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Estudo de variáveis que atuam no processo de cimentação de restaurações cerâmicas: atenuação da luz, modos de ativação e tratamento superficial

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"Aqueles que estão apaixonados pela prática sem a ciência são iguais ao piloto que navega sem lente ou bússola e nunca tem a certeza para onde vai. A prática deve estar sempre baseada em um perfeito conhecimento da teoria".

Leonardo da Vinci

RESUMO

A durabilidade de uma restauração cerâmica depende de fatores como grau de conversão do agente de cimentação e interação deste agente de cimentação com a superfície interna da cerâmica. Estes estudos *in vitro* avaliaram: (1) a influência de diferentes cerâmicas e o efeito de diferentes modos de ativação na microdureza Knoop (KHN) de um agente de cimentação resinoso dual, imediatamente e 24 horas após a polimerização e (2) a resistência de união de um novo tratamento de superfície à base de glaze na união entre a cerâmica à base de zirconia e um agente de cimentação resinoso dual, em associação com os tratamentos: jateamento com partículas de óxido de alumínio com 50 e 110µm, condicionamento com ácido fluorídrico a 10% e silanização. Para avaliação da atenuação da luz e dos modos de ativação, 10 discos do cimento resinoso Panavia F 2.0 foram confeccionados para cada grupo e ativados por fotoativação direta, ativação química ou através de discos de diferentes cerâmicas com 1.2mm de espessura. Os valores de KHN foram obtidos imediatamente e após 24 horas de armazenagem a 37 ^{o}C. Para a análise da resistência de união à cerâmica à base de zircônia, 80 discos de cerâmica à base de zirconia tetragonal estabilizada por ítrio foram confeccionados e receberam 8 tratamentos de superfície: Grupo I- jateamento com partículas de óxido de alumínio com 110µm, Grupo II- jateamento com partículas de óxido de alumínio com 110µm e silanização, Grupo III- jateamento com partículas de óxido de alumínio com 50µm, Grupo IV- jateamento com partículas de óxido de alumínio com 50µm e silanização, Grupo V- glaze e condicionamento com ácido fluorídrico 10%, Grupo VI- glaze, condicionamento com ácido fluorídrico 10% e silanização, Grupo VII- glaze e jateamento com partículas de óxido de alumínio com 50µm e Grupo VIII- glaze, jateamento com partículas de óxido de alumínio com 50µm e silanização. Após os tratamentos, o cimento resinoso dual Enforce foi inserido em microtúbulos Tygon em contato com a superfície das cerâmicas e fotoativado por 40s. A resistência de união foi obtida por ensaio de microcisalhamento. Os dados foram submetidos à Análise de Variância e ao teste de Tukey (5%) e mostraram que o agente de cimentação fotoativado através das cerâmicas à base de vidro e di-silicato apresentaram

KHN superiores aos fotoativados através das cerâmicas à base de alumina e zircônia, imediatamente e após 24 horas. A fotoativação direta levou a valores de KHN superiores aos grupos que foram ativados através das cerâmicas e ativação química, para o tempo imediato e 24 horas. A resistência de união à cerâmica zircônia foi afetada pelos diferentes tratamentos de superfície e a associação entre o glaze e o ácido fluorídrico apresentou os melhores resultados, independentemente do processo de silanização. Dessa forma, pode-se concluir que a fotoativação através das cerâmicas promove atenuação da luz e KHN superior para o tempo de armazenagem de 24 horas, exceto para o modo de ativação direto. O tratamento com glaze promove significante aumento nos valores de resistência de união.

Palavras-chave: Cerâmica, Fotopolimerização, Cisalhamento, Cimentos Dentários.

ABSTRACT

The durability of ceramic restoration depends of factors like the conversion degree of a luting agent and the interaction of this luting agent with the ceramic internal surface. These *in vitro* studies evaluate: (1) the influence of different ceramics and the effect of different activation modes on Knoop Hardness Number (KHN) of a dual agent luting, immediately and 24 hours after polymerization and (2) the bond strength of a novel surface treatment that use a glaze for promotes a bond between zirconia-based ceramic and a dual resin luting agent, in association with the treatments: 50 e 110μm air particle abrasion, 10% hydrofluoric acid etching and silanization. For the light attenuation and activated modes evaluation, 10 discs of Panavia F 2.0 resin cement were fabricated for each group and activated by directly photoactivation, chemical and activation through different ceramic discs of 1.2 mm thickness. KHN was obtained using microhardness immediately and after storage at 37$^{\text{o}}$C for 24 hours. For the bond strength analysis to zirconia-based ceramic, 80 ceramic discs based on Yttrium-stabilized tetragonal Zirconia were fabricated and received 8 different surface treatments: Group I- 110μm aluminum oxide particle abrasion, Group II- 110μm aluminum oxide particle abrasion and silanization, Group III- 50μm aluminum oxide particle abrasion, Group IV- 50μm aluminum oxide particle abrasion and silanization, Group V- glaze and hydrofluoric acid conditioning, Group VI- glaze, hydrofluoric acid conditioning and silanization, Group VII- glaze and 50μm aluminum oxide particle abrasion, and Group VIII- glaze, 50μm aluminum oxide particle abrasion and silanization. After the treatments, Enforce resin cement was placed into micro bore Tygon tubing in contact with the ceramic surfaces and photoactivated for 40s. The bond strength was obtained by the microshear bond test. The data were submitted to Analysis of Variance and Tukey's test (5%) and showed that the glass and di-silicate based ceramics presented higher KHN than alumina and zirconia based ceramics, immediately and after 24 hours. The direct photoactivation showed higher KHN than the activated through the ceramics groups and chemical activation for immediate and 24 hours. The bond strength to zirconia was affected by different treatments and the association between glaze

and fluoric acid showed the best results, independent of the silanization process. Of this form, it can be concluded that the photoactivation through the ceramics promotes light attenuation and an improvement of KHN was found after 24 hours storage except for directly activated mode. The treatment with glaze promoted a significant increase of bond strength values.

Key-Words: Ceramic, Photoactivation, Shear strength, Dental cements.

SUMÁRIO

INTRODUÇÃO

A busca por tratamentos estéticos abrange todos os campos da Odontologia que buscam reabilitar, tratar e promover saúde bucal ao longo do tempo. As restaurações cerâmicas apresentam papel inquestionável dentro deste cenário, apresentando excelentes propriedades estéticas que simulam a aparência da dentição natural. O conhecimento do comportamento frente à diversidade de composição dos sistemas cerâmicos, atenuação da luz durante o processo de fotoativação, tratamentos de superfície, interação com os agentes de cimentação, bem como seu comportamento frente aos estudos clínicos e laboratoriais, são fatores dominantes dentro das investigações científicas na atualidade.

A translucidez, fluorescência, estabilidade química, biocompatibilidade, alta resistência à compressão e coeficiente de expansão térmica similar à estrutura dental são propriedades que contribuem para o grande uso das cerâmicas nas reabilitações dentais (Aboushelib *et al.*, 2006; Pazin *et al.*, 2008). A interação entre o agente de cimentação e a superfície cerâmica tratada é considerada vital para a longevidade das restaurações e deve se apresentar forte o suficiente para resistir às cargas mastigatórias (Blatz *et al.*, 2004; Peumans *et al.*, 2004; Aboushelib *et al.*, 2007). As vantagens da utilização dos agentes de cimentação resinosos como selamento marginal, boa retenção inicial e aumento da resistência à fratura da cerâmica, colocam estes materiais como primeira escolha no processo de cimentação (Burke *et al.*, 2002; Guazzato *et al.*, 2004a).

O sucesso das restaurações cerâmicas cimentadas com agentes resinosos depende diretamente do grau de conversão dos monômeros resinosos, influenciando nas propriedades como dureza, resistência ao desgaste, absorção de água, presença de monômeros residuais e biocompatibilidade (Rasetto *et al.*, 2001). Os agentes de cimentação resinosos apresentam como vantagem a possibilidade da polimerização química e da fotoativação (Peutzfeldt, 1995; El-Mowafy & Rubo, 2000; Caughman & Rueggeberg,

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2002; Fonseca *et al.*, 2004), embora esta necessite de adequada quantidade de luz para iniciar o processo de polimerização (Tarle *et al.*, 2006). A intensidade de luz e a distância da fonte de luz durante a polimerização são outros fatores importantes que devem ser levados em consideração durante o processo de cimentação (Tashiro *et al.*, 2004; Koch *et al.*, 2007).

A composição, espessura, opacidade e cor das cerâmicas podem atenuar a passagem da luz usada para a ativação da reação de polimerização do agente de cimentação resinoso (El-Mowafy & Rubo, 2000). As cerâmicas disponíveis apresentam diferenças quanto à composição e ao conteúdo cristalino, comportando-se de formas diferentes frente ao processo de fotoativação e levando atenuação da luz característica para cada material. As cerâmicas à base de vidro e di-silicato apresentam atenuação de luz diferente das cerâmicas reforçadas com alto conteúdo cristalino à base de óxido de alumínio e zircônio (Tango *et al.*, 2007). Entretanto, há limitadas informações visando correlacionar o efeito da composição, opacidade e espessura das diferentes cerâmicas na atenuação de luz.

O tratamento de superfície das cerâmicas é outro importante fator que deve ser considerado dentro do processo de cimentação. A durabilidade da resistência de união dos agentes de cimentação resinosos é dependente da interação com a superfície da cerâmica e pode ser obtida através de microretenção ou interação química. Tratamentos convencionais como jateamento com partículas de óxido de alumínio, condicionamento com ácido fluorídrico e silanização não são capazes de promover forte e durável resistência de união entre o cimento resinoso e as cerâmicas com alto teor mineral à base de óxido de alumínio e zircônio (Guazzato *et al.*, 2004b; Spohr *et al.*, 2008), embora sejam tratamentos eficientes para as cerâmicas à base de vidro e di-silicato (Blatz *et al.*, 2003).

As cerâmicas à base de zircônia apresentam alta resistência à flexão e fratura, estabilidade química e biocompatibilidade, estas características as colocam em destaque em relação aos diversos sistemas cerâmicos (Aboushelib *et al.*, 2006). Estas cerâmicas apresentam um mecanismo de aumento da tenacidade por transformação induzida por

tensão, podendo resistir ativamente à propagação de trincas devido à mudança microestrutural da fase tetragonal para monoclínica na região de propagação da trinca, promovendo aumento volumétrico de 3-4% que resulta em tensões de compressão que irão tentar fechar a trinca ou dificultar sua propagação (Piconi & Maccauro, 1999; Blatz *et al.*, 2004; Guazzato *et al.*, 2004b). Baseada nestas características, as cerâmicas à base de zircônia apresentam abrangente e promissora utilização na Odontologia e, atualmente são utilizadas como implante dental, pinos pré-fabricados, infra-estrutura para prótese sobre implante, bráquete ortodôntico e infra-estrutura protética para coroa e prótese fixa (Derand & Derand, 2000; Ozcan *et al.*, 2007; Wolfart *et al.*, 2007; Aboushelib *et al.*, 2008).

O processo de união química às cerâmicas à base de zircônia é um desafio devido à alta concentração de cristais inerentes à sua composição. Vários métodos que promovem aumento da rugosidade têm sido investigados na literatura, sempre com o intuito de promover união micromecânica satisfatória à superfície da zircônia. Dentre estes métodos, destacam-se a cobertura de sílica seguida do processo de silanização (Amaral *et al.*, 2007), spray de plasma (Derand *et al.*, 2005), associação entre jateamento com partículas de óxido de alumínio e o monômero MDP (10-metacriloxil decil dihidrogenofosfato) (Wolfart *et al.*, 2007), *primers* para cerâmica (Aboushelib *et al.*, 2008), Er: YAG Laser (Spohr *et al.*, 2008) e condicionamento com infiltração seletiva e maturação induzida por sinterização (Aboushelib *et al.*, 2007; Aboushelib *et al.*, 2008). Em todos estes estudos, foi observado aumento imediato da resistência de união, porém estudos clínicos e laboratoriais que simulam o desgaste ao longo do tempo, bem como o estabelecimento de um protocolo simples e de fácil acesso aos clínicos, ainda não está fundamentado na literatura.

Baseado nestas considerações, a durabilidade de uma restauração cerâmica depende de fatores como grau de conversão do agente de cimentação e interação deste agente de cimentação com a superfície interna da cerâmica. Dessa forma, avaliar a atenuação de luz através de cerâmicas de diferentes composições e o modo de ativação do agente de cimentação, bem como avaliar o efeito de um novo glaze à base de porcelana de

baixa fusão na superfície de uma cerâmica à base de zircônia em associação com os tratamentos convencionais, apresentam-se como tentativas no intuito de promover maior durabilidade das restaurações de cerâmicas, servindo de base para estudos longitudinais e buscando predizer o comportamento clínico das restaurações.

CAPÍTULOS

Esta tese está baseada na Resolução CCPG/002/06UNICAMP, que regulamenta o formato alternativo para teses de Mestrado e Doutorado. Dois capítulos contendo artigos científicos compõem esta tese, conforme descrito abaixo:

Capítulo 1: Effect of Activation Modes and Different Ceramics on Knoop Hardness Number of Dual Resin Cement. Artigo enviado para publicação no periódico: **Journal of Adhesive Dentistry.**

Capítulo 2: Influence of glazed zirconia on dual luting agent bond strength. Artigo a ser enviado para publicação no periódico: **Journal of Operative Dentistry.**

CAPÍTULO 1

Effect of Activation Modes and Different Ceramics on Knoop Hardness Number of Dual Resin Cement

Clinical relevance: Polymerization of dual cure resin cement through different ceramics is significantly affected by activation modes, ceramic composition and post activation times.

ABSTRACT

Purpose: To investigate (1) the influence of ceramic compositions on Knoop Hardness Number (KHN) of a resin cement, immediately and 24-hour after polymerization and (2) the effect of different activation modes (direct light activation, light activation through ceramics and chemical activation) on the KHN of resin cement.

Materials and Methods: Ten resin cement discs (Panavia F 2.0, Kuraray Co., Ltd) were fabricated in a Teflon mold covered with a polyester film. These discs were activated either directly using curing light, or chemically without applying light, or through ceramic discs of 1.2 mm thickness. The ceramics evaluated were Duceram, Cergogold, IPS Empress, IPS Empress 2, Procera, Cercon, In Ceram Alumina and In Ceram Zirconia. The KHN was obtained using microhardness test immediately and after 24-hour testing time. Two-way ANOVA and Tukey's test were performed for statistical analysis, with significance set at $p < 0.05$.

Results: The direct activation groups showed higher KHN than the activated through ceramics groups and chemical activation groups for both immediately and 24-hour testing time. The KHN for 24-hour post activation time was superior to the immediately post activation time except for direct activation mode. The glass and di-silicate based ceramics showed higher KHN than alumina and zirconia based ceramics, immediately and after 24-hour.

Conclusion: (1) The reinforced and opaque ceramics result in one of the lowest KHN values, (2) The ceramic composition results in light attenuation, lower polymerization

effectiveness, and lower hardness values, and (3) The 24-hour testing time promotes an improvement of microhardness except for the directly activated mode.

Key words: dental ceramic, hardness, luting agent, thickness, light polymerization.

INTRODUCTION

Dental ceramics are appreciated as highly esthetic restorative materials with optimal esthetic properties that simulate the natural dentition appearance. Other desirable characteristics include translucence, fluorescence, chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to tooth structure.[17] In spite of their many advantages, ceramics are fragile under tensile.[2, 14, 24] Several different ceramic systems are available such as In-Ceram Alumina and In-Ceram Zirconia (Vita Zahnfabrik, Seefeld, Germany), IPS Empress and IPS Empress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein), Cergogold (Degussa Dental, Hanau, Germany), Procera (Nobel BioCare, Gothenburg, Sweden), and Cercon (Degussa Dental, Hanau, Germany).

Luting materials are vitally important for the longevity of dental restorative materials.[1,18] Resin cements offer distinct advantages such as adhesion to both ceramic and dental structure substrates, as well as low solubility, easy manipulation, and favorable esthetic.[10] Higher fatigue and compressive strength of all-ceramic restorations is observed in these cements when compared with glass ionomer cements.[10] However, to achieve a better restoration retention to a tooth structure and allow efficient polymerization of the resin cement, it has to flow smoothly with unbroken continuity.[20,23] Survival of these restorations also depends on the degree of conversion of these luting agents which influences the properties like hardness, wear resistance, water absorption, residual monomer, and biocompatibility.[20]

Dual cure resin cements have the advantage of both chemical cure and light cure materials.[5,7,8,19] Even though the materials are dual cured, an adequate quantity of light is required to initiate the polymerization process.[22] The composition, thickness, opacity, and shade of the ceramic material may attenuate the light from the curing unit which was used to polymerize the resin cement under the ceramic restoration.[7] Light intensity and the distance of the curing unit during polymerization are other important factors that may be concerning while ceramics restorations are luting.[13, 23] Ceramics from different manufacturers have different compositions and crystal content, that may impact the light quantity that passes through them for luting cements activation. Once crystalline ceramics are opaque, it could be expected to attenuate more light. Limited information has been published on the composition effect, opacity, and ceramic materials thickness on the light attenuation.

In-Ceram Alumina, an aluminous ceramic with 82 % weight of alumina and infiltrated by glass[25], is indicated for anterior and posterior full crowns and anterior three-unit fixed partial dentures. In-Ceram Zirconia, 67% by weight alumina and 13% zirconia, is indicated for posterior full crowns and posterior three unit-fixed partial dentures. IPS Empress and Cergogold, glass-ceramic materials containing leucite crystals[1], are indicated only for fabricating single unit crowns. IPS Empress 2 is a multiphase glass ceramic with 60% by volume and consists of two crystal phases: lithium disilicate based ($Li_2O \cdot SiO_2$) crystals as the main phase and lithium orthophosphate crystals as the second phase.[12] It is advocated for anterior and posterior full crowns, and anterior three unit fixed partial dentures.[11] Procera, a high density alumina containing 99.5% of aluminum oxide, is indicated for full crowns and laminates. Cercon, a zirconia based ceramic which contains

94% ZrO_2 stabilized by 5% Y_2O_3, is indicated for crowns fabrication and up to four unit posterior fixed partial dentures. Despite the differences among these ceramic materials, similar procedures for photo-polymerization have been used to activate resin cements under these restorations. Manufacturers of Duceram, Cergogold and IPS Empress recommend resin cement for luting, while IPS Empress 2, In Ceram, Procera and Cercon claim that their restorations can be luted with either resin cement or conventional glass ionomer cements. Furthermore, some zirconia based ceramic systems recommend the use of chemically-activated resin cement Panavia 2.1.

The light attenuation from curing unit to polymerize the resin cement under different ceramics has not been adequately studied. Therefore, the purposes of this study were: (1) to evaluate the influence of ceramics composition on KHN of a resin cement, immediately and 24-hour after polymerization; (2) the effect of different activation modes (direct light-activation, light activation through ceramics and chemical activation) on the KHN of a resin cement. The null hypotheses were: (1) Ceramics do not affect the KHN of the resin cement. (2) Time conditions do not affect the KHN of the resin cement.

MATERIAL AND METHODS

Materials, brand names, manufacturers, composition and batch used are listed in Table 1.

Ceramic Specimen Fabrication

Porcelain Duceram Plus: Shade dentin A3 was condensed in a metallic mold to form a cylinder of 8 ($\pm$ 0.01) mm that was sintered in a ceramic furnace (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany), according to manufacturer's instructions. After, it was sectioned under water with a diamond disc at low speed to obtain discs with 1.2 mm thickness, which were then finished and glaze fired.

Cergogold: A wax pattern of 8 mm diameter and 1.3 mm thickness was sprued and invested using Cergofit investment (Degussa Dental). It was then placed in a burnout furnace (7000-5P; EDG Equipments Ltda, Sao Carlos, Brazil) to eliminate the wax. The Cergogold ingot (shade A3) was pressed in an automatic press furnace (Cerampress Qex, Ney Dental Inc, Bloomfield, Conn.). After cooling, the specimen was divested with air abrasion using 50-µm glass beads at 4-bar pressure, followed by 100-µm aluminum oxide at 2-bar pressure, to remove the refractory material and finally with 100-µm aluminum oxide at 1-bar pressure. It was then sectioned under water with a diamond disc at low speed to obtain 3 discs with 1.2 mm thickness, which were finished and stain fired.

IPS Empress: Wax patterns of 8 mm diameter and 1.3 mm in thickness were sprued and invested in IPS Empress investment (Ivoclar-Vivadent, Schaan, Liechtenstein) and then eliminated in a burnout furnace (7000-5P; EDG Equipments Ltda, Sao Carlos,

Brazil) by heating the refractory die. Simultaneously, the IPS Empress ingots (shade A3) and the alumina plunger were heated at an increase of 3°C per minute to 850°C and held for 90 minutes. After the above procedure was completed, the investment, plunger, and ingot were transferred to a furnace (EP 500; Ivoclar-Vivadent, Schaan, Liechtenstein) that increased the temperature to 1180°C. After pressing the melted ingot into the mold and slowly allowing to cool at room temperature, the ceramic was divested with air abrasion using 50µm glass beads at 2-bar pressure, then ultrasonically cleaned in a special liquid (Invex liquid; Ivoclar-Vivadent) for 10 minutes, washed in running water, and dried. The ceramic disc was then treated with 100µm aluminum oxide at 1-bar pressure. They were then sectioned under water with a diamond disc at low speed to obtain 1.2 mm thickness. The discs were finished and stain fired.

IPS Empress 2: Wax patterns of 8 mm diameter, and 0.7 mm thickness were sprued and invested in IPS Empress 2 Speed investment (Ivoclar-Vivadent, Schaan, Liechtenstein). The wax was eliminated in a burnout furnace (700-5P; EDG Equipments Ltda, São Carlos, Brazil). Then, the investment, plunger, and 2 ingots of IPS Empress 2 (shade 300) were transferred to a furnace (EP 500, Ivoclar-Vivadent, Schaan, Liechtenstein) and automatically pressed in accordance with manufacturer's instructions. After cooling to room temperature, the ingots were divested with air particle abrasion 50-µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid; Ivoclar-Vivadent), washed in running water, and dried. It was then treated with 100-µm aluminum oxide at 1-bar pressure. Porcelain Eris shade dentin A3 (Ivoclar-Vivadent) was

applied and fired over the di-silicate disc. The porcelain was ground and submitted to finishing and glaze firing to achieve 0.5 mm, providing a total disc thickness of 1.2 mm.

Procera: A brass plate of 8 mm diameter and 0.5 mm thickness was fabricated on a lathe (Nardini ND 250 BE, Sao Paulo, Brazil). The plate was measured after finishing by using a precision electronic micrometer (Electronic Micrometer; LS Starrett, Athol, MA) with an accuracy of 0.002 mm. It was then sent to Gothenburg, Sweden and a ceramic plate of sintered high-purity aluminum-oxide ceramic was fabricated following the CAD/CAM technique used by Nobel Biocare (Gothenburg, Sweden). Porcelain AllCeram shade dentin A3 (Degussa Dental, Hanau, Germany) was applied and fired over the alumina disc. The porcelain was ground and submitted to finishing and glaze firing to achieve 0.7 mm. Thus, a 1.2 mm thickness disc was obtained.

In-Ceram Alumina and In-Ceram Zirconia: A mold of stainless steel (20 x 20 x 5 mm) with a central depression 8 mm diameter and 0.5 mm thickness was obtained. An impression of this model was made with polyvinyl siloxane, and then duplicated in a plaster (Special plaster; Vita Zahnfabrik, Bad Sackingen, Germany). The aluminum oxide powder or the aluminum zirconia powders were mixed with a special liquid as instructed by the manufacturer. The slurry mixture was then painted into the depression in the special plaster die and fired at 1120° C in the furnace (Inceramat II; Vita Zahnfabrik) for 10 hours. Glass infiltration was achieved by coating the aluminum oxide frameworks with glass powder (silicate-aluminum-lanthanum) mixed with distilled water, and fired for 4 hours at 1100° C. Then, the excess glass was removed by use of a fine-grained diamond (Renfert, Hilzingen, Germany). Subsequently, the specimens were air abraded using 100-μm aluminum oxide at

a pressure of 3-bar. Porcelain VM7™ shade dentin A3 (Vita Zahnfabrik, Seefeld, Germany) was applied and fired over the infiltrated alumina and zirconia disc. The porcelain was ground and submitted to finishing and glaze firing to achieve a total disc thickness of 1.2 mm.

Cercon: A wax pattern of 8 mm diameter and 0.4 mm in thickness was obtained. The wax model was placed in the Cercon brain (DeguDent) unit for scanning. The confocal laser system measured the wax to an absolute precision of 10 µm and reproducibility of < 2 µm, scanning was accomplished in 4 minutes. A Cercon base blank of pre-sintered zirconia was milled and then sintered to a fully dense structure in the Cercon Heat (DeguDent) at 1350° C for 6 hours. The specimens were finished by using 100-µm aluminum oxide at a pressure of 3-bar. Cercon Ceram S shade dentin A3 (DeguDent, Hanau, Germany) was applied and fired on the zirconia disc. The porcelain was ground, finished, and glazed to achieve a total disc thickness of 1.2 mm thickness.

Resin cement activation

The resin cement Panavia F 2.0 (Kuraray Co. Ltd, Osaka, Japan), shade A3, was mixed according to manufacturer's directions and inserted in a nylon mold having a centered hole with a 5.0 (±0.01) mm diameter and 1.0(±0.01) mm thickness. The nylon mold was pre-coated with black paint (Colorgin Spray, Sherwin-Wiliams do Brasil Ind Com Ltda, São Bernardo do Campo, Brazil) to limit light transmission through the ceramic and the resin cement only.[22] A polyester film (25 µm thickness) was placed above the mold.

The cement was mixed under controlled temperature 23 (±1) ℃ and relative humidity (higher than 30%), according to ISO 4049.

For the activation modes, the resin cement was chemically activated (chemical activation mode) and photo/chemical activated by two modes: directly photo-activated (direct activation mode) and photo activated through the discs manufactured for 8 different ceramics (activated through ceramic mode). Two post-cure times were investigated, immediately and 24-hour post-cure. In immediately post-cure, specimens were evaluated between 10-20 minutes after light-activation or between 20-30 minutes for the chemically-activated groups. They were then stored in dry and dark condition at 37 ° C. In 24-hour post-cure, specimens were stored in dry and dark conditions at 37℃ for approximately 24 hours.

Specimens in which the cement was light-activated through different ceramic discs, the discs were interposed between the tip of the light source unit and the polyester film that covered the resin cement before irradiation. The resin cement was light-activated by a quartz-tungsten-halogen light unit XL 2500 (3MESPE, St. Paul, MN, USA) with an irradiance of 650 mW/cm2 for 40 seconds. The light intensity of the curing unit was measured with a hand held radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT, USA).

Knoop Hardness Testing

Ten discs of resin cement for each group tested were finished through 1200 SiC discs and subjected to a universal indenter tester (HMV – 2, Shimadzu, Tokyo, Japan) for Knoop Hardness testing (KHN). Measurements were obtained at 40 X magnification and

values were obtained at 100 μm from the irradiated surface after a force of 50 grams-force was applied for 15 seconds. Three indentations were made in each specimen (n=30), with a distance of 1 mm between them and mean was calculated for each specimen. The KHN was calculated automatically by the tester's software. Data were analyzed statistically using two-way analysis of variance (ANOVA) and multiple comparisons were conducted by Tukey's test. All tests were performed at $p < 0.05$.

RESULTS

The means and standard deviations of KHN for each group are shown in Table 2 and Figure 1. The KHN of the resin cement was not only affected by the ceramic composition, but also by the post activation testing time (p=0.00001).

The direct activation mode showed higher statistical significance KHN than activated through ceramics groups and chemical activation groups for both testing time, immediately and 24-hour.

Both testing time, immediately and 24-hour, Duceram, Cergogold, IPS Empress and IPS Empress 2 showed higher statistical significance KHN than Procera, Cercon, In Ceram Alumina and In Ceram Zirconia.

Through ceramics activation groups and chemical activation groups, 24-hour testing time showed higher statistical significant KHN than immediately, but statistical significance was not observed for direct activation mode.

Immediately testing time, the direct activation mode was statistically superior to the activated through ceramics groups and chemical activation mode. Duceram and Cergogold showed KHN statistically significant higher than IPS Impress 2, and IPS Empress showed intermediate KHN. Procera and Cercon showed lower KHN than IPS Empress 2, however, higher than chemical activation mode. On the other hand, the KHN for In Ceram Alumina and In Ceram Zirconia groups did not show significant statistical difference than the chemical mode activation.

For 24-hour testing time, the activated through ceramics groups showed intermediate KHN between direct activation mode (higher values) and chemical activation mode (lower values). Duceram and Cergogold showed a statistically significant higher KHN than IPS Empress 2, and IPS Empress showed intermediate values. Procera, Cercon, In Ceram Alumina and In Ceram Zirconia showed a statistically significant lower KHN within the activated through ceramics groups.

DISCUSSION

The direct mode resulted in a statistically significant higher KHN than the activated through the ceramics groups and chemical mode groups for both testing time. The KHN for 24-hour post activation was always statistically superior to the immediately post activation time except for direct activation mode. The glass and di-silicate based ceramics showed statistically higher KHN than the alumina and zirconia based ceramics in both testing time (Table 2 and Figure 1). Therefore, the null hypotheses evaluated in this study were rejected.

The present study suggested the ceramic type and composition are critical factors for the hardness development in indirectly activated dual-cured resin luting tested. Furthermore, it could be assumed that the degree of polymerization depends on the interaction between ceramic composition and light attenuation, and the indirect activation decreases the level of irradiance reaching the luting material. As a result, the polymer network development could be affected by diminishing the monomer conversion, interfering with the type and degree of cross-linking.[17] The results of the present study confirm that the most translucent ceramics, like Duceram, Cergogold, IPS Empress and, IPS Empress 2, showed statistically higher KHN than the most opaque ones, like Procera, Cercon, In Ceram Alumina and, In Ceram Zirconia (Table 2 and Figure 1).

The direct activation mode showed statistically higher KHN than the activated through ceramics groups and chemical activation mode groups for both testing time, immediately and 24-hour (Table 2 and Figure 1). Therefore, the self-curing component

itself might not be enough to ensure the adequate polymerization.[6] In addition, the results showed the luting agent formulation is a critical factor for the development of hardness indirectly dual-cured activated. Even though it is difficult to predict whether different clinical performances are likely to occur for restorations luted under similar conditions to those tested in the present study, the use of high intensity light source or increase the light exposure time is advisable when cementing more opaque ceramic restoration.[17] In the present study the KHN was measured immediately after mixing and 24-hour. Further researches to study the effect of time on the KHN over a long period of time are encouraged.

Since it has been shown that even well polymerized resin cements can release some residual monomers, it is reasonable to conclude that more substances would elute from poorly polymerized resin cement. These substances have the potential to irritate soft tissues and pulp, stimulate the growth of bacteria and promote allergic reactions.[16] Furthermore in a real clinical situation, many times we confront with a cement line that stays in direct contact with the gingiva in the intrasulcular margin of crown preparation.[3,4,7,9] Therefore, those monomers released from not well polymerized cement would potentiate the gingival irritation, and lead adverse clinical consequences such as microleakage, postoperative sensitivity, discoloration, and secondary caries.[3,4,7,9,15,16,26]

As shown in results, the immediately testing time showed lower KHN than 24-hour testing time. Similar findings have been observed in previous studies evaluating different cements.[13, 16, 21, 23, 25] Hence, restorations placed are unstable at the immediately time, and could be dislocated during the mastication process. Thus we recommend that a clinical protocol may be created for a cementation process that includes additional time to

allow adequate polymerization. Moreover, this protocol should include an advice to the patients to avoid chewing hard-based diet for at least 24 hours.

CONCLUSIONS

Within the limitations of this study the following conclusions may be drawn:

1. Polymerization through ceramic affects the activation mode.

2. Ceramic composition affects the polymerization of the dual resin cement.

3. An improvement of microhardness was found after 24-hour testing time except for directly activated mode.

4. Alumina and zirconia based ceramics demonstrate a significantly greater decrease in Knoop hardness values than did silica and di-sicate based ceramics.

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Table 1. Materials, Brand Names, Manufacturers, Composition and Batch Used.

| Materials	Brand Name	Manufacturer	Composition*	Batch [#]
Feldspatic porcelain	Duceram Plus	Degussa Dental, Hanau, Germany	K_2O_2, Al_2O_3, SiO_2, SnO, ZrO, Na_2O, CaO, pigments	0122/5
Feldpatic ceramic	Cergogold	Degussa Dental, Hanau, Germany	SiO_2, Al_2O_2, K_2O, Na_2O, CaO	2018/12
Feldspatic porcelain	Duceragold	Degussa Dental, Hanau, Germany	SiO_2,Al_2O_3,K_2O,Na_2O,CaO,BaO, SnO_2,Li_2O,F,Sb_2O_3,$CeO2$,B_2O_3,TiO_2	0230/4
Leucite ceramic	IPS Empress	Ivoclar,Vivadent, Schaan, Liechtenstein	SiO_2, Al_2O_3, K_2O, Na_2O, CeO_2, B_2O_3, CaO, BaO, TiO_2	F68542
Lithium di-silicate ceramic	IPS Empress 2	Ivoclar-Vivadent, Schaan, Liechtenstein	SiO_2, Al_2O_3, La_2O_3 , MgO, ZnO, K_2O, Li_2O, P_2O_5	G02567
Feldspatic porcelain	Eris	Ivoclar-Vivadent, Schaan, Liechtenstein	SiO2, K2O, ZnO, ZrO2, Li2O, CaO, Na2O, Al2O3)	F69117
Alumina high content ceramic	Procera	Nobel Biocare, Gothenburg, Sweden	Al_2O_3	03/2003
Feldspatic porcelain	AllCeram	Degussa Dental, Hanau, Germany	SiO_2,Al_2O_3,K_2O,Na_2O,CaO,Y_2O_3,SnO_2,Li_2O, ZrO_2	0182/1
Alumina ceramic	In Ceram Alumina	Vita Zanfabrik, Seefeld, Germany	Al_2O_3, La_2O_3, SiO_2, CaO, other oxides	10780
Zirconia ceramic	In Ceram Zirconia	Vita Zanfabrik, Seefeld, Germany	Al2O3 (62%), ZnO (20%), La2O3 (12%), SiO2 (4.5%), CaO (0.8%), other oxides (0.7%)	22470
Feldspatic porcelain	VM7	Vita Zanfabrik, Seefeld, Germany	SiO_2,Al_2O_3,B_2O_3,Na_2O,K_2O,CaO and TiO_2	62530
Zirconia ceramic	Cercon	DeguDent, Hanau, Germany	ZrO_2, Y_2O_3, Hf O_2, SiO_2,Al_2O_3	2001866 9
Feldspatic porcelain	Cercon Ceram S	DeguDent, Hanau, Germany	SiO_2,Al_2O_3,K_2O,Na_2O and silicate glasses	30240
Resin cement	Panavia F 2.0	Kuraray, Osaka, Japan	Paste A: BPEDMA, MDP, DMA, silica, barium sulfate, dibenzoylperoxide. Paste B: N,N-diethanol-p-toluidine, silica sodiumfluoride, Polyethyleneglycol, glycerine, sodium benzenesulfinate cont. gel.	51581

*[#] Manufactures information

Table 2. Knoop Hardness Number (KHN) of Panavia F 2.0 Resin Cement as a Function of Activation Mode and Testing Time (Mean ± Std dev) n=30.

Activation Mode	Testing time	
	Immediate	24 Hours
Direct	51.56 (2.93) Aa	52.03 (3.63) Aa
Duceram	22.26 (1.85) Bb	31.33 (2.84) Ba
Cergogold	22.09 (3.83) Bb	32.20 (3.07) Ba
IPS Empress	18.09 (2.02) BCb	30.19 (3.91) Bca
IPS Empress 2/Eris	15.85 (3.15) Cb	26.96 (3.30) Ca
Procera/Allceram	12.77 (1.54) Db	20.10 (2.61) Da
Cercon/CerconCeram	11.20 (1.79) Db	17.79 (2.06) Da
In Ceram/VM7 D	9.19 (0.73) DEb	18.90 (1.54)Da
In Ceram/Zirconia	8.78 (0.90) DEb	18.71 (2.06) Da
Chemical	8.63 (1.29) Eb	14.98 (2.09) Ea

Upper cases denote significant differences between Activation Modes (p≤ 0.05) and lower cases denote significant differences between Testing Times (p≤ 0.05) (Tukey's test).

Figure 1. Knoop Hardness Number (KHN) of Panavia Resin Cement as a Function of Activation Mode and Testing Time (Immediate and 24 hours).
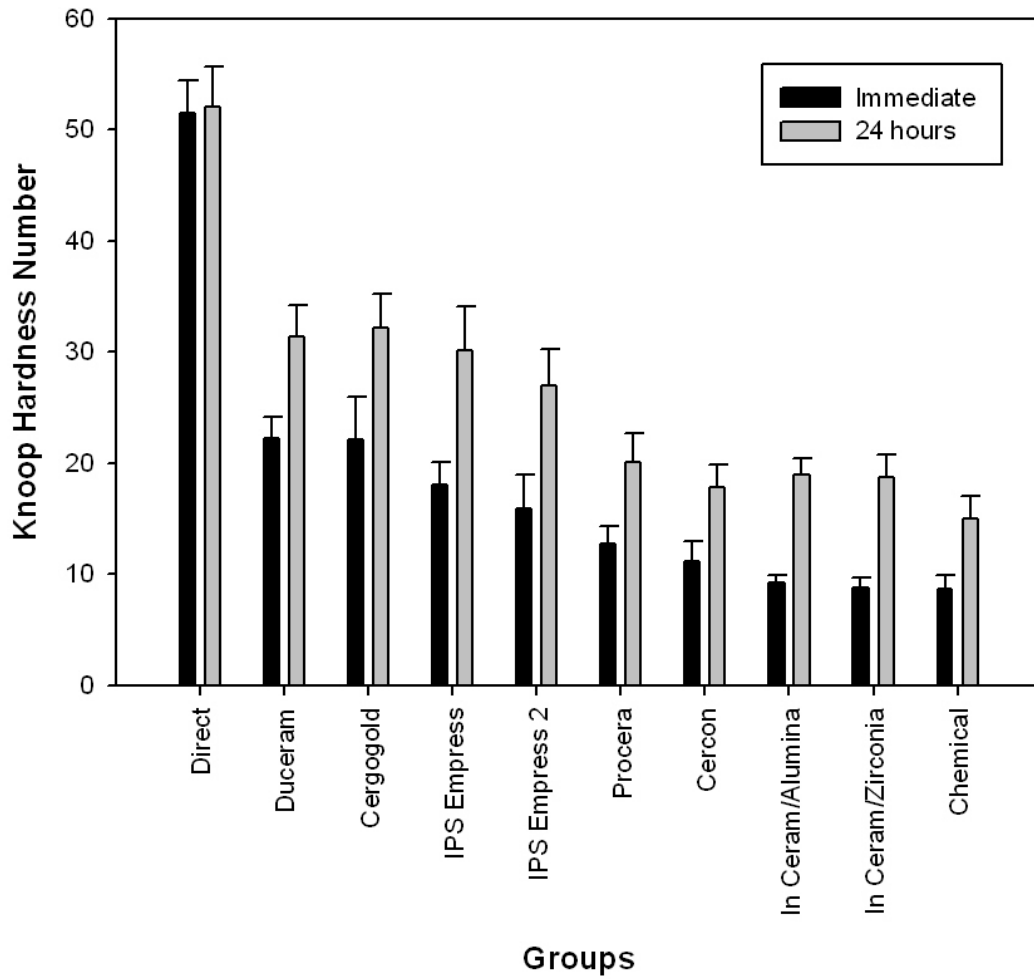
CAPÍTULO 2

Influence of glazed zirconia on dual luting agent bond strength

Running title: Bond strength of a dual luting agent to zirconia

Clinical significance: Treatment of zirconia ceramic surfaces with a low fusing porcelain glaze significantly increased the bond strength of dual resin cement to the ceramic surface.

Abstract

Purpose: The aim of this study was to evaluate a novel surface treatment that use a low fusing porcelain glaze for promoting a bond between zirconia-based ceramic and a dual resin luting agent and to analyze the association of this surface treatment with air particle abrasion, hydrofluoric acid etching, and silanization treatments.

Materials and Methods: Eighty Yttrium-stabilized tetragonal Zirconia ceramic discs (Cercon, Degudent, Hanau, Germany) were fabricated and received eight different surface treatments: Group I - 110 μm aluminum oxide particle abrasion. Group II - 110 μm aluminum oxide particle abrasion and silane, Group III - 50 μm aluminum oxide particle abrasion, Group IV - 50 μm aluminum oxide particle abrasion and silane, Group V - low fusing porcelain glaze and hydrofluoric acid, Group VI - low fusing porcelain glaze, hydrofluoric acid, and silane, Group VII - low fusing porcelain glaze and 50 μm aluminum oxide particle abrasion, and Group VIII - low fusing porcelain glaze, 50 μm aluminum oxide particle abrasion, and silane. After the treatments, Enforce resin cement (Dentsply, Caulk, Milford, DE, USA) was used to fill an iris that was cut from micro bore Tygon tubing which was put on the ceramic surface and photo-cured for 40 seconds by 800mW/cm^2 halogen light curing intensity. The microshear bond test was performed at a cross-head speed of 0.5 mm/min until failure, totalizing ten specimens per ceramic disk and ten disks per group. Data and multiple comparisons were statistically analyzed using one-way ANOVA and Tukey's test respectively, both at 0.05 significance level.

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Results: The bond strength was only affected by the different treatments, but not by the silanization. The low fusion porcelain glaze groups in association with air particle abrasion or hydrofluoric acid showed bond strength values statistically superior to the groups that utilized the conventional air particle abrasion treatments with 50 and 110 µm aluminum oxide particles.

Conclusion: The low fusing porcelain glaze treatment promotes a significant improvement of zirconia-resin bond strength, zirconia-based ceramic surfaces are not affected by the air particle abrasion with aluminum oxides, and silane agent not influences the bond strength to zirconia-based ceramic.

Key words: dental ceramic, agent luting, microshear, zirconia, low fusing porcelain glaze.

Introduction

The unique mechanical properties, chemical stability, and biocompatibility make zirconia-based ceramic an attractive core material for fabrication of all-ceramic restorations.[1] Combined with CAD/CAM technology, the fabrication of complex restorations incorporating zirconia cores has become a completely digitalized process and a relatively simple procedure.[2,3] The flexural and fracture resistance are considerably higher than those of other dental ceramics[4] and the zirconia-based ceramic showed a distinct mechanism of stress-induced transformation toughening, which means the material that undergoes microstructural changes when submitted to stress.[4-6] Zirconia-based ceramic can actively resist crack propagation through a transformation from a tetragonal to a monoclinic phase at the tip of a crack, which is accompanied by a 3-4% volume increase.[5] Based on these characteristics, zirconia-based ceramic is used as a prosthetic implant for medical and dental applications, posts, implant abutments, orthodontic brackets and frameworks for crown and bridges.[2,7,8,9]

Long-term durable bond strength to ceramic surface is the aim for the dental clinical application and depends to the micromechanical and chemical interaction between luting agent and ceramic surface. The retention and the stability of the ceramic restorations primarily depend on the adhesive bond strength, which must be strong enough to resist the expected functional loads.[10] The luting of a zirconia restoration can be done with zinc phosphate or with modified glassionomer cements.[11] However, the advantages of resin luting agents, for example marginal seal, good retention and improvement of fracture

resistance, have made them frequently more used even for high strength ceramics.[11,12] Several studies investigated the bond strength to zirconia-based ceramic and several methods are purposed for promote a durable chemical and micromechanical bond with zirconia. The conventional treatments for surface ceramic such as oxides air abrasion and hydrofluoric acid etching are not able to promote a strong and stable bond with zirconia.[13] The air-abrasion might affect the ceramic surface by creating microcracks which might reduce the fracture strength of the ceramic[14] and the hydrofluoric acid etching combined with silanization, which is used with other glass and disilicate-based ceramics, was not successful with acid resistant and glass-free zirconia ceramics.[7,13]

In the last years, the literature showed new treatments that aimed optimal bond strength to zirconia-based ceramic. However, a chemical bonding to zirconia was limited by the inertness of the ceramic composition and various surface roughening methods were investigated to promote an optimal bond to zirconia, such as silica coating followed by silanation,[15] plasma spraying,[11] association between air particle abrasion with the phosphate ester monomer (MDP),[8] ceramic primers,[7] Er:YAG laser,[16] selective infiltration etching technique,[7] and heat-induced maturation.[10] For all this treatments was observed an immediately increase of the bond strength, however, the association between bond strength increase and durability ahead of clinical performance, as well as the development of a simple and easier surface treatment protocol for the clinicians, are still not full defined and needs more clinical and longitudinal laboratories researches for promote a safe and easy protocol for zirconia ceramic.

The purpose of this study was to evaluate a novel surface treatment that use a low fusing porcelain glaze for promotes a bond between zirconia-based ceramic and a dual

resin luting agent, and analyzing the association of this surface treatment with the conventional air particle abrasion, hydrofluoric acid etching and silanization treatments. The null hypothesis was that the ceramic conventional treatments and a novel glaze surface treatment not influence the microhardness of a dual cured agent luting.

Materials and Methods

Ceramic Surface Treatments

Eighty ceramic discs based on Yttrium-stabilized tetragonal Zirconia (Cercon, Degudent, Hanau, Germany) were fabricated; all measuring 16 mm in diameter and 1 mm thickness. The ceramic discs were randomly assigned to eight treatment sequences (10 discs for each) and then received one of the following surface treatments:

Group I: 110 µm aluminum oxide particle abrasion as finished by the manufacturer. No additional treatment was applied, but the ceramic surface was washed with tap water for 1 minute, ultrasonically cleaned in water bath for ten minutes, and air-dried.

Group II: No additional treatment was applied, but the ceramic surface was washed with tap water for 1 minute, ultrasonically cleaned in water bath for ten minutes, and air-dried. A silane agent (Scotchbond Ceramic Primer, 3M ESPE, Germany) was applied on the ceramic surface and allowed to dry for 5 minutes.

Group III: The ceramic surface received air particle abrasion with 50 µm aluminum oxide for 5 seconds at 4-bar pressure. The distance of the tip from the ceramic surface was approximately 4 mm, and washed with tap water for 1 minute, ultrasonically cleaned in water bath for ten minutes, and air-dried.

Group IV: The ceramic surface received air particle abrasion with 50 µm aluminum oxide for 5 seconds at 4-bar pressure. The distance of the tip from the ceramic surface was approximately 4 mm. and washed with tap water for 1 minute, ultrasonically

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cleaned in water bath for ten minutes, and air-dried. A silane agent (Scotchbond Ceramic Primer, 3M ESPE, Germany) was applied on the ceramic surface and allowed to dry for 5 minutes.

Group V: At the beginning a low fusing porcelain glaze was applied on the ceramic surface with a brush # 1 (Ney, Germany) and sintered following manufacturer's instructions. After that, the glaze was conditioning by acid etched with hydrofluoric at 10% (Dentsply, USA) during 20 seconds and washed with tap water for 1 minute, and finally ultrasonically cleaned in water bath for ten minutes, and air-dried.

Group VI: At the beginning a low fusing porcelain glaze was applied on the ceramic surface with a brush # 1 (Ney, Germany) and sintered following manufacturer's instructions. After that, the glaze was conditioning by acid etched with hydrofluoric at 10% (Dentsply, USA) during 20 seconds and washed with tap water for 1 minute, and finally ultrasonically cleaned in water bath for ten minutes, and air-dried. A silane agent (Scotchbond Ceramic Primer, 3M ESPE, Germany) was applied on the ceramic surface and allowed to dry for 5 minutes.

Group VII: At the beginning a low fusing porcelain glaze was applied on the ceramic surface with a brush no 1 (Ney, Germany) and sintered following manufacture's instruction. After that, the glaze excess was removed with air particle abrasion with 50 µm aluminum oxide for 5 seconds at 4-bar pressure. The distance of the tip from the ceramic surface was approximately 4 mm, and washed with tap water for 1 minute, ultrasonically cleaned in water bath for ten minutes, and air-dried.

Group VIII: At the beginning a low fusing porcelain glaze was applied on the ceramic surface with a brush no 1 (Ney, Germany) and sintered following manufacture's

instruction. After that, the glaze was conditioning with air particle abrasion with 50 µm aluminum oxide for 5 seconds at 4-bar pressure. The distance of the tip from the ceramic surface was approximately 4 mm. and washed with tap water for 1 minute, ultrasonically cleaned in water bath for ten minutes, and air-dried. A silane agent (Scotchbond Ceramic Primer, 3M ESPE, Germany) was applied on the ceramic surface and allowed to dry for 5 minutes.

Bonding procedure

The materials used in this study are listed in Table 1.

After the treatments, in order to prepare the resin cement cylinder for cementation, equal lengths of Enforce resin cement (Dentsply, Caulk, Milford, DE, USA) base and catalyst pastes were mixed for 20 seconds and then used to fill an iris that was cut from micro bore Tygon tubing (TYG-030, Small Parts Inc., Miami Lakes, FL) with an internal diameter and height of approximately 0.75 and 0.50 mm, respectively (Figure 1). The Tygon tubing containing resin luting agent was put on the ceramic surface and photo-cured for 40 seconds by $800mW/cm^2$ halogen light curing intensity. In this manner, each ceramic surface was bonded at ten different locations with the resin cylinders. The assembly of ceramic/resin luting agent was stored at room temperature (23° C $\pm$ 2° C) for 1 hour prior to removal of the Tygon tubing, then, the specimens were immersed in distilled water at 37° C for 24 hours before proceeding for the microshear bond test.

Microshear bond test

Before the test, all the ceramic/resin cylinder interfaces were analyzed with an optical microscope 40 X (Olympus, Tokyo, Japan) for bonding defects. The cylinders with apparent interfacial gap formation, bubble inclusion, or any other defects were excluded and replaced by another one. Three sets of ceramic/resin luting agents (30 cylinders of resin cement to each treatment group) were used for each test group.

The assembly of the ceramic plate and the resin cement was adhered to the testing device using cyanoacrylate adhesive (Superbond, Loctite, Sao Paulo, Brazil), which in turn was placed in a universal testing machine (EMIC DL-3000, São José dos Pinhais, PR, Brazil) for microshear bond testing (Figure 2). An edge of stainless steel with 0.5 mm in thickness was fixed on the superior part of a universal testing machine, and was gently adapted against the ceramic/resin luting agent interface. A microtensile bond test was applied to each specimen at a cross-head speed of 0.5 mm/min until failure, totalizing ten specimens per ceramic disk and three disks per group.

Statistical Analysis

The data were statistically analyzed using one-way ANOVA and multiple comparisons were made using Tukey's test. Statistical significance level was set at $\alpha =$ 0.05.

Results

The bond strength of the dual resin cement was only affected by the different treatments ($p < 0.001$), but not by the silanization (Table 2). The means and standard deviations of microshear bond strength values for the groups tested are shown in Table 3.

The groups that utilized the low fusing porcelain glaze in association with air particle abrasion or hydrofluoric acid showed bond strength values statistically superior to the groups that utilized the conventional air particle abrasion treatments with 50 and 110 µm aluminum oxide particles (Figure 3 and 4) which is the treatment recommended for the zirconia manufacturer ($p < 0.001$).

The low fusing porcelain glaze treatment in association with hydrofluoric acid, independent of the silanization process, showed bond strength values statistically superior to the others groups tested. The groups that associated low fusing porcelain glaze and 50 µm aluminum oxide air particle abrasion showed statistically intermediate bond strength values. The air particle abrasion (groups I, II, III, and IV) showed bond strength values statistically inferior to glaze (groups V, VI, VII, and VIII), and not differing in relation to different oxides granulations tested, as well as silanization process (Table 3).

Discussion

The surface roughness methods to densely sintered zirconia ceramics are limited by the inertness and hardness of this ceramics. [10] These characteristics become difficult to create grooves for micro retention and chemical bond for the optimal interaction with luting agents. The novel surface treatment evaluated that use a low fusing porcelain glaze was able to promote superior bond strength values between zirconia-based ceramic and a dual resin luting agent, and the association with the traditional surface treatment methods, conventional air particle abrasion (Figures 3 and 4) and hydrofluoric acid etching (Figure 5), showed strong bond strength in relation to the zirconia manufacture's treatment (Table 3). Based on the results obtained, the proposed null hypothesis that the ceramic conventional treatments and a novel glaze surface treatment would not influence the microhardness of a dual cured agent luting was not accepted.

In attempt of promote an optimal bond strength to zirconia-based ceramic, several roughening methods have been investigated. [7,810,11,15,16] For all this roughening methods, that utilized silica coating followed by silanation, [15] plasma spraying, [11] association between air particle abrasion with the phosphate ester monomer (MDP), [8] ceramic primers, [7] Er:YAG laser, [16] selective infiltration etching technique, [7] and heat-induced maturation; [10] was observed an immediately increase of the bond strength. However, the methods' durability ahead of clinical performance and aged laboratories researches require further investigations for stabilized a safe protocol for the zirconia-based ceramics. The low fusing porcelain glaze treatment was able to promote a bond strength increase and showed to be a

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simple treatment that correlated traditional treatments for the clinicians like hydrofluoric acid or air particle abrasion, becoming an advantage.

The ceramic surface treatments should follow some pre-requisites like not compromise the integrity of the ceramic, not promote additional problems with crown adaptation and good interaction with the agent luting.[11] On the contrary, the surface roughening methods resulted in structural damage, material loss, grain pullout, and creation of sharp crack tips,[17] thus, the bonded restorations becomes more susceptible to radial cracking under functional loads.[10] The low fusion porcelain glaze promotes a vitreous layer into the zirconia-based ceramic surface. The zirconia-base ceramic surface treated with glaze seems to be similar to glass-based ceramic surface and become possible the creation a ceramic surface that is susceptible to air abrasion and hydrofluoric acid treatments, as well as an interaction with the silane agent for promoting a chemical reactivity surface.

The highest bond strength was observed for the low fusion porcelain glaze groups, and the association with hydrofluoric acid conditioning showed superior statistical bond strength values and promoted a better interaction within the cementation process (Table 3). The use of silane agent did not improve the bond strength to zirconia-based ceramic for groups tested and this results corroborated with the some studies which was observed that silane utilization not presented good performance for high crystals ceramics.[6,11,18,19] The zirconia manufacture's treatment, as well as air particle abrasion with 50 μm aluminum oxide, is not able to promote satisfactory bond strength to zirconia surface due the ceramic composition, and other pre-treatments that aimed to promote roughness increase or chemical bond interaction are necessaries for the immediately bond strength.

The optimal interaction between ceramic surface and agent luting are necessary for the success and the long-term durability restoration.[19] The mechanical properties make zirconia-based ceramic a promising core material for fabrication of all-ceramic restorations and others dental applications;[1,2,7,8,9] and new clinical and artificial aging researches are still necessary for promote a safe and easy protocol of the zirconia surface treatment. The low fusing porcelain glaze treatment seems to be a promising treatment for the zirconia-based ceramics and should be considered in new researches like the influence of this treatment in the marginal adaptation.

Conclusions

Within the limitations of this study, these findings may be drawn:

1. The bond strength to zirconia-based ceramic surface is not affected by the air particle abrasion with aluminum oxides.

2. The low fusing porcelain glaze treatment promotes a significant improvement of zirconia-resin bond strength.

3. Silane agent not influences the bond strength to zirconia-based ceramic.

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Table 1. Material type, brand name, manufacturer, and composition.

Material type	Brand name	Manufacturer	Compositions*
Zirconia Ceramic	Cercon	Degudent	ZrO_2 stabilized by Y_2O_3
Low Fusion Porcelain Glaze	Cercon Ceram Kiss	Degudent	Vitreous porcelain and pigments
Resin Cement	Enforce	Dentsply	BisGMA, BHT, EDAB, TEGDMA, Fumed Silica, Silanized Barium, Aluminum Borosilicate Glass (66% wt)
Ceramic Primer	Scotchbond Ceramic Primer	3M ESPE	Bisphenol A polyethoxy dimethacrylate 3-Methacryloyloxypropyl trimethoxysilane

* Manufactures information

Table 2. Results of one-way ANOVA for microshear bond test of Enforce dual resin cement bonded to zirconia-based ceramic.

Source of Variation	Df	Sum of Squares	Meam Square	F	P value
Treatment	7	15367.386	5122.462	96.781	<0.001
Error	90	1026.5252220	11.4058358	---	---

**Table 3.** Means and standard deviations of micro shear bond strength in MPa.

Zirconia-Based Ceramic Treatment							
110 μm Air Particle Abrasion		50 μm Air Particle Abrasion		Glaze + 50 μm Air Particle Abrasion		Glaze + Hidrofluoric Acid	
Nosilane	Silane	Nosilane	Silane	Nosilane	Silane	Nosilane	Silane
4.06 (1.36) C	5.33 (1.58) C	3.95 (1.24) C	6.02 (1.61) C	17.45 (8.55) B	18.41 (7.47) B	20.75 (8.29) A	25.17 (8.37) A

Capital letters denote significant differences among the zirconia-based surface treatments ($p < 0.001$) (Tukey's test).
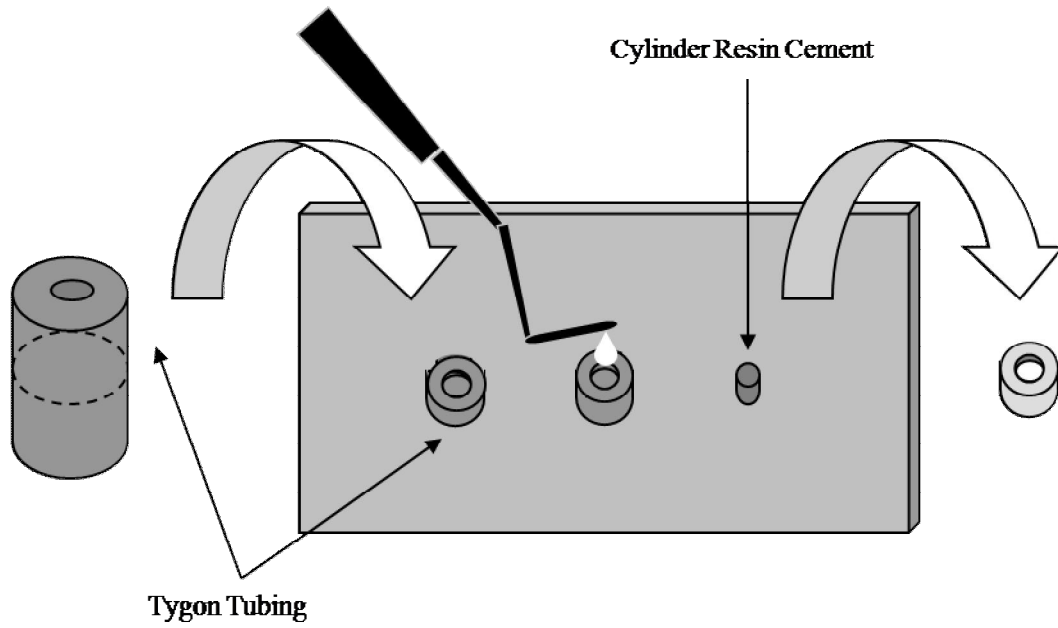
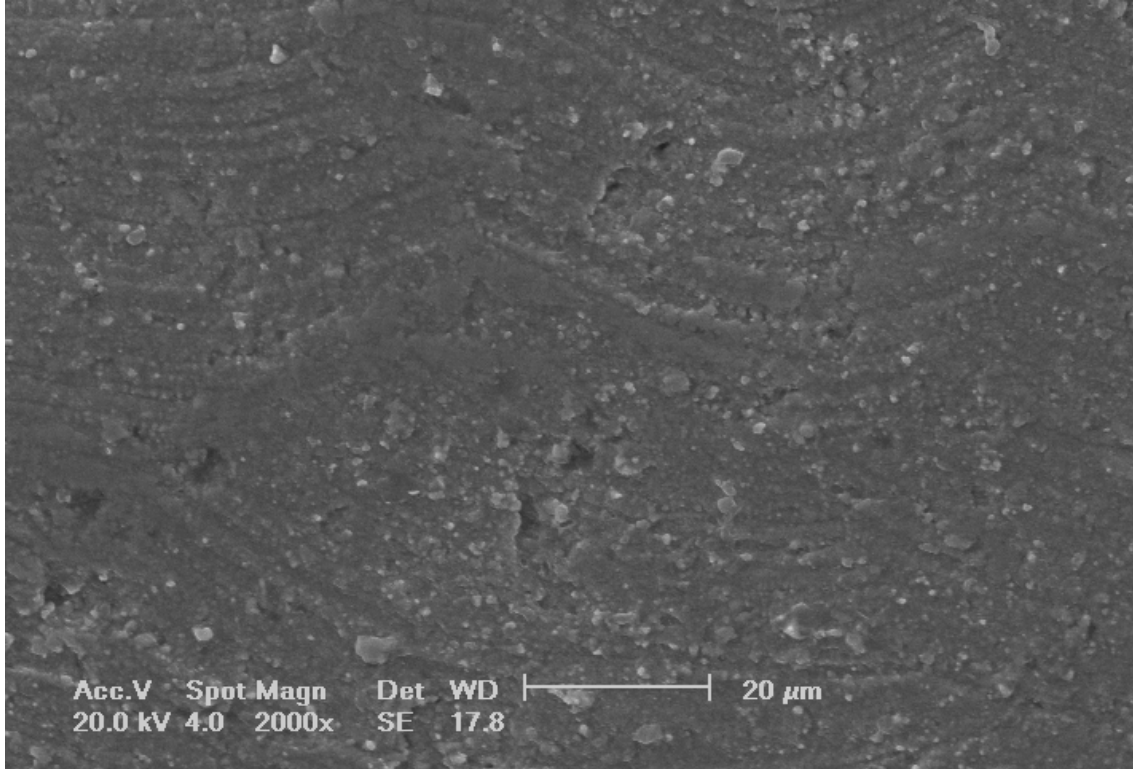**Figure 1.** Bonding procedure of Enforce resin cement to zirconia-based ceramic
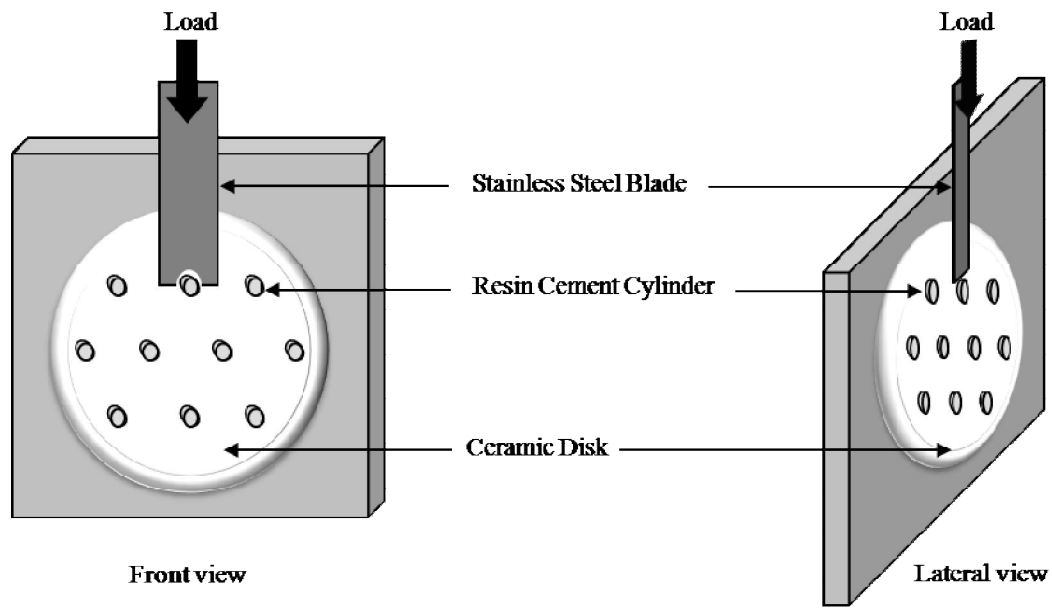
Figure 2. Diagram of micro shear bond Test
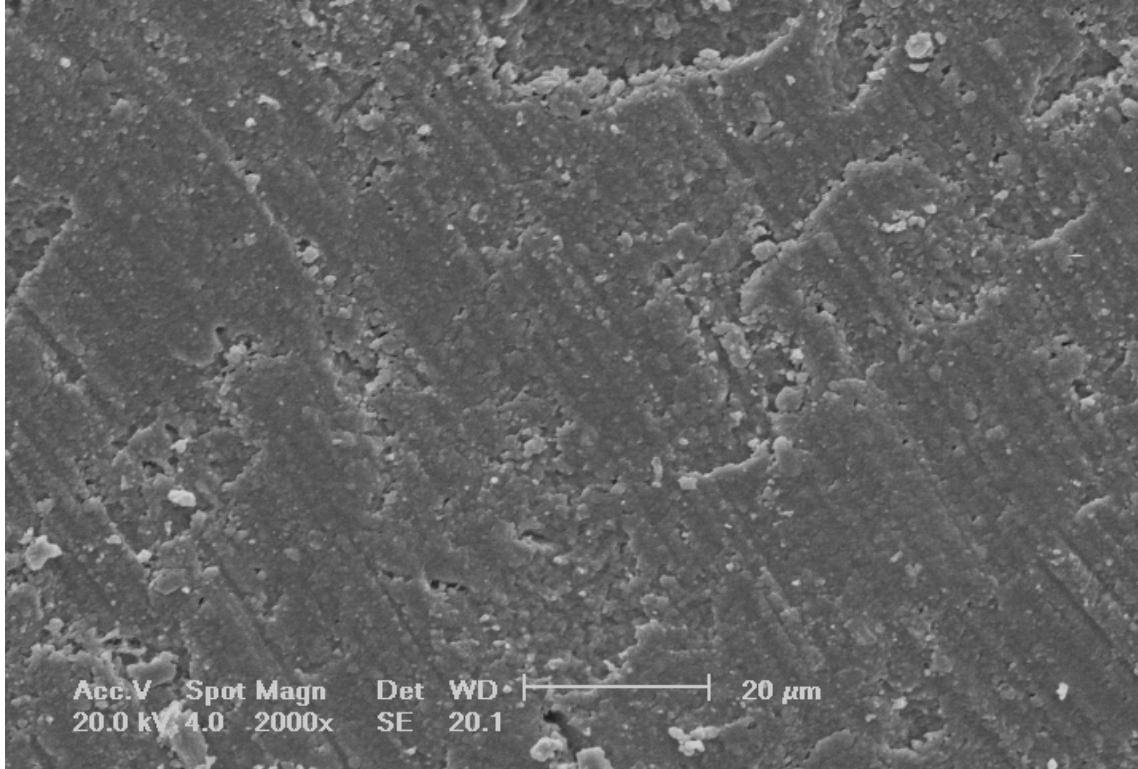
Figure 3. Zirconia-base ceramic treated with 110 µm aluminum oxides air particle abrasion
(Manufacture's recommendation)
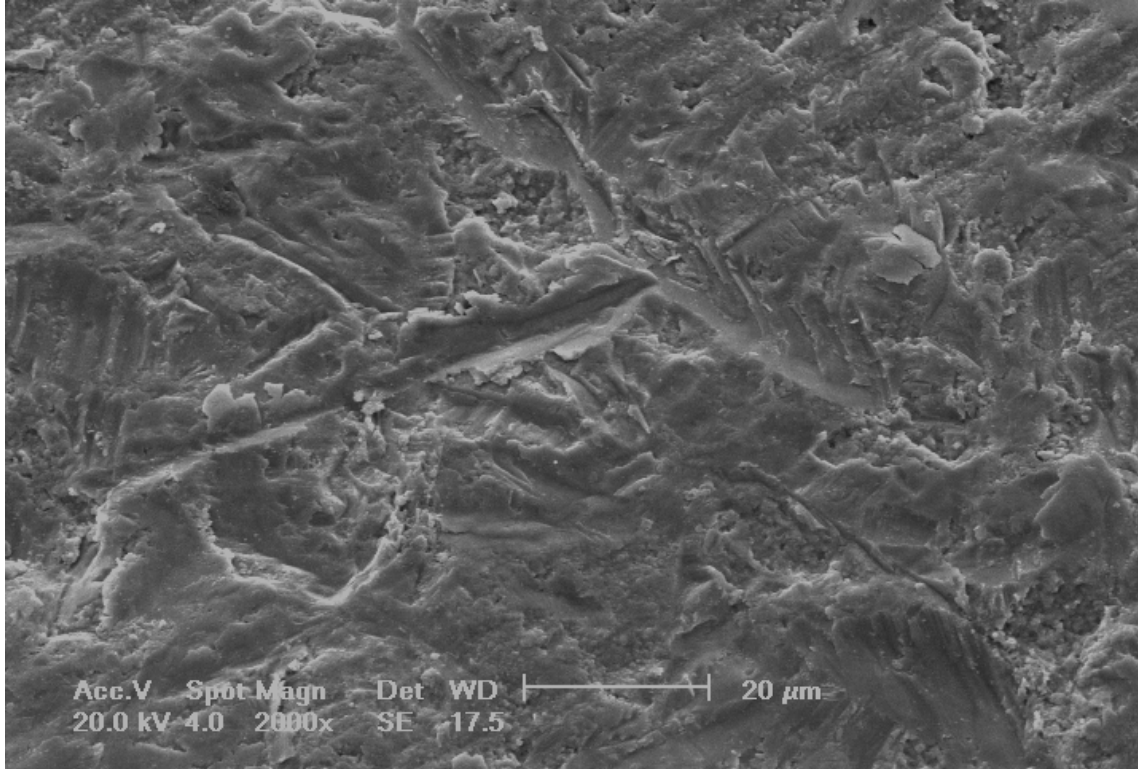
Figure 4. Zirconia-base ceramic treated with 50 μm aluminum oxides air particle abrasion

Figure 5. Zirconia-base ceramic treated with 10% hydrofluoric acid conditioning

CONCLUSÕES

Com base nos resultados obtidos e dentro das limitações destes estudos *in vitro*, pode-se concluir que:

1. A fotoativação de um agente de cimentação resinoso dual através de cerâmicas reforçadas e opacas resulta em menores valores de KHN.

2. A composição e a espessura da cerâmica promovem atenuação da luz e menor efetividade de polimerização, resultando em menor valor de KHN.

3. Superfícies cerâmicas à base de zircônia não são significantemente afetadas pelo jateamento com partículas de óxido de alumínio.

4. O tratamento à base glaze promove um aumento significante da resistência de união à cerâmica à base de zircônia.

5. A silanização não promove aumento significante da resistência de união para a cerâmica à base de zircônia.

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