geurtsen W, Vargas MA. Microleakage of porcelain veneer restorations bonded to enamel and dentin with a new self-adhesive resin-based dental cement. *Dent Mater* 2007; 23(2):218-25.
39. Okeson JP. Tratamento das disfunções temporomandibulares e oclusão. Rio de Janeiro: Elsevier; 2010.
40. Fisher H, Schafer M, Marx R. Effect of surface roughness on flexural strength of veneer ceramics. *J Dent Res* 2003; 82(12):972-5.

41. Lohbauer U, Muller FA, Petschelt A. Influence of surface roughness on mechanical strength of resin composite versus glass ceramic materials. *Dent Mater* 2008; 24(2):250-6.
42. Kawai K, Urano M, Ebisu S. Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans. *J Prost Dent* 2000; 83(6):664-7.
43. Auschill TM, Arweiler NB, Brex M, Reich E, Sculean A, Netuschil L. The effect of dental restorative materials on dental biofilm. *Eur J Oral Sci* 2002; 110(1):48-53.
44. Raimondo RL Jr, Richardson JT, Weidner B. Polished versus autoglazed dental porcelain. *J Prosthet Dent* 1990; 64(5):553-7.
45. Al-Wahadani A, Martin DM. Glazing and finishing dental porcelain: a literature review. *J Canad Den Assoc* 1998; 64(8):580-3.
46. Sarikaya I, Guler AU. Effect of different polishing techniques on the surface roughness of dental porcelains. *J Appl Oral Sci* 2010; 18(1):10-6.
47. Yilmaz K, Ozkan P. Profilometer evaluation of the effect of various polishing methods on the surface roughness in dental ceramics of different structures subjected to repeated firings. *Quintessence Int* 2010; 41(7):125-31.
48. Wright MD, Masri R, Driscoll CF, Romberg E, Thompson GA, Runyan DA. Comparison of three systems for the polishing of ultra-low fusing dental porcelain. *J Prosthet Dent* 2004; 92(5):486-90.
49. Sarac D, Sarac YS, Yusbasioglu E, Bal S. The effect of porcelain polishing systems on the color and surface texture of feldspathic ceramic. *J Prosthet Dent* 2006; 96(2):122-8.
50. Sarac YS, Turk SE, Sarac D, Turk T. Surface conditioning methods and polishing techniques effect on surface roughness of feldspar ceramic. *Angle Orthodontist* 2007; 77(4):723-8.

Autorizo a reprodução e divulgação total ou parcial desta obra, por qualquer meio convencional ou eletrônico, para fins de estudo e pesquisa, desde que citada a fonte.
Frederico dos Reis Goyatá
Taubaté, Agosto de 2010