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Influência do conteúdo de carga inorgânica nas propriedades ópticas e mecânicas de agentes de cimentação resinosos

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Dissertação apresentada, como requisito parcial, para obtenção do grau de Mestre em Odontologia, Programa de Pós-Graduação em Odontologia, Faculdade de Odontologia, Universidade Federal de Pelotas.

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Dedicatória

Aos meus pais, José Carlos Barbon e Maria Elisabete de F. Jardim,
que sem medir esforços fizeram de meus sonhos seus

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Epígrafe

Que os vossos esforços desafiem as impossibilidades,
lembrai-vos de que as grandes coisas do homem
foram conquistadas do que parecia impossível.
Charles Chaplin

Notas Preliminares

A presente dissertação foi redigida segundo o Manual de Normas para Dissertações, Teses e Trabalhos Científicos da Universidade Federal de Pelotas de 2013, adotando o Nível de Descrição 4 – estrutura em Artigos, descrita no Apêndice D do referido manual. <http://sisbi.ufpel.edu.br/?p=documentos&i=7> Acesso em: 10 de maio de 2017.

O projeto de pesquisa contido nesta dissertação é apresentado em sua forma final após qualificação realizada em 03 de dezembro de 2015 e aprovado pela Banca Examinadora composta pelos Professores Doutores Noéli Boscato, Rafael Ratto de Moraes e Marina Kaizer.

Resumo

BARBON, Fabíola Jardim. **Influência do conteúdo de carga inorgânica nas propriedades ópticas e mecânicas de agentes de cimentação resinosos.** 2017. 119f. Dissertação de Mestrado em Odontologia – Programa de Pós Graduação em Odontologia. Universidade Federal de Pelotas, Pelotas, 2017.

O conteúdo de carga inorgânica do agente de cimentação resinoso poderia influenciar a qualidade e durabilidade da adesão entre cerâmica e agente de cimentação, bem como, as propriedades ópticas das restaurações cerâmicas. Dois artigos *in vitro* serão apresentados neste estudo. O artigo 1 avaliou a influência do conteúdo de carga inorgânica de agentes de cimentação resinosos (ACRs) e o uso de adesivo na resistência de união à microtração (μTBS) e morfologia da interface adesiva da cerâmica cimentada. ACRs com baixo, intermediário, e alto conteúdo de carga inorgânica (respectivamente 55%, 65% e 75%/peso) foram preparados. O módulo de elasticidade (*E*), viscosidade, coeficiente de Poisson *(v),* grau de conversão *(GC)* dos agentes de cimentação (n=3) foram também avaliados. O RelyX Veneer (3M ESPE) foi usado como referência comercial. Os blocos de cerâmica (Vitablocks Mark II; Vita Zahnfabrik) foram cimentados aos de resina composta e de acordo com o agente de cimentação, e uso ou não de adesivo, originaram 8 grupos (n=30). Dados de *E*, *v, GC* e viscosidade foram analisados usando análise de variância de uma via seguida do teste de Tukey (α= 0,05) e os intervalos de confiança (IC 95%) foram calculados para μTBS, resistência característica (σ_0) e modulo de Weibull (*m).* O aumento do conteúdo inorgânico foi associado com o aumento da viscosidade dos experimentais ACRs, enquanto *v* e *GC* não foram influenciados. O uso do adesivo melhorou a μTBS e σ_0 do comercial e experimental ACR com alto conteúdo de carga. O artigo 2 avaliou a influência do conteúdo de carga inorgânica na alteração de cor (ΔE_{00}), coordenadas individuais de cor CIEL*a*b* e parâmetros de translucidez (PT) de simulados laminados cerâmicos (LCs). O ΔE_{00} e PT foram obtidos baseados nas coordenadas de cor CIEL*a*b* medidos com um espectrofotômetro. O ΔE_{00} foi calculado pela diferença de cor CIEDE2000 obtida para cada espécime de cerâmica (1,2 mm x 0,8 mm, A1C) cimentada ao substrato de resina composta (1,6 mm × 1,2 mm, A2D) usando os cimentos resinosos testados (cor translúcida) em 3 condições (antes, imediatamente e 24 h após a cimentação). O PT foi calculado a partir das medidas dos espécimes obtidas sobre fundo branco e preto padrão. Morfologia da superfície dos ACR´s e composição do comercial foram analisadas. Os testes de análise de variância de uma e de duas vias seguidos do teste Tukey foram utilizados respectivamente para calcular TP and ΔE_{00}. Para as coordenadas CIEL*a*b*, cada par de variáveis foi comparada usando o teste t de Student (α= 0,05). Em geral, os ACRs testados apresentaram valores de ΔE_{00} clinicamente visíveis nas três condições. Para todos os ACRs, maiores valores de ΔE_{00} foram observados entre as medidas obtidas antes e imediatamente após a cimentação. A variação da quantidade de conteúdo

inorgânico não influenciou significativamente o PT dos simulados LCs. As coordenadas de cor individuais L*, a* e b* foram dependentes da cimentação.

Palavras-chave: cerâmica; fractografia; resistência mecânica; análise weibull; cor; estética; translucidez; cimentação.

Abstract

BARBON, Fabíola Jardim. **Influence of the inorganic filler content on optical and mechanical properties of resin-based luting agents.** 2017. 119p. Dissertation Master degree in Dentistry. Graduate Program in Dentistry. Federal University of Pelotas, Pelotas, 2017.

The inorganic filler content of resin-based luting agent could influence the quality and durability of the adhesion between ceramic and luting agent, as well as, the optical properties of the ceramic restorations. Two in *vitro studies* are presented in this study. The article 1 evaluated the influence of inorganic filler content of resin-based luting agents (RBLAs) and the adhesive use on the bond strength (μTBS) and interface morphology of luted feldspar ceramic. RBLAs with low, intermediate and high inorganic filler content (55%, 65% and 75% of mass fraction, respectively) were prepared. The modulus of elasticity (*E*), Poisson's coefficient (*v*), viscosity and degree of conversion (*DC*) of these luting agents were also measured (n=3). The RelyX Veneer (3M ESPE) was used as commercial reference. Feldspar ceramic blocks (Vitablocks Mark II; Vita Zahnfabrik) were luted to composite resin, originating eight groups according to the different RBLAs and use of adhesive (n=30). Data of *E*, *v, DC* and viscosity were analyzed using one-way analysis of variance followed by post-hoc Tukey's test (α= 0.05) and confidence intervals for the means (95% CI) were calculated for μTBS, σ_0, and *m*. The increase of their inorganic filler content was associated with increased *E* and viscosity of the experimental RBLAs, while *v* and *DC* were not influenced. The use of adhesive improved the μTBS and σ_0 for the commercial and RBLAs with high filler content. The article 2 evaluated the influence of inorganic filler content of resin-based luting agents (RBLAs) on color change (ΔE_{00}), CIEL*a*b*individual color coordinates) and translucency parameters (TP) of simulated ceramic laminate veneer (CLV). The ΔE_{00} and PT were calculated based on the CIEL*a*b* color coordinates measured with a spectrophotometer. The ΔE_{00} was calculated by the color difference CIEDE2000 obtained for each ceramic specimen (1.2 mm x 0.8 mm, A1C) luted to the composite resin substrate (1.6 mm × 1.2 mm, A2D) using the luting agents In the (translucent shade) under three conditions (baseline, immediately and 24h after luting). The PT was calculated from the measurements of the specimens obtained over white and black backgrounds. Surface morphology of RBLAs and composition of commercial also were analyzed. One-way and two-way analyses of variance with a post-hoc Tukey test were used respectively to calculate TP and ΔE_{00}. For CIEL*a*b* coordinates, each pair of variable was compared using the Student t-test (α= 0.05). Overall the RBLAs tested presented clinically visible ΔE_{00} values under the three conditions. For all RBLAs, higher ΔE_{00} values were observed between measurements obtained before and immediately after luting. The variation of inorganic filler content did not influence significantly the TP of simulated CLV. The L*, a*, and b* individual color coordinates were cementation dependent.

Key-words: ceramics; fractography; mechanical strength; weibull analysis; color; aesthetic; translucency.

Sumário

1 Introdução

Atualmente a busca por procedimentos estéticos aumentou ainda mais e os sistemas cerâmicos têm sido bastante utilizados nestes tratamentos restauradores (RASHID et al., 2016). Os laminados cerâmicos (LCs) representam uma modalidade de tratamento estético comumente usada na prática odontológica tendo em vista que mínima remoção de estrutura dental é necessária para a sua confecção e porque restabelecem satisfatoriamente os padrões estéticos e anatômicos dos elementos dentais (GUESS et al., 2011; DAOU 2015; RASHID et al., 2016).

Inúmeros sistemas cerâmicos estão disponíveis no mercado, entre eles os blocos de cerâmica feldspática para usinagem tais como os Vitabocs Mark II (Vita Zahnfabrik, Bad Säckingen, Alemanha). Este sistema é uma opção bastante utilizada para confecção de restaurações em dentes anteriores devido às suas excelentes propriedades estéticas atribuídas à sua composição com alto conteúdo de fase vítrea (VARGAS, 2002; FASBINDER, 2002; CONRAD; SEONG; PESUN, 2007; GUESS et al., 2011). Em função deste mesmo aspecto, as restaurações confeccionadas a partir da cerâmica feldspática apresentam limitadas propriedades mecânicas (MARTINS et al., 2010) e seu sucesso clínico e longevidade dependem da sua união ao substrato dental a partir dos procedimentos de cimentação (ADDISON; MARQUIS; FLEMING, 2008).

A qualidade desta união depende dos mecanismos adesivos que são controlados em parte pelo tratamento de superfície da cerâmica e em parte pelos materiais utilizados nos procedimentos adesivos (FLEMING et al., 2006). Adicionalmente, os procedimentos que promovem a união da cerâmica à estrutura dental reforçam as propriedades mecânicas do conjunto restaurador (ADDISON; MARQUIS; FLEMING, 2007; SPAZZIN et al., 2017). Este mecanismo de reforço obtido pelos procedimentos adesivos é parcialmente atribuído às propriedades intrínsecas dos cimentos tais como o módulo de elasticidade (SPAZZIN et al., 2017) e diferentes tamanhos de carga (VALENTINI et al., 2014).

A adesividade da Vitabloc Mark II aos cimentos resinosos é baseada em mecanismos de retenção micromecânica, originados principalmente pela ação de

ácidos, e pela união química propiciada pelo uso de organosilanos. Quando estes materiais são devidamente utilizados propiciam o aumento da energia de superfície e diminuem o ângulo de contato entre os materiais favorecendo o processo adesivo entre a superfície cerâmica e o cimento resinoso (ÖZCAN et al., 2004; DELLA BONA et al., 2004; LEE et al., 2015). Inúmeros mecanismos que têm sido propostos para explicar o reforço obtido pela cimentação adesiva. Entre eles incluem-se o selamento e a interrupção da propagação das microtrincas existentes no interior do material e aquelas originadas pelo condicionamento ácido (ADDISON; MARQUIS; FLEMING, 2007; 2008).

A literatura reporta que o aumento da quantidade de matriz inorgânica dos cimentos resinosos melhora as propriedades mecânicas deste material (SPAZZIN et al., 2017). No entanto, associado a este aspecto também ocorre o aumento da sua viscosidade que pode culminar no aumento da espessura de película do cimento. Tal aspecto poderia limitar a penetração do material nas retenções criadas pelo tratamento de superfície (BEUN et al. 2009). Isso seria um aspecto desfavorável tendo em vista que se as retenções originadas pelo condicionamento não forem adequadamente preenchidas pode ocorrer o comprometimento da durabilidade e desempenho da interface adesiva e a diminuição da resistência de união (LEE et al, 2015; SPAZZIN et al., 2016). O uso do adesivo poderia auxiliar no preenchimento das retenções originas na superfície da peça cerâmica; no entanto, ainda há pouca evidência científica sobre a sua influência na morfologia da interface adesiva (YOUM et al., 2015). Desse modo, parece que a quantidade de carga do agente de cimentação e o uso de adesivo poderiam ter influência na qualidade e durabilidade da interface adesiva uma vez que estes fatores poderiam aumentar ou reduzir o embricamento mecânico do cimento nas retenções originadas pelo tratamento de superfície (BEUN et al., 2009; YOUM et al., 2015).

Adicionalmente, é importante notar que os procedimentos que promovem a união da cerâmica à estrutura dental não só reforçam as propriedades mecânicas do conjunto restaurador, como também influenciam na mimetização da restauração aos dentes remanescentes (ADDISON; MARQUIS; FLEMING, 2007). De fato, a cor final do conjunto cerâmica-cimento-substrato é um complexo fator que pode ser influenciada por vários aspectos incluindo aqueles relacionados às propriedades dos cimento (PERRONI et al., 2016), principalmente quando usados abaixo de delgadas

restaurações confeccionadas com cerâmica de alta translucidez (BOSCATO et al., 2015; DOZIC et al., 2003). Estudos prévios reportaram que a cor do agente de cimentação (PERRONI et al., 2016) e o tipo de cimento (ALMEIDA et al., 2015) podem causar significativas alterações no resultado estético final de LCs. A quantidade de carga poderia também influenciar a cor final de LCs desde que a luz pode ser transmitida através de sua fina estrutura, permitindo assim a reflexão da cor do substrato dental e do agente de cimentação (CHAIYABUTR et al., 2011).

Baseado no que acima foi exposto, o objetivo deste estudo foi avaliar a influência do conteúdo de carga inorgânica de agentes de cimentação resinosos, e o uso ou não de adesivo previamente à cimentação, na resistência de união à microtração e morfologia da interface adesiva, bem como nas propriedades ópticas de simulados LCs.

2 Projeto de pesquisa

2.1 Introdução

A aparência estética do sorriso é considerada cada vez mais referencial de saúde e sucesso nos dias atuais e os tratamentos odontológicos têm buscado atingir esses resultados. Entre os materiais restauradores estéticos, a cerâmica representa uma excelente alternativa, devido à possibilidade de reproduzir a beleza e naturalidade de um dente, além das propriedades biomecânicas semelhantes à estrutura dental (HÖLAND et al., 2000; JUNIOR; OLIVEIRA, 2007). Entretanto, as cerâmicas são frágeis e apresentam baixa resistência mecânica, quando submetidas principalmente às tensões de tração, o que compromete seu desempenho clínico em alguns aspectos (MARTINS et al., 2010).

O mercado odontológico oferece uma grande variedade de sistemas cerâmicos classificados geralmente pela composição química e processamento laboratorial (GOMES et al., 2008). Dentre estas, as cerâmicas feldspáticas como a Vitabloc Mark II (Vitablocs Mark II for Cerec; Vita Zahnfabrik, Germany) são bastante usadas para confecção de laminados em dentes anteriores devido às suas significantes propriedades estéticas, que são atribuídas à sua composição com grande conteúdo de fase vítrea (BURKE; QUALTROUGH; HALE, 1998; FAUNCE, 1977; GUERRA et al., 2007). Em função deste mesmo aspecto, estas cerâmicas apresentam limitadas propriedades mecânicas (MARTINS et al., 2010), tendo seu sucesso clínico dependente dos procedimentos de cimentação (ADDISON; MARQUIS; FLEMING, 2008).

Além disso, o desenvolvimento de técnicas adesivas eficientes e o uso de materiais aprimorados para a cimentação aumenta significantemente a resistência à fratura das cerâmicas (FLEMING et al., 2006). Tal aspecto é muito importante porque, no meio bucal as restaurações indiretas envolvem a sobreposição de materiais, uma vez que o material restaurador será unido à estrutura dental, resultando em conjuntos restauradores com múltiplas interfaces tais como cerâmica-cimento-dente (ADDISON; FLEMING, 2008). A adesividade da Vitabloc Mark II à

cimentos resinosos é baseado em mecanismos de retenção micromecânica originados principalmente pela ação de ácidos e da união química (organosilanos), os quais quando devidamente utilizados têm a propriedade de aumentar a energia superficial e diminuir o ângulo de contato, favorecendo o processo adesivo com o cimento resinoso (ÖZCAN et al., 2004; DELLA BONA et al., 2004; LEE et al., 2015).

Inúmeros mecanismos têm sido propostos para explicar o reforço obtido pela cimentação adesiva, como o selamento de microtrincas no interior do material, interrupção da propagação das mesmas (ADDISON; MARQUIS; FLEMING, 2007a; 2008a) e formação de conjunto cerâmica–cimento–dente que se comporta como corpo único (SOARES et al., 2009). Porém, os aspectos relacionados ao reforço fornecido à cerâmica após a cimentação com cimentos resinosos, ainda não está bem estabelecido, embora já tenha sido proposto que cimentos resinosos deveriam ter um módulo de elasticidade (E) intermediário entre a dentina e cerâmica (ADDISON; MARQUIS; FLEMING, 2007a). No que diz respeito aos cimentos resinosos este módulo pode variar de 1,2 a 16,5 GPa (SASKALAUSKAITE; TAM; MCCOMB, 2008).

Estudos têm mostrado aumento na resistência à flexão biaxial da cerâmica feldspática associado ao aumento do módulo de elasticidade do cimento resinoso (FLEMING; HOOI; ADDISON, 2012, KLINK; HUETTIG, 2013). Porém esses estudos, avaliam cimentos resinosos comerciais, o que dificulta o controle de formulação destes, quando comparados aos cimentos resinosos experimentais.

Atualmente os cimentos resinosos estão sendo mais utilizados porque necessitam de técnicas de uso menos sensíveis (BOSCATO; PEREIRA-CENCI; MORAES, 2014) e de melhorias nas suas propriedades mecânicas. O aumento no conteúdo de partículas do material pode providenciar melhorias nas propriedades mecânicas deste material. Por outro lado, a viscosidade que ocorre em função disso também aumentará a espessura de película, o que pode limitar a penetração do cimento nas retenções criadas na superfície da cerâmica após efetuado o tratamento de superfície (BEUN et al. 2009). Se estas retenções, originadas após o condicionamento com ácido fluorídrico não forem preenchidas, a interface adesiva pode ficar comprometida porque estudos comprovam que se há espaços na área de cimentação a resistência de união diminui (LEE et al, 2015; SPAZZIN et al.,2016).

Esses espaços podem ser preenchidos por adesivo que é utilizado na superfície da peça cerâmica após o tratamento de superfície e antes da cimentação adesiva, porém ainda há pouca evidência científica sobre o que o uso ou não de adesivo e sua polimerização, originam na interface adesiva do conjunto cerâmica-cimento (YOUM et al., 2015). Sabe-se que cimentos resinosos com diferentes tamanhos de partículas (VALENTINI et al., 2014) e variação na quantidade de carga podem alterar a capacidade de molhamento da cerâmica e promover diferentes resultados a testes mecânicos (SPAZZIN et al., 2016). No entanto, sabe-se também que materiais com reduzida quantidade de carga, tais como a resina fluida, têm sido usados com êxito como agentes de cimentação de laminados cerâmicos (ALMEIDA et al., 2015).

Para avaliar a integridade da interface adesiva *in vitro*, estudos sugerem que testes de resistência adesiva à tração podem ser os mais apropriados, pois produzem uma distribuição mais uniforme do estresse nesta interface. Os testes de microtração, com uma área de teste reduzida, produzem resultados ainda mais adequados, porque as falhas ocorrem quase exclusivamente na interface adesiva, permitindo uma análise da real resistência de união (DELLA BONA et al*.,* 2000; FRIEDERICH & KERN 2002; WEGNER; GERDES; KERN, 2002; ZOHAIRY, 2003). Já o teste de resistência à flexão biaxial proporciona conhecimento acerca da performance mecânica e a resistência à flexão de materiais utilizados clinicamente, incluindo as cerâmicas feldspáticas unidas aos cimentos resinosos (ADDISON et al., 2007).

Assim, é importante a avaliação *in vitro* com diferentes formulações de cimentos resinosos experimentais e procedimentos adesivos, com rigor e padronização metodológica para que seja possível apontar quais fatores poderiam promover o melhor desempenho do conjunto restaurador, oferecendo assim longevidade ao tratamento para que estes materiais possam ser usados clinicamente com segurança (ADDISON; MARQUIS; FLEMING, 2007a).

2.2 Objetivo

2.2.1 Objetivo Geral

O objetivo deste estudo será avaliar a resistência à flexão biaxial, a resistência de união à microtração e a morfologia da interface adesiva em função da variação na quantidade de partículas de carga de cimentos experimentais e o uso ou não de adesivo previamente a cimentação.

2.2.2 Objetivos Específicos

i. Obter e caracterizar cimentos resinosos experimentais (módulo de elasticidade, viscosidade e coeficiente Poisson) com diferentes quantidades de carga (% em peso): 55%, 65% e 75%;
ii. Avaliar a resistência à flexão biaxial da cerâmica feldspática unida a cada um dos cimentos experimentais;
iii. Caracterizar a morfologia da interface adesiva do conjunto cerâmica-cimento, e ou cerâmica-adesivo-cimento, nas diferentes condições avaliadas (cimentos experimentais, uso ou não de adesivo) em microscopia eletrônica de varredura;
iv. Avaliar a resistência de união entre a cerâmica feldspática e os cimentos resinosos nas diferentes condições experimentais;
v. Caracterizar o modo de fratura na interface adesiva após o teste de microtração, em microscópios ópticos e eletrônico de varredura.

2.2.3 Hipótese

Será testada a hipótese de que a resistência de união, a resistência à flexão biaxial, e morfologia da interface adesiva serão influenciadas pela quantidade de carga dos cimentos resinosos experimentais, e o uso ou não de adesivo.

2.3 Justificativa

Inúmeros mecanismos têm sido propostos para explicar o reforço das cerâmicas após a cimentação adesiva (ADDISON; MARQUIS; FLEMING, 2007a; 2008a; VALENTINI et al., 2014), fazendo com que o conjunto cerâmica-cimento-dente se comporte como corpo único (SOARES et al., 2006; SOARES et al., 2008).

Sabe-se que o sucesso clínico da cimentação de restaurações cerâmicas vítreas, depende da qualidade e durabilidade da adesão entre cerâmica e cimento resinoso. A qualidade desta adesão depende dos mecanismos adesivos que em parte são controlados pelos tratamentos de superfície que originam a retenção micromecânica e/ou química na superfície da cerâmica, pelo adesivo que penetra nestas retenções promovendo adesão ao cimento, e pelo cimento resinoso propriamente dito. Os cimentos resinosos possuem um papel importante no fortalecimento da cerâmica, sendo que esse conjunto pode ser ainda melhorado devido com o aumento de partículas de carga, o que influência também no módulo de elasticidade. (SPAZZIN et al., 2016)

No que diz respeito a influência do agente cimentante na qualidade e durabilidade da interface adesiva, pode-se elencar entre outros aspectos, aqueles relacionados a quantidade de carga do cimento, uma vez que este fator poderia aumentar ou reduzir o embricamento mecânico do cimento às retenções originadas pelo tratamento de superfície realizado na cerâmica (BEUN et al., 2009; YOUM et al., 2015). As avaliações qualitativas como o modo de falha e a topografia da interface adesiva observadas a partir do uso dos diferentes cimentos, e do uso ou não de adesivo na peça cerâmica, podem trazer importantes informações quando correlacionados com as avaliações quantitativas obtidas nos mais diversos testes mecânicos.

Neste estudo será usado, além dos cimentos resinosos experimentais com diferentes cargas em peso (40%, 60%, 80%), o cimento resinoso comercial RelyX Veneer (3M ESPE, St. Paul, USA), especialmente formulado pelo fabricante para a cimentação de facetas, como controle. Este cimento foi escolhido a partir da análise da quantidade de partícula de carga em peso observada em cimentos disponíveis no mercado odontológico. Tal observação foi realizada através de informações fornecidas pelos fabricantes e pela avaliação de perfis técnicos dos produtos usados atualmente na cimentação de laminados cerâmicos. O RelyX Veneer foi selecionado

para ser o controle porque possui carga de 66% em peso, que é uma carga intermediária aos cimentos experimentais que serão formulados.

2.4 Metodologia

2.4.1. Delineamento experimental

Material	Fabricante
Cerâmica Feldspática (Vitablocs Mark II for Cerec)	Vita Zahnfabrik, GERMANY
Cimento comercial (RelyX Veneer)	3M ESPE, USA
Cimentos experimentais	-
Adesivo (Single Bond)	3M ESPE, USA
Silano (RelyX Ceramic Primer)	3M ESPE, USA
ácido Fluorídrico 10% (Condac Porcelana)	FGM, BRASIL
Resina Composta (Filtek Z350)	3M ESPE, USA

Tabela 1: Materiais utilizados.

Será realizado um estudo experimental *in vitro*, onde os fatores cimento e adesivo serão observados, sendo os seguintes níveis acompanhados de suas variáveis respostas: **(i) Resistência de união à microtração** (**carga do cimento experimental** em 8 níveis: 55%, 65%, 75% [experimentais] ; cimento comercial [controle] com adesivo e 55%, 65%, 75% [experimentais] ; cimento comercial [controle] sem adesivo; **(ii) Resistência à Flexão Biaxial** (**carga do cimento experimental** em 8 níveis: 55%, 65%, 75% [experimentais] ; cimento comercial [controle] com adesivo e 55%, 65%, 75% [experimentais]; cimento comercial [controle] sem adesivo; **(iii) Morfologia da Interface adesiva** (**carga do cimento experimental** em 8 níveis: 55%, 65%, 75% [experimentais]; cimento comercial [controle] com adesivo e 55%, 65%, 75% [experimentais] ; cimento comercial [controle] sem adesivo. Os materiais utilizados nesse estudo estão citados na tabela 1.

Para o teste de resistência **à flexão biaxial** serão confeccionados espécimes em formato de disco, nos quais a cerâmica será unida ao cimento determinado a

cada grupo, (n=30). Para o **teste de resistência de união à microtração** serão confeccionados 5 blocos de cerâmica e de resina composta com dimensões idênticas (6mm × 10mm × 10mm) após cortados serão obtidos corpos-de-prova em forma de barras (n=30 por grupo). O **modo de fratura** resultante do teste de microtração e a **morfologia da interface adesiva** serão avaliados em microscópios ópticos e eletrônico de varredura, a partir da observação de espécimes (n=5). A **caracterização de cada cimento experimental** será realizada pela avaliação do módulo de elasticidade através do teste de dureza Knoop, tenacidade a fratura, viscosidade e grau de conversão, sendo um n=5 para cada cimento experimental.

2.4.2 Divisão dos Grupos

Os grupos desse estudo serão divididos de acordo com o cimento utilizado e ao uso de adesivo ou não. Serão produzidos três cimentos resinosos experimentais, os quais terão diferentes quantidades de carga, em peso (%). Será usado um cimento comercializado como controle, na qual possui indicação para a cimentação de laminados cerâmicos. Os seguintes grupos sem adesivo que serão originados: Grupo **controle (CSA)**, com cimento RelyX Veneer (3M ESPE, St. Paul, USA) sem adesivo; **Grupo C55**, com cimento experimental de baixa carga em peso (55%) e sem adesivo; **Grupo C65**, com cimento experimental de carga intermediária em peso (65%) e sem adesivo; **Grupo C75**, com cimento experimental de alta carga em peso (75%) e sem adesivo. Os seguintes grupos com adesivo serão originados: Grupo **controle (CCA)**, com cimento RelyX Veneer (3M ESPE, St. Paul, USA) com adesivo; **Grupo CA55**, com cimento experimental de baixa carga em peso (55%) e com adesivo; **Grupo CA65**, com cimento experimental de carga intermediária em peso (65%) e com adesivo; **Grupo CA75**, com cimento experimental de alta carga em peso (75%) e com adesivo.

2.4.3 Procedimentos

2.4.3.1 Preparo dos cimentos resinosos

Cimentos resinosos experimentais serão obtidos por uma mistura dos monômeros bisfenol-A glidicil dimetacrilato (Bis-GMA), bisfenol-A dimetacrilato etoxilado (Bis-EMA), trietileno glicol dimetacrilato (TEGDMA) e uretano dimetacrilato (UDMA) provenientes do fabricante Esstech Inc. (Essington, PA, EUA). Serão utilizadas partículas de carga silanizadas, micrométricas, baseadas em vidro de Ba-Al-Si. Serão formulados cimentos resinosos experimentais com três diferentes cargas (55%, 65%, 75%)

Criterioso processamento será realizado para que as propriedades dos materiais sejam obtidas com alta precisão e reprodutibilidade. Após mistura dos monômeros, serão adicionadas concentrações em peso dos seguintes reagentes: canforoquinona (0,4% - Sigma-Aldrich, St. Louis, MO, EUA) como fotoiniciador, etil-4 dimetilamino benzoato (0,8% - Sigma-Aldrich) como co-iniciador. Todos os reagentes serão pesados em balança analítica digital com precisão de 0,01mg (modelo DV215CD; Ohaus Discovery, Toledo, São Bernardo do Campo, SP). As concentrações dos reagentes descritos acima condizem com valores médios encontrados em cimentos resinosos comercialmente disponíveis.

2.4.3.2 Determinação de viscosidade dos cimentos resinosos

Um reômetro (Rheometer R/S-CPS; Brookfield Engineering Laboratories, Inc., Middleboro, MA, EUA) com pratos paralelos acoplados a um controlador de temperatura será utilizado para mensurar o comportamento reológico dos materiais. Em seguida, 0,5mL do material será dispensado no prato inferior do reômetro e o prato superior, com diâmetro de 25mm, será deslocado para baixo até obter a distância de 0,05mm entre os pratos. O comportamento reológico será analisado em 30 pontos, com taxa de cisalhamento constante de 100s-1, sob temperatura controlada (23°C) por 30s. Os dados serão obtidos em Pa.s, plotados e analisados qualitativamente.

2.4.3.3 Análise do grau de conversão (C=C) e taxa de polimerização

A reação de polimerização em tempo real será avaliada em espectrofotômetro infravermelho com Transformada de Fourier (Prestige21 Spectrometer; Shimadzu, Tóquio, Japão) equipado com dispositivo de refletância total atenuada composto por um cristal horizontal de diamante. O material (~3μL) será dispensado sobre o cristal de diamante (n=3) e a conversão de C=C será monitorada por 10min através do *software* IRSolution utilizando apodização de Happ-Genzel, faixa espectral entre 1800 e 1500cm^{-1}, 1 scan/s, resolução de 4 cm^{-1} e velocidade de deslocamento de espelho de 2,8mm/s. O grau de conversão de C=C (%), por segundo, será calculado considerando a intensidade da vibração de estiramento de C=C alifáticas na região 1635cm^{-1}. O estiramento simétrico do anel aromático em 1710cm^{-1} será utilizado como padrão interno. Para calcular da taxa de polimerização, os dados obtidos serão plotados em uma curva ajustada pelo parâmetro regressivo não-linear de Hill e analisados qualitativamente.

2.4.3.4 Determinação do módulo de elasticidade e coeficiente de Poisson dos cimentos

Serão feitas barras dos cimentos resinosos (60 mm × 10 mm × 4 mm) utilizando uma matriz previamente fabricada a partir de polivinil siloxano (Yller, Pelotas, RS, Brasil). O equipamento Sonelastic (ATCP Engenharia Física, Ribeirão Preto, SP) será utilizado para obter v e E (n = 3 / cimento). As frequências de vibração naturais do material serão capturadas usando o software após a emissão de um som de um leve impacto mecânico. Um espectro com sinais de freqüência e amplitude, por meio de vibração flexional-torcional, será registrado considerando a isotropia do material.

2.4.3.5 Preparo dos espécimes cerâmicos para o teste de resistência à flexão biaxial

Para obtenção dos espécimes cerâmicos serão utilizados blocos de cerâmica feldspática (I14 A1C Vitablocs Mark II for Cerec; Vita Zahnfabrik, Germany) com as dimensões de 12mm × 14mm × 18mm. Os blocos cerâmicos serão usinados em torno mecânico sob irrigação de água em formato cilíndrico mantendo diâmetro de aproximadamente 12mm. Os blocos cerâmicos serão então seccionados em discos

de aproximadamente 0,8mm de espessura (±0,1mm) em fresagem com CADCAM (Fresadora modelo M1, Zirkonzahn Worldwide, Gais/Sudtirol, Itália).

Após o corte, qualquer desajuste decorrente do corte necessita ser removido do espécime, e isso será feito com turbina de baixa rotação e ponta diamantada de acabamento sob refrigeração à agua. O polimento dos discos será realizado com lixas de SiC granulações 600 e 1200 (Norton S.A.) e água. As dimensões de cada espécime serão aferidas utilizando paquímetro digital com precisão de 0,01mm (Mitutoyo, Tóquio, Japão).

2.4.3.6 Aquecimento do cimento resinoso com alta carga

Para aumentar a superfície de molhamento do cimento resinoso experimental com 75% de carga em peso, o cimento será aquecido a 60ºC, temperatura máxima sem ocorrer polimerização e também a temperatura ambiente de 22ºC. O aquecimento será feito através de estufa com controle de temperatura por 30 min.

2.4.3.7 Procedimentos de condicionamento para o teste de resistência à flexão biaxial

Os discos de cerâmica serão condicionados durante 60s (ADDISON; MARQUIS; FLEMING, 2007b), utilizando gel de ácido fluorídrico 10% (Condac Porcelana 10% - FGM, São Paulo, Brasil). Após os espécimes serão lavados com jato água/ar durante 30s, seguido de secagem com jato de ar durante 30s, segundo indicação do fabricante (FGM, São Paulo, Brasil). Será então aplicado duas camadas do agente de silanização (Silano Agente de União RelyX Ceramic Primer – 3M ESPE, St. Paul, USA) utilizando microbrush e, após 1min, a superfície será seca com jato de ar por 30s e duas camadas finas de adesivo Single Bond (3M ESPE) serão aplicadas.

O cimento resinoso será aplicado sobre o centro da superfície cerâmica condicionada e silanizada, sendo coberto com tira transparente de poliéster pressão será de 5N durante 1 min para extrusão do cimento e espalhamento do mesmo por toda superfície da cerâmica utilizando dispositivo previamente confeccionado (MORAES et al., 2008). Após remoção do excesso de cimento resinoso, utilizando microbrush, o material será fotoativado através da cerâmica por 60s (LED – Radii; SDI Limited, Bayswater, Victoria, Austrália) com irradiância de 1200mW/cm^2. Quaisquer excessos de cimento serão removidos com lâminas de bisturi. A

espessura final do conjunto cerâmica–cimento será mensurada com paquímetro digital, sendo descartados espécimes que por ventura apresentarem espessura de cimento fora da faixa de 120±20µm. Tal espessura será obtida com a interposição de espaçadores entre a placa de vidro e tira de poliéster (ADDISON; MARQUIS; FLEMING, 2007a). Os espécimes serão mantidos em ambiente umidificado. Esse protocolo será seguido para todos os grupos.

2.4.3.8 Teste de resistência à flexão biaxial

A resistência à flexão biaxial dos conjuntos cerâmica–cimento de todos os grupos será determinada conforme metodologia descrita anteriormente (ADDISON; MARQUIS; FLEMING, 2007a; ISGRO; ADDISON; FLEMING, 2011). Os espécimes serão posicionados no dispositivo biaxial “ball on ring” (pistão-anel). Para controle de possíveis irregularidades será colocado sobre o anel do dispositivo um dique de borracha cortado. O disco de cerâmica-cimento será posicionado com a face não-condicionada voltada para o pistão do dispositivo “ball-on-ring”. Uma extremidade esférica de 4mm de diâmetro, acoplada à célula de carga da máquina de ensaios, será posicionada no centro do disco cerâmico e utilizada para aplicação de carga compressiva até a fratura do corpo-de-prova. A tensão de flexão biaxial será calculada no centro do espécime em posições axiais ao longo de sua espessura (posições z), conforme previamente descrito (ADDISON; MARQUIS; FLEMING, 2007a; ADDISON; SODHI; FLEMING, 2010; FLEMING; HOOI; ADDISON, 2012; ISGRO; ADDISON; FLEMING, 2011). O plano neutro (tn) será calculado em função das espessuras (t_1 e t_2) e módulos de elasticidade (E_1 e E_2) da cerâmica e do cimento, respectivamente, como segue:

$$t_n = \frac{E_1^* (t_1)^2 - E_2^* (t_2)^2}{2(E_1^* t_1 + E_2^* t_2)} \qquad E^* = \frac{E}{1 - \nu^2}$$

A tensão de flexão biaxial (σ) poderá ser calculada em posições axiais (z) no centro do espécime, sendo a superfície da cerâmica na interface adesivo ($z = t_1$) e a superfície da resina voltada para o anel ($z = -t_2$), como segue:

$$\sigma = \frac{-3P(1+\nu)(z-t_n)}{2\pi(t_1+t_2)^3}\left[1+2\ln\left(\frac{a}{b}\right)+\frac{1-\nu}{1+\nu}\left(1-\frac{b^2}{2a^2}\right)\frac{a^2}{R^2}\right] \times \left[\frac{E1^*(E_1^*t_1+E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2+(E_1^*t_2^2)^2+2E_1^*E_2^*t_1t_2(2t_1^2+2t_2^2+3t_1t_2)}\right] \quad (0 \le z \le t_1)$$

$$\sigma = \frac{-3P(1+\nu)(z-t_n)}{2\pi(t_1+t_2)^3}\left[1+2\ln\left(\frac{a}{b}\right)+\frac{1-\nu}{1+\nu}\left(1-\frac{b^2}{2a^2}\right)\frac{a^2}{R^2}\right] \times \left[\frac{E_2^*(E_1^*t_1+E_2^*t_2)(t_1+t_2)^3}{(E_1^*t_1^2)^2+(E_2^*t_2^2)^2+2E_1^*E_2^*t_1t_2(2t_1^2+2t_2^2+3t_1t_2)}\right] \quad (-t_2 \le z \le 0)$$

$$\nu = \frac{\nu_1 t_1 + \nu_2 t_2}{t_1 + t_2}$$

em que P será a carga na fratura, v_1 e v_2 os coeficientes de Poisson da cerâmica e resina mensurados previamente (CAVALCANTE et al., 2008), e a, b e R os raios do suporte, da região de carga e do espécime, respectivamente.

2.4.3.9 Preparo dos espécimes cerâmicos para a resistência de união com teste de microtração

Vinte e cinco blocos de cerâmica (I14 A1C Vitablocs Mark II for Cerec; Vita Zahnfabrik, Germany) serão obtidos nas dimensões de 6mm × 10mm × 10mm. A superfície dos blocos cerâmicos será planificada e polida em politriz APL-4 (Arotec Indústria & Comércio, São Paulo, SP, Brasil), empregando-se lixas SiC de granulações 280, 400, 600, 800, 1200 *waterproof* (Carborundum, Saint Gobain Abrasivos LTDA, Iguarassu, SP, Brasil). Os blocos de cerâmica serão distribuídos aleatoriamente em cada grupo experimental de acordo com os cimentos utilizados. Serão obtidos também blocos de resina composta (Filtek Z350 A3 – 3M ESPE, St. Paul, USA) em número e dimensões idênticas aos blocos cerâmicos. Estes blocos serão obtidos a partir da moldagem de um bloco de cerâmica, no qual será

condensada a resina composta em camadas de 2mm, fotopolimerizadas por 40s com fotopolimerizador (LED – Radii; SDI Limited, Bayswater, Victoria, Austrália).

2.4.3.10 Procedimentos de condicionamento e cimentação para a resistência de união com teste de microtração

Os discos de cerâmica serão condicionados durante 60s (ADDISON; MARQUIS; FLEMING, 2007b), utilizando gel de ácido fluorídrico 10% (Condac Porcelana 10% - FGM, São Paulo, Brasil). Após os espécimes serão lavados com jato água/ar durante 30s, seguido de secagem com jato de ar durante 30s, segundo indicação do fabricante (FGM, São Paulo, Brasil). Será então aplicado duas camadas do agente de silanização (Silano Agente de União RelyX Ceramic Primer – 3M ESPE, St. Paul, USA) utilizando microbrush e, após 1min, a superfície será seca com jato de ar por 30s e duas camadas finas de adesivo Single Bond (3M-ESPE) serão aplicadas.

Os blocos de cerâmica serão unidos aos blocos de resina composta com o cimento determinado a cada grupo. Uma carga de 5N será aplicada por 1min sobre os blocos cimentados, após os blocos serão fotopolimerizados por 40s em cada interface. Os blocos de cerâmica unidos à resina serão seccionados em dois eixos, x e y, com disco diamantado com espessura de 127mm × 0,4mm × 12,7mm (Sultrade Comércio e Exportacão Ltda, São Paulo, SP, Brasil) em cortadeira mecânica sempre sob intensa refrigeração com água, obtendo-se corpos-de-prova em forma de barras (n=30 por grupo), com área adesiva de aproximadamente 1,0mm^2 (PASHLEY et al., 1999; DELLA BONA et al., 2000 Os espécimes serão mantidos em ambiente umidificado.

Os espécimes provenientes das faces externas dos blocos serão descartados devido à possibilidade de haver excesso ou ausência de adesivo na interface, que podem influenciar os resultados (VAN NOORT et al., 1991; DELLA BONA et al., 2000). Os critérios para rejeição incluirá presença de defeitos e falhas nos corpos-de prova.

2.4.3.11 Resistência de União com Teste de Microtração

Cada espécime será fixado com adesivo cianocrilato gel (Super Bonder gel, Loctite Ltda, São Paulo, SP, Brasil) ao equipamento de teste Bencor (Danville Co., CA, EUA) e posicionado paralelo ao longo eixo do dispositivo, a fim de minimizar as

forças de torção na zona adesiva. O posicionamento será auxiliado pela guia presente no dispositivo de teste. Somente as extremidades de cada espécime será fixada, permanecendo livre a zona adesiva. Esse dispositivo será fixado na máquina de ensaio universal (DL500; EMIC, São José dos Pinhais, Paraná, Brasil), onde será aplicada uma carga de tração, com velocidade de 0,5mm.min-1 (PASHLEY et al.,1999; DELLA BONA et al., 2000).

A resistência de união de cada espécime será calculada conforme a fórmula R=F/A, onde “R” é a resistência, “F” é a carga para ruptura do corpo-de-prova e “A” é a área do corpo-de-prova. Os valores serão obtidos em Kgf/mm^2 e transformados em MPa (N/mm^2), multiplicando-se esses valores por 9,807 (gravidade). A área adesiva do corpo-de-prova será medida com paquímetro digital logo após o teste. O modo de fratura será examinado em microscópios ópticos e eletrônico de varredura com base nos princípios de fractografia e classificado em falhas adesivas, mistas e coesivas (VALENTINI et al., 2014); falhas adesivas – entre o adesivo e a resina, e entre o adesivo e a cerâmica; falhas coesivas – na cerâmica ou na resina; e ou falhas mistas – que podem ocorrer em ambos os sistemas em conjunto (GARCIA et al., 2008).

2.4.3.12 Análise da morfologia da interface de união através da microscopia eletrônica de varredura

Para avaliar a imbricamento mecânica do cimento na cerâmica, além de homogeneidade e continuidade da união ao longo da interface, discos adicionais de cerâmica serão utilizados. Seis discos de cerâmica serão confeccionados e cimentados a outro disco utilizando um cimento experimental e o cimento controle, totalizando três espécimes cerâmica-cimento-cerâmica para cada cimento (n=24).

Para isso, os espécimes serão cortados perpendicularmente à interface de união. Após inclusão em resina epóxi de presa fria (Buehler, Lake Bluff, IL, EUA), as superfícies seccionadas serão polidas sequencialmente utilizando lixas de granulações 600, 1200, 2000 e 2500 (Norton S.A.), e posteriormente pastas de diamante com tamanho e partículas de 3, 1 e 0,25µm (Metadi II; Buehler). Debris serão removidos com lavagem em ultrassom durante 5min após cada passo de polimento. As análises em MEV serão realizadas a partir de amostras preparadas de acordo com as especificações para o equipamento de análise. Na preparação para a análise, as superfícies das amostras serão cobertas com ouro-paládio numa Balzers

SCD 050Q, durante 3min, em uma corrente de 10mA e vácuo de 130mTorr. Imagens digitais representativas de cada amostra serão obtidas em aumento de 100 e 500µm (MEV−EDS; JSM-6610/LV; JEOL, Peabody, MA, EUA) (BOSCATO; DELLA BONA; CURY, 2007, VALENTINI et al., 2014).

2.4.4 Análise Estatística

Os dados quantitativos serão submetidos a análise estatística em nível de significância de 5%. A distribuição da resistência à flexão biaxial bem como a resistência de união de materiais friáveis é mais propriamente descrita pelo método estatístico de Weibull (WEIBULL, 1951) comparado a análise dos valores médios de resistência baseada na distribuição Gaussiana (ADDISON; FLEMING, 2008). Dessa forma, este estudo se propõe a analisar estatisticamente os dados de resistência com base na análise de Weibull, cuja forma básica de distribuição segue:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right]$$

em que σ_0 é a constante de parâmetro de escala e *m* o módulo de Weibull (que reflete a friabilidade do material) (ADDISON; FLEMING, 2008). O módulo de Weibull reflete a distribuição resultante e a confiabilidade dos dados de resistência à flexão (valores maiores indicam agrupamento mais próximo dos dados). O número de espécimes (n=30) utilizados no experimento para determinação do módulo de Weibull determina a confiança e precisão das análises (RITTER; BANDYOPADHYAY; JAKUS, 1981). Os valores de resistência à tração e resistência à flexão biaxial serão ainda analisados estatisticamente utilizando análise de variância de uma via, seguido pelo teste de Tukey, em caso de diferenças entre os grupos (α=0,05). Para avaliar a diferença no módulo de elasticidade do cimento obtido no teste de dureza de Knoop os dados serão submetidos a análise de variância de uma via seguido pelo teste de Newman Keuls.

2.5 Orçamento

Descrição	Quantidade	Custo (unidade)	Custo (total)
I14 A1C Vitablocs Mark II for Cerec; Vita Zahnfabrik, Germany	40 Un.	R$ 72,80	R$ 2.912,00
Silano Agente de União RelyX Ceramic Primer (3M ESPE, St. Paul, USA)	1 un	R$ 245,93	R$ 245,93
Relyx Veneer (3M ESPE, St. Paul, USA)	5 un	R$ 300,00	R$ 1.500,00
Single Bond (3M ESPE, St. Paul, USA)	1 un	R$ 110,00	R$ 110,00
Filtek Z350 – 3M ESPE, St. Paul, USA	12 un	R$ 155,00	R$ 1.860,00
Lixas SiC 400 (3un), 600(6un),1200(6un), 1500(3un), 2000(3un) e 2500(3un).	50 un	R$ 6,00	R$ 300,00
Condac Porcelana 10% (FGM, São Paulo, Brasil)	10 un	R$ 18,44	R$ 184,40
Microbrush (KG SORENSEN, São Paulo, Brasil	1 un	R$ 11,98	R$ 11,98
Disco diamantado Buehler	1 un	R$2500,00	R$2500,00
Super Bonder gel (Loctite Ltda, São Paulo, SP, Brasil)	10 un	R$5,50	R$55,50
TOTAL			**R$ 9.679,81**

2.6 Cronograma

Mês/Ano	Revisão de Literatura	Qualificação do Projeto de Dissertação	Preparo dos espécimes	Obtenção dos Dados	Redação	Apresentação da Dissertação e Submissão de artigos
Ago/2015	X					
Set/2015	X					
Outub/2015	X					
Nov/2015	X					
Dez/2015	X	X				
Jan/2016	X		X			
Fev/2016	X		X			
Mar/2016	X		X			
Abril/2016	X		X			
Maio/2016	X		X	X		
Jun/2016	X		X	X		
Jul/ 2016	X			X		
Ago/ 2016	X			X		
Set/ 2016	X			X		
Out/2016	X			X		
Nov/2016	X			X		
Dez/2016	X			X		
Jan/2017	X			X	X	
Fev/2017	X				X	
Mar/2017	X				X	X
Abril/2017	X				X	X
Maio/2017	X				X	X
Jun/2017	X					X
Jul/2017	X					X

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3 Relatório do trabalho de campo

A metodologia proposta inicialmente no projeto foi seguida, praticamente sem alterações. No entanto, o teste de resistência à flexão biaxial não foi realizado devido a atrasos ocorridos no envio de materiais importados adquiridos para o experimento. Esse teste tem grande relevância para finalização das avaliações referentes à influência da quantidade de carga inorgânica dos agentes de cimentação resinosos experimentais na resistência da cerâmica feldpspática.

Adicionalmente foi acrescentado ao projeto de pesquisa qualificado, o artigo 2, que avalia a influência do conteúdo de matriz inorgânica dos agentes de cimentação resinosos experimentais nas propriedades ópticas de simulados LCs. Tal avaliação foi realizada tendo em vista a escassez de evidência científica disponível sobre este assunto. Tal avaliação tem grande importância em virtude dos diversos cimentos resinosos disponíveis atualmente no mercado, os quais apresentam diferentes quantidades de carga e formulações.

Realizar estes estudos trouxe conhecimento e informações sobre a influência do conteúdo de carga inorgânica sobre os aspectos mecânicos e ópticos dos cimentos resinosos, o que fortaleceu meu conhecimento sobre este assunto. Esperamos que nossos resultados tenham grande relevância para a comunidade científica e para profissionais que atuam apenas na prática clínica.

4 Artigo 1[§]

The role of inorganic filler content of resin-based luting agent and use of adhesive underlying feldspar ceramic bonding

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The role of inorganic filler content of resin-based luting agent and use of adhesive underlying feldspar ceramic bonding

ABSTRACT

Objectives: To evaluate the influence of inorganic filler content of resin-based luting agents (RBLAs) and the adhesive use on the bond strength and interface morphology of luted feldspar ceramic.

Methods: RBLAs with low, intermediate and high inorganic filler content (55%, 65% and 75% of mass fraction, respectively) were prepared. The modulus of elasticity (*E*), Poisson's coefficient (*v*), viscosity and degree of conversion (*DC*) of these luting agents were measured. The RelyX Veneer, 3M ESPE was used as commercial reference. Feldspar ceramic blocks (Vitablocks Mark II; Vita Zahnfabrik) were luted to composite resin blocks, originating eight groups according to the different RBLAs and use of adhesive. The response variables (n=30) were the bond strength (μTBS, MPa), characteristic strength (σ_0, MPa), and Weibull modulus. Fractographic analysis and morphology of the bonded interfaces were analyzed. Data of *E*, *v, DC* and viscosity were analyzed using one-way analysis of variance followed by post-hoc Tukey's test (α= 0.05) and confidence intervals for the means (95% CI) were calculated for μTBS, σ_0, and *m*.

Results: The increase of their inorganic filler content was associated with increased *E* and viscosity of the experimental RBLAs, while *v* and *DC* were not influenced. The use of adhesive improved the μTBS and σ_0 for the commercial and RBLAs with high filler content.

Conclusion: The RBLAs with higher filler content presented lower bond strength when the adhesive was not used. The adhesive use should be considered, after

etching and silanizing ceramic surface, when resin-based luting agents with high viscosity are used.

Key words: Dental Cements, Adhesive, Acid-sensitive ceramics, Fractography, Mechanical strength, Weibull analysis.

1. INTRODUCTION

Feldspar ceramic is often used for laminate veneers because the excellent optical properties [1] attributed the its composition with high content of vitreous phase [2-4], and the bonding to tooth structure [5,6]. The clinical success and longevity of thinner feldspar ceramic restorations is linked to the luting procedures [7-9] and proper bond interface between luting agent and ceramic [7].

The quality of bond interface has been associated to the luting agent particle sizes [9], YoungTINmodulus of elasticity (*E*) [8,10] and inorganic filler content [11]. The influence of inorganic filler content on bond interface of luted feldspar ceramic has been poorly studied and doubt still remains [10]. The inorganic filler content changes the viscosity of the resin-based materials [12,13]. This suggests that the inorganic filler content could interfere in the penetration of the resin-based luting agents into the retention created by acid etching of the feldspar ceramic [14,15]. Unfilled grooves created by acid etching result in uneven surface that gives rise to fractures and cracks due to high concentration of stress at adhesive interface [16-18].

Studies have suggested the use of unfilled resin on the ceramic surface, previous to the application of the luting agent, to improve the filling of the irregularities created by acid etching [15,19]. However, it is believed that resin-based materials with *E* may decrease the strength of the feldspar ceramic. This issue remains controversial in the available literature.

Therefore, the aim of this study was to characterize the *E,* viscosity, Poisson's coefficient *(v)* and degree of conversion *(DC)* of experimental resin-based luting agents with different inorganic filler content (55%, 65% and 75% of mass fraction); and also to investigate the influence of these luting agent proprieties and the use of adhesive on the microtensile bond strength (μTBS) and bond interface morphology

with the feldspar ceramic. The hypotheses tested were that the inorganic filler content of the resin-based luting agents and adhesive use have influence on the μTBS and bond interface morphology with the feldspar ceramic.

2. MATERIALS AND METHODS

2.1 Study design

In this *in vitro* study, experimental resin-based luting agents were prepared with different inorganic filler content at three mass fractions: 55% wt. (low), 65% wt. (intermediate) and 75% wt. (high). A commercial reference (RelyX Veneer, 66%/wt., 3M ESPE, St Paul, MN, USA) was used as commercial group. The *E*, viscosity, *v*, *DC* of the resin-based luting agents were measured. Feldspar ceramic blocks were luted to composite resin blocks, originating a total of eight groups (n=30) according to the resin-based luting agent and adhesive use. The response variables were microtensile bond strength (μTBS, MPa), characteristic strength (σ_0, MPa), and Weibull modulus (*m*). Scanning electron microscopy (SEM) was used to evaluate the morphology of the bonded interfaces, and also the failure mode of the fractured surfaces based on fractographic analysis.

2.2. Formulation of experimental resin-based luting agents

The experimental resin-based luting agents were formulated combining 50% of mass fraction of the monomers urethane dimethacrylate (UDMA) and triethyleneglycol dimethacrylate (TEGDMA) (Esstech Inc., Essington, PA, USA). For the two luting agents, 0.4% mass fraction of camphorquinone (Sigma-Aldrich, St. Louis, MO, USA) was used as photosensitizer, and 0.8% mass fraction of ethyl 4-dimethylamino benzoate (Sigma-Aldrich, St. Louis, MO, USA) was used as

coinitiator. The monomers and mass fractions of the luting agents were tuned in pilot studies. Therefore, three different resin-based luting agents were created using different inorganic filler contents (% mass fraction), 55% wt. (low), 65% wt. (intermediate) and 75% wt. (high). Barium-borosilicate glass particles (2 μm average size) coated with 1% mass fraction of silane coupling agent (V-119-4120; Essington, PA, USA) were used as inorganic filler content. The materials were mechanically mixed using a centrifugal mixer (SpeedMixer DAC150; FlackTek, Landrum, SC, USA) at 1500 RPM during 20 s to produce homogeneous materials [10].

A light-cured resin-based luting agent (RelyX Veneer; 3M ESPE, St Paul, MN, USA) was used as commercial reference, widely used for the adhesive cementation of ceramic laminates veneer. This luting agent presents a 66% mass fraction of inorganic filler content according to manufacturer [20], which is similar to the intermediate experimental luting agent tested.

2.3. Determination of E, v, and DC of the resin-based luting agents

Three bars of each luting agent were made using a rectangular silicone mold (60 × 10 × 4 mm). Sonelastic equipment (ATCP Engenharia Física, Ribeirão Preto, SP) was used to obtain *E* and *v* (n=3) of the resin-based luting agents using the impulse excitation technique. The samples were positioned in the specific device and submitted to a short-time impact using a pulsator. The acoustic response was translated into an electric sign in order to read the resonance frequencies [21,22].

The *DC* of all luting agents (n=3) was evaluated with Fourier-transformed infrared spectroscopy (Prestige 21; Shimadzu, Tokyo, Japan) equipped with an attenuated total reflectance diamond device. A preliminary reading for the unpolymerized material (monomer) was taken under absorbance mode using 32

scans and 4 cm^{-1} resolution. The material was then light-cured with a light-emitting diode curing unit (Radii; SDI, Bayswater, Victoria, Australia) for 30 s at 1200 mW/cm^2 irradiance, then another spectrum was acquired. The *DC* (% per second) was calculated considering the intensity of the stretching vibration of aliphatic *DC* in the 1635 cm^{-1} region. The symmetrical drawing of the aromatic ring at 1716 cm^{-1} was used as an internal standard. The data obtained were plotted in a curve adjusted by the non-linear regression parameter of Hill to calculate the polymerization rate [23].

2.4 Viscosity of the resin-based luting agents

The rheological analysis was conducted using a dynamic oscillation rheometer (R/S Plus, Brookfield, Middleboro, USA) at 25 °C (room temperature mean), with a viscometric module of parallel plates (Ø 25 mm) of 0.05 mm distance with 0.5 ml quantity of material. The test was performed for 300 s at an initial shear rate of 4 s^{-1} and final at 100 s^{-1}. Thirty spot measurements were performed by the Peltier plate system. A shear sweep was used to measure the viscosity. The geometry used in this test was a parallel plate design of Peltier using 30 spot measurements [24].

*2.5. Microtensile Bond Strength (*μTBS*) and Failure Mode*

Feldspar ceramic blocks (Vitabloks Mark II A1C; Vita Zahnfabrik, Bad Säckingen, Germany) 12 mm × 14 mm × 7 mm were prepared and sequentially polished through 600 and 1200-grit SiC abrasive papers (Norton SA, Guarulhos, SP, Brasil) under running water. All blocks were ultrasonically cleaned in distilled water for 10 min and dried. Polished ceramic surfaces were treated with 10% hydrofluoric acid for 60 s (Condac Porcelain 10%; FGM, Joinville, SC, Brazil), washed for 60 s,

and dried with water- and oil-free compressed air for 30 s [25]. For cleaning, the specimens were etched with 37% phosphoric acid (Condac 37, FGM, Joinville, SC, Brazil) for 30 s, washed and dried as previously described. Two layers of silane coupling agent (RelyX Ceramic Primer; 3M ESPE, St Paul, MN, USA) were applied, and after 60 s, they were dried with compressed air for 30s. In four blocks, a thin layer of adhesive (Adapter Single Bond 2; 3M ESPE, St Paul, MN, USA) was applied randomly allocated in the groups that received it, which was air-dried and light-cured for 40 s using LED unit (Radii; SDI, Bayswater, Victoria, Australia) with irradiance of 1200 mW/cm^2 [26].

Resin composite blocks (Llis A2D; FGM, Joinville, SC, Brazil) with the same dimensions as the ceramic specimens were luted to the ceramic blocks using one of the resin-based luting agents. A 750 gF-luting load was applied for 2 min, and the luted blocks were light-cured for 40 s on all interfaces. Therefore, eight groups were created according to resin-based luting agent and adhesive use: C, commercial reference of resin-based luting agent without adhesive; Low, 55% mass fraction of inorganic filler content without adhesive; Intermediate, 65% mass fraction of inorganic filler content without adhesive; High, 75% mass fraction of inorganic filler content without adhesive; and other four groups (C-Ad, Low-Ad, Intermediate-Ad and High-Ad) using adhesive previous to the resin-based luting agents.

After 24 h, each bonded specimen was sectioned into ~30 stick-shaped specimens (0.8 mm^2 of bonded area) using a diamond saw under water-cooling (Isomet1000; Buehler, Lake Bluff, IL, USA), [10,25]. The sticks were submitted to μTBS test on a mechanical testing machine (DL500; EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min until failure [27]. The mean μTBS values were calculated in MPa.

Fractured specimens were examined under 40 × optical magnification and failure modes were classified as: premature debonding (specimens debonded spontaneously during sectioning or testing); adhesive (interfacial failure involving only adhesive); mixed failure (ceramic interface, bonding agent, and resin-based luting agent); or cohesive failure (failures within ceramic or resin composite) [9,28]. Descriptive statistics was used to report the failure mode data.

2.6. Statistical analysis

Data of *E*, *v, DC* and viscosity were analyzed using one-way analysis of variance followed by post-hoc Tukey's test (α= 0.05). Confidence intervals for the means (95% CI) were calculated for microtensile bond strength (μTBS, MPa), characteristic strength (σ_0, MPa), and Weibull modulus (*m*). Groups were considered significantly different when the 95% CI bounds did not overlap.

2.7. SEM analysis of failure mode

For the evaluation of the failure mode, fractured specimens (n=5) were examined after μTBS test using *SEM* to determine the failure mode based on the fracture origin and fractrographical principles [29-31]. The specimens were ultrasonically cleaned with distilled water for 30 min, dried at 37°C, coated with gold, and examined using scanning electron microscope (SEM) (SSX-550; Shimadzu, Tokyo, Japan).

2.8. SEM analyses: luting agent surfaces and bonded interface

Ceramic-luting agent-ceramic sandwiched specimens were obtained for the eight groups tested (n=3) to observe the morphology [9]. The specimens were

embedded cross-sectionally in epoxy resin for the ceramic-luting agent interfaces to be viewed. The specimens were ultrasonically cleaned with distilled water for 30 min, dried at 37 °C and polished using 600, 1200, 2000 and 2500-grit SiC abrasive papers followed by diamond suspensions of 3 and 1 μm. After they were cleaned, dried, sputter-coated with gold-palladium, and examined using SEM (SSX-550, Shimadzu, Tokyo, Japan). These specimens were also used to evaluate the film thickness obtained in the different groups using SEM. The film thickness (adhesion zone) was defined as the region in which the luting agents interact with the etched ceramic surface, coated with silane and adhesive. Four discs (6 mm × 2 mm) of all luting agents were prepared, following the same methodology described previously, for surface topography investigation [9].

3. RESULTS

3.1. E, v, and DC of the resin-based luting agents

The resin-based luting agents characterization values (*E*, *v*, and *DC*) are shown in Table 1. Concerning *E*, the commercial and high filler content luting agents presented the highest values and low filler content the lowest values with significant statistical difference between them ($p = 0.004$); while the luting agent with intermediate filler content was similar to the high filler content. For the *v*, there was no statistical significant difference among the groups ($p = 0.416$). The commercial luting agent had lower conversion rates (%) than other experimental luting agents ($p < 0.001$).

3.2. Viscosity of the resin-based luting agents

The viscosity analysis of the resin-based luting agents is shown in Figure 1 and Table 1. The luting agent with high inorganic filler content presented the highest viscosity (78.4), and at 220 s it reached 0 Pa-s with the applied pressures; while the luting agent with low inorganic filler content presented the lowest viscosity (2.40) values with significant statistical difference between them ($p = 0.013$); while the intermediate filler content showed similar viscosity values than the commercial luting agent.

*3.3. Microtensile Bond Strength (*μTBS*) and Failure Mode*

The μTBS, σ_0, m, and Weibull and failure mode are summarized in Table 2 and Figure 2. The μTBS and σ_0 were higher for C-Ad group followed by Low-Ad group, and lower for High group ($p < 0.001$). The use of the adhesive increased significantly the μTBS and σ_0 for High-Ad and C-Ad groups ($p < 0.001$) (Table 2). However, the use of adhesive had no influence on μTBS and σ_0 values for low and intermediate resin-based luting agents. The m presented significant difference between the Low-Ad and High-Ad groups, without crossing other groups.

Figure 3 shows the most frequent failure modes for all groups. Adhesive failures were predominant found for Low-Ad (57%), Low (70%) and High (51%) groups. Whereas, mixed failures were predominantly found in the Intermediate (60%), Intermediate-Ad (64%), High-Ad (58%) and C-Ad (64%) groups. Commercial group without adhesive (C) presented similar mixed (41%) and adhesive (41%) failures.

3.4. SEM analyses: luting agent surfaces and bonded interface

Representative *SEM* images of resin-based luting agent surfaces are shown in Figure 4. All specimens revealed increased agglomerates of particle fillers according to the proposed loading concentration, except the commercial luting agent, which presented greater surface homogeneity.

Figure 5 shows the morphology of the ceramic-luting agent bonded interfaces. The adhesive helped to fill irregularities in the grooves created by the acid etching. The analysis of the film thickness is presented in Figure 6. The High and High-Ad groups presented higher film thickness measurements at bonded interface, while the lower values of film thickness were found for the Low, Low-Ad and C groups. Overall, the use of adhesive increased the film thickness for all groups, except for Low-Ad group.

4. DISCUSSION

The clinical success of feldspar ceramic restorations depends on the quality and durability of the hybrid layer formed between ceramic and luting agent [7,29]. The composition of the resin-based luting agents, and consequently their mechanical properties, has influence on the bonded interface [7], including the content of methacrylate monomers, initiators, shape, composition and content of inorganic filler particles [9,10,32]. When commercial materials are tested, the shortcoming is that their formulation is not thoroughly known and this information is not fully available for researchers [10]. In this study, the effect of the inorganic filler content on bonded interface using experimental resin-based luting agents with same organic matrix and initiators was evaluated avoiding the influence of these compounds [10,11,32].

Concerning the characterization of the luting agent proprieties, overall the increase of the inorganic filler content increased the *E* of the resin-based luting agents. The commercial (10.7 GPa) and higher inorganic filler content (10.4 GPa) resin-based luting agents showed higher *E* followed by intermediate filler content (7.8 GPa), and the lowest *E* for the low filler content (6.6 GPa). These findings are in agreement with previous studies presenting a positive correlation between filler content and mechanical properties [33-36]. Therefore, the mechanical performance of resin composites can be improved by increasing their filler content, assuming that integrity is maintained between the resin matrix and filler particles mediated through silane coupling agent [33]. However, it has been questioned that highly filled resin-based luting agents have higher viscosity and may differ from less viscous materials regarding their potential to penetrate the etched ceramic surface and limiting the film thickness of the luting agent [10,32]. The current study showed that the resin-based luting agent with high filler content presented the highest viscosity, followed by commercial and intermediate filler content, and the lowest for the low filler content. These results are in agreement with previous studies reporting that the filler content alter the wettability of the resin-based luting agents due to increase in their viscosity [11,37].

The *DC* is important due to a higher conversion rate of monomers in polymers that provides improved physical-mechanical properties of the resin-based luting agents [38]. The *DC* presented no statistical difference between the experimental resin-based luting agents prepared, which presented a *DC* above 50% corroborating the findings from the literature for the light-curing resin-based luting agent [38-40]. In addition, the *DC* of the experimental resin-based luting agents was higher than the commercial luting agent.

The inorganic filler content and adhesive use influenced the bond strength and morphology of the bonded interface between ceramic and resin-based luting agents, confirming the hypothesis of the study. When the different resin-based luting agents were used without adhesive previously, the results of the current study showed that the μTBS and σ_o values were the highest for Intermediate group, similar to C and Low groups, but it was higher compared to High group. These results can suggest that resin-based luting agent with higher viscosity have difficulty to penetrate the irregularities of the etched ceramic surface.

This hypothesis may be better observed comparing the groups using resin-based luting agent with high inorganic filler content with (High-Ad) and without adhesive (High), since the application of the adhesive on etched ceramic surface, previous to the resin-based luting agent with high filler content increased the μTBS and σ_o. Similar behavior was found comparing the C and C-Ad groups, where the use of the adhesive increased the μTBS and σ_o. In addition, the mode of failure was changed for the C and C-Ad, and High and High-Ad groups, and overall the use of adhesive increasing the number of mixed failures using these luting agents. For these groups using adhesive, the fractured surfaces revealed fracture events starting at the adhesive and reaching luting agent in the bonded interface (Figure 3). The highest viscosity values were found for the higher filler content followed by intermediate filler content and commercial luting agent; and with exception of the intermediate filler content, the other luting agents presented higher bond strength.

The difference concerning the use of adhesive between the intermediate filler content and commercial luting agent can be associated with the different filler type and organic matrix. The commercial resin-based luting agent (RelyX Veneer) presented higher μTBS compared to the luting agent with similar filler content and

viscosity, when the adhesive was used. Therefore, other factors than the inorganic filler content and viscosity might affect the bond strength when different materials are tested [10,41]. The great difficulty of further comparisons between products is that manufacturers provide few details of their products as the mass fraction of monomer and their proportions, their methods of loading and silanization, which can change their properties [42]. According to the manufacturer, this commercial luting agent is composed of BisGMA and TEGDMA monomers, zirconia and silica filler particles (average size of approximately 0.6 μm), and a patented dimethacrylate polymer that modifies the material rheology and provides a unique handling characteristic allowing the cement to flow easily under pressure [20]. The SEM analysis of the luting agent surface showed more spherical and lower particles than experimental luting agents (Figure 4). Therefore, despite the mass fraction be similar to the material with intermediate filler content, the volume of particles is higher for commercial luting agent. This difference may change the wettability of the luting agent, mainly when adhesive is used previously. In addition, the commercial luting agent had higher *E* compared to the luting agent with similar filler content. Previous studies have showed to improve strengthening for the feldspar ceramic using luting agents with higher elastic modulus [8,10,14,32].

The independent variables tested presented few influence on *m*, once a significant difference was found between the Low-Ad and High-Ad groups, without crossing other groups. Although the use of adhesive increased the μTBS and σ_o for the High-Ad group, the reliability was decreased compared to Low-Ad group. Moreover, the High-Ad group presented higher number of mixed failures compared to Low-Ad, suggesting that highly viscous resin-based luting agent may present higher difficulty to interact with the adhesive anteriorly applied. When compared the Low

and Low-Ad groups, these presented similar results for the bond strength and analysis of the bonded interface, suggesting that it is not necessary the use of adhesive on silanized and etched ceramic surface, when a resin-based luting agent with low viscosity was chosen. Thus, it should consider the adhesive use to fill the irregularities of the etched ceramic surface when resin-based luting agents with high viscosity are used.

The SEM analysis of the film thickness showed higher film thickness for the more viscous materials at bonded interface. Overall, the use of adhesive increased the film thickness for all groups, except for group Low-Ad showing thinner film thickness. Probably, this occurred due to the mixing of the adhesive and luting agent, decreasing more its viscosity. These findings are in agreement with a previous study [12] that suggested as ideal film thickness between 100 and 250 μm [43-45]. Then, reduced film thickness was observed for Low (77.3 μm) and Low-Ad (66 μm) and C (68 μm) even they filling the grooves.

The High group and mainly, the C produced unfilled spaces in the bonded interface and lower μTBS and σ_o, suggesting the necessity of adhesive use. However, the better penetration observed for the High group compared to the C group probably occurred because over time the luting agent filled with high inorganic filler content showed decreased viscosity (Figure 1) resulting in better interpenetrated than the commercial reference. Irregularities not properly filled and no continuous and homogeneous interface impairs the quality of adhesive interface and μTBS values [14,15]. The SEM analysis showed that the resin-based luting agent with high filler content without adhesive use (Figure 3G) produced reduced penetration into the grooves created by acid, consequently, the High group showed lower μTBS values than Intermediate and Low groups. Probably, the high inorganic content filler

produced no homogeneous organic-inorganic matrix [46], resulting in formation of clusters changing the dissipation of tensions yielding the fracture to occur in a modified form [47]. The experimental luting agents were not mixed by hand; nevertheless, the resin-based luting agent with high inorganic filler content had the handling difficulty even with the use of mixing device and being heated until maximum temperature in order not to occur monomer conversion (60ºC) [48]. The clinicians should avoid the use of resin composite heated without adhesive since luting agents with higher filler content positively influenced mechanical properties; however, yielded low μTBS and σ_o values when the adhesive was not used.

Since the simply selecting of an appropriate luting agent and the use of adhesive can increase structural reliability of luted feldspar ceramic restorations, further studies should be performed to improve the knowledge about the relationship between viscosity and *E* on the bond strength of aged interfacial bonding and ceramic strengthening.

5. CONCLUSION

Resin-based luting agents with higher inorganic filler content presented higher elastic modulus, viscosity and film thickness of the luting agent layer. However, the resin-based luting agent with the highest filler content presented lower bond strength when the adhesive is not used. In the clinical scenario, the clinicians should consider the adhesive use to fill the irregularities of the etched ceramic surface when resin-based luting agents with high viscosity are used.

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Table 1. Mean of modulus of elasticity (*E*), Poisson's coefficient *(v)*, degree of conversion *(DC)* and viscosity for resin-based luting agents tested (n=3).

Resin-based luting agents	***E* (GPa)**	***v***	***DC* (%)**	***Viscosity***
RelyX Venner (Commercial)	10.7 A	0.36 A	41.3 B	48.0 A
Low (55%)	6.6 C	0.43 A	52.7 A	2.4 AB
Intermediate (65%)	7.8 BC	0.44 A	52.6 A	33.9 AB
High (75%)	10.4 AB	0.61 A	51.7 A	78.4 B

Different capital letters in the column show significant differences ($p<0.001$).

Table 2 – Estimates (95% confidence intervals) for mean microtensile bond strength (μTBS), characteristic strength (σ_o), and Weibull modulus (*m*), n=30.

Groups	μTBS (MPa)	σ_o (MPa)	*M*	Failure mode (%)		
				Adhesive	Mixed	Cohesive
CA	33.9 (30.2 – 37.5) A	37.5 (33.8 – 41.7) A	3.6 (2.8 – 4.8) AB	33	64	3
C	24.2 (21.0 – 27.4) BC	27.0 (24.0 – 30.6) B	3.0 (2.3 – 4.0) AB	47	47	6
Low-Ad	23.1 (21.3 – 24.9) BC	25.0 (23.2 – 27.1) BC	4.9 (3.8 – 6.5) A	57	37	6
Low	25.1 (22.6 – 27.6) BC	27.6 (25.2 – 30.3) B	4.2 (3.1 – 5.6) AB	70	24	6
Intermediate-Ad	27.9 (25.2 – 30.5) B	30.7 (28.2 – 33.4) B	4.5 (3.4 – 6.0) AB	30	64	6
Intermediate	26.9 (24.2 – 29.6) B	29.7 (26.9 – 32.8) B	3.8 (2.9 – 5.0) AB	37	60	3
High-Ad	25.4 (22.0 – 28.8) B	28.3 (24.7 – 32.6) B	2.7 (2.1 – 3.5) B	36	58	6
High	20.0 (18.1 – 21.9) C	22.0 (20.8 – 23.9) C	4.6 (3.4 – 6.2) AB	51	40	9

Different letters in the same column indicate significant differences between the groups;

Luting agents: C, commercial group; Ad, adhesive used; Low, 55% mass fraction; Intermediate, 65% mass fraction; High, 75% mass fraction.

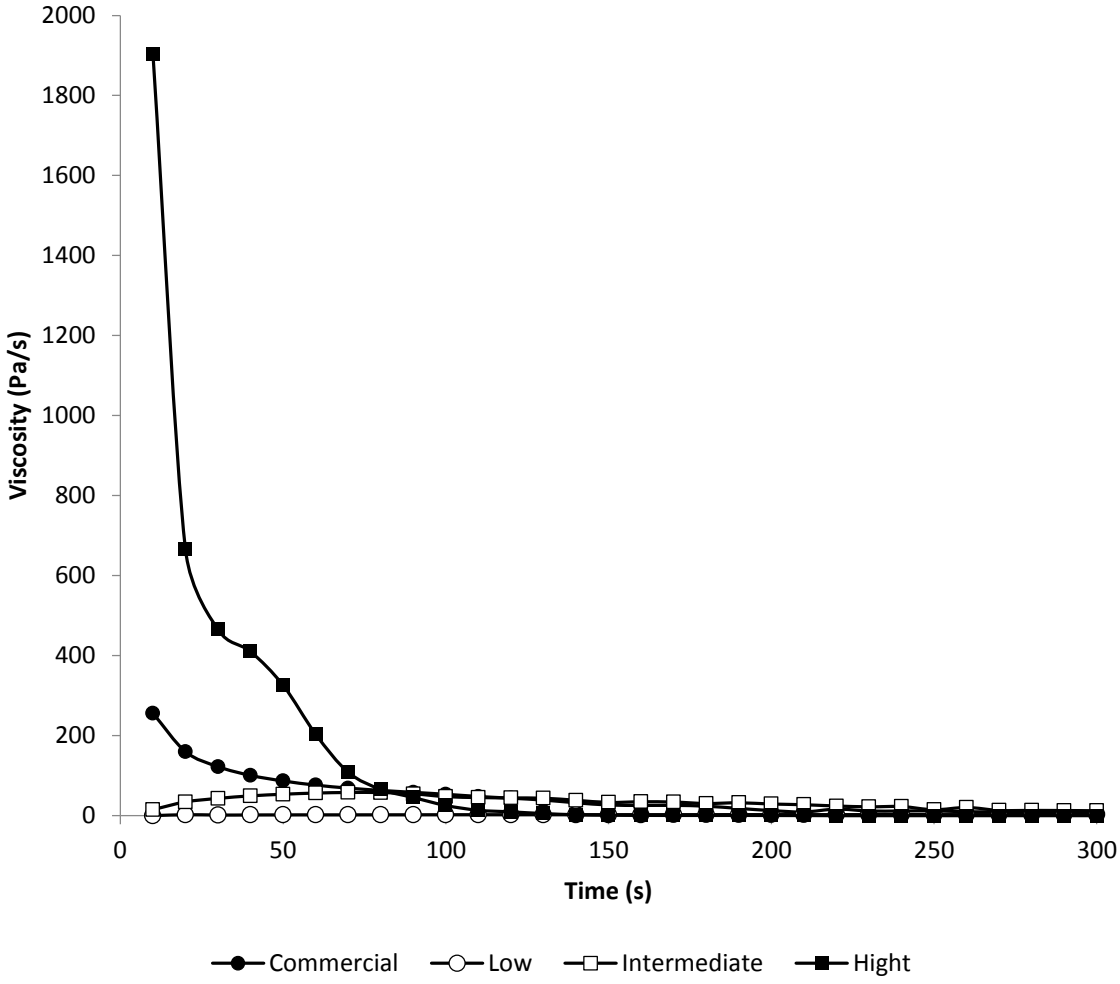

Figure 1. Complex viscosity of resin-based luting agents with different inorganic filler content (% mass fractions) and time.

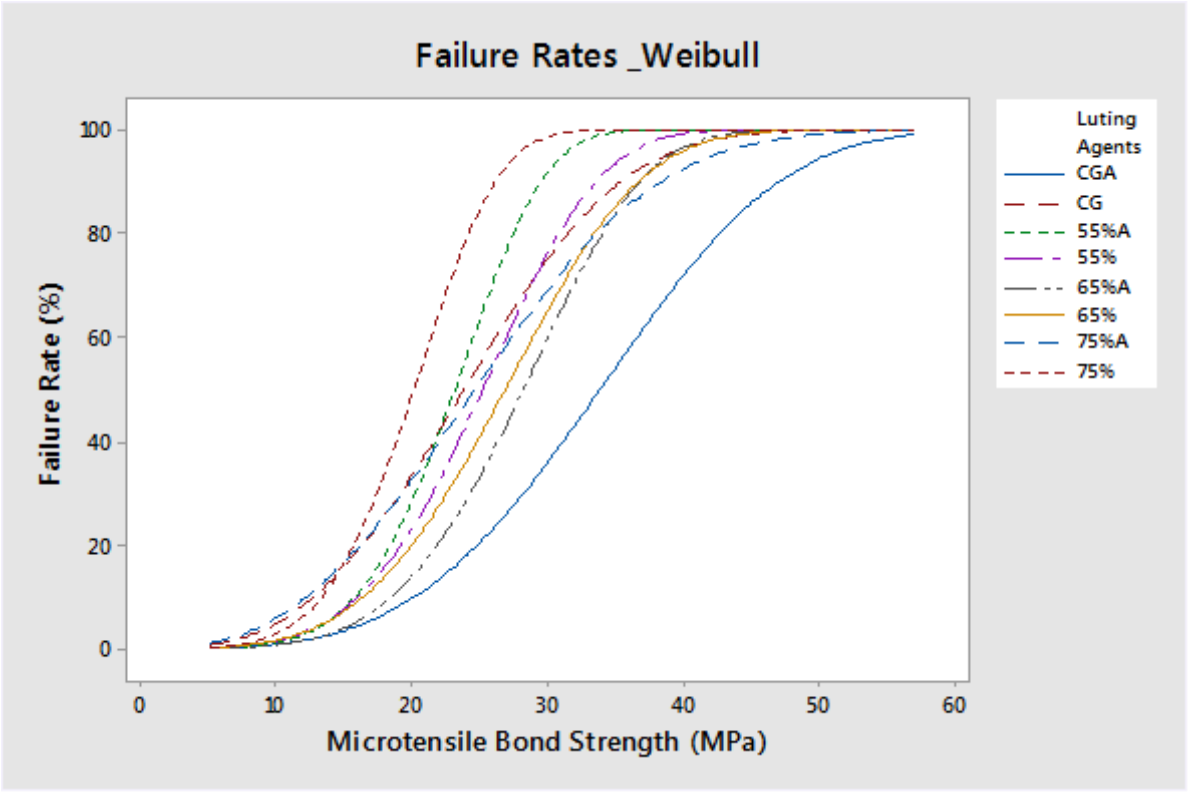

Figure 2. Weibull plot showing the probability of failure (%) *versus* bond strength (MPa) for all resin-based luting agents.

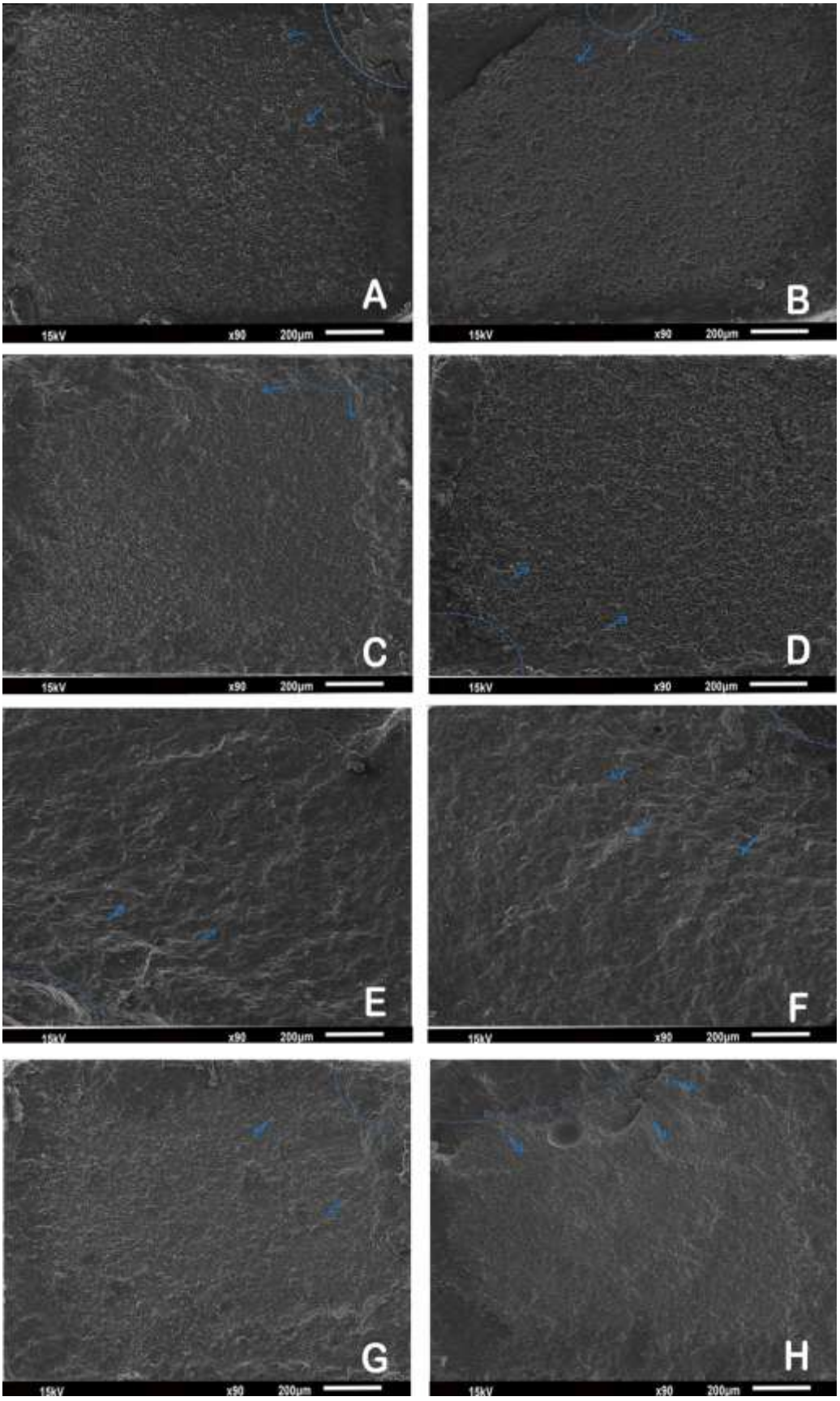

Figure 3. Representative *SEM* micrographs of fractured surfaces show modes of failure predominantly found observed at ×90. Dotted lines indicate critical flaws and

arrows indicate hackles. Fractured surface of the specimen from C (A), Low (C), Low-Ad (D) and High (G) groups presenting adhesive failure; and fractured surface of the specimen from C-Ad (B), Intermediate (E), Intermediate-Ad (F) and High-Ad (H) groups presenting mixed failure. In the mixed failures, fractured surfaces revealed fracture events starting in the adhesive and reaching luting agent in the bonded interface.

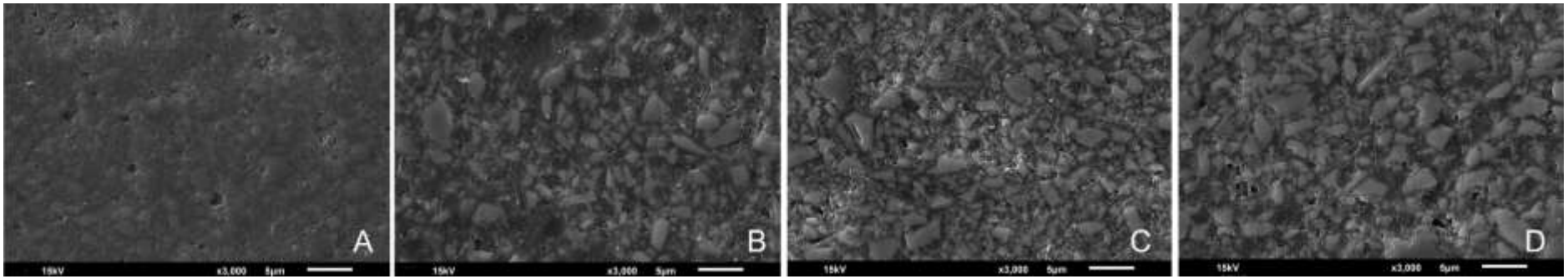

Figure 4. *SEM* images show information about the distribution of the inorganic fillers in matrix of the resin-based luting agents: (A) Luting agent "C" showed spherical fillers particles and homogeneous matrix; (B), (C) and (D) represent the experimental resin-based luting agents with low (55% mass fractions), intermediate (65% mass fractions) and high (75% mass fractions) inorganic filler content at ×3000, respectively.

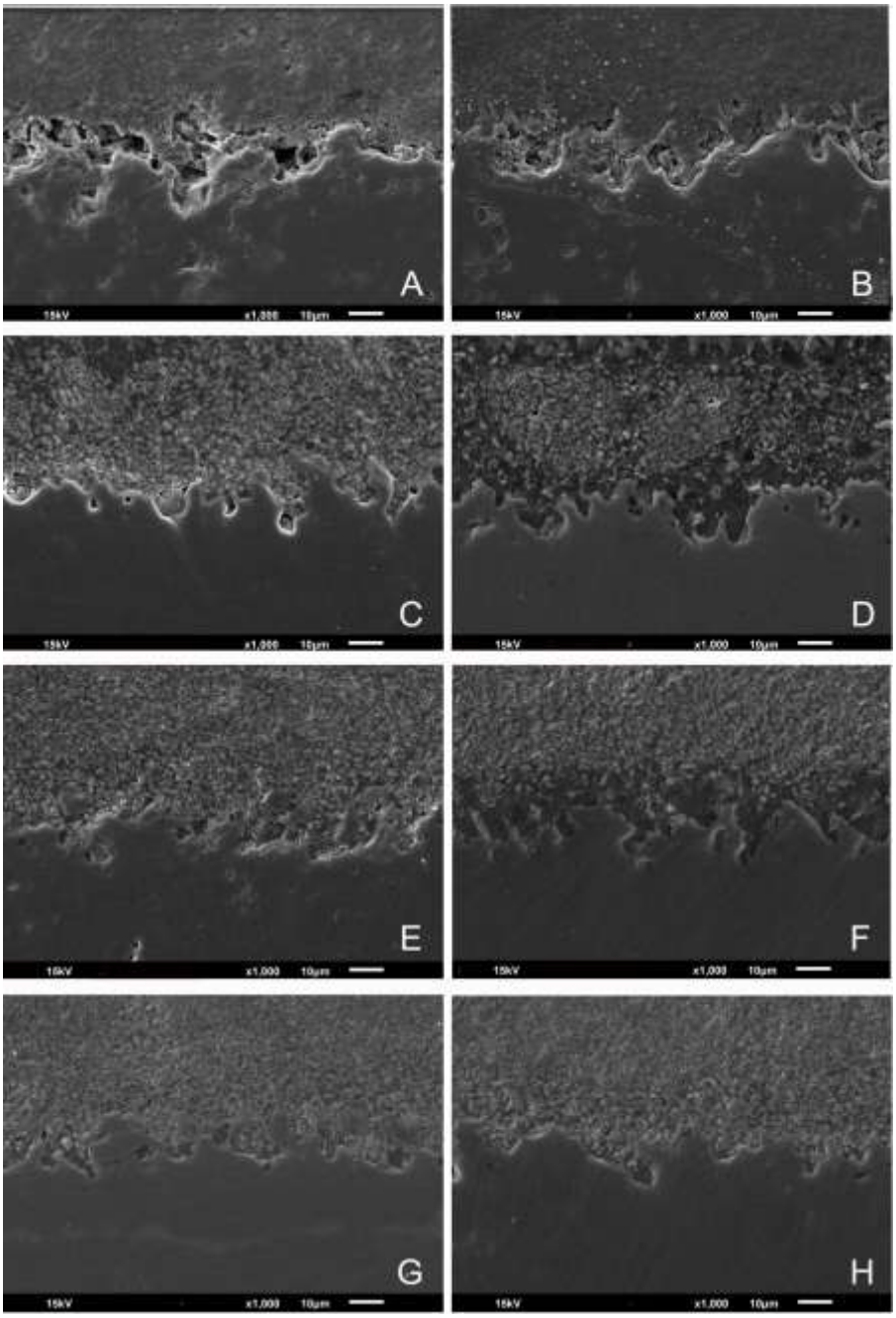

Figure 5. *SEM* images of the bonded interface of ceramic-luting agent sandwiched assemblies observed at ×1000: C (A); C-Ad (B); Low (C); Low-Ad (D); Intermediate (E); Intermediate-Ad (F); High (G); High-Ad (H).

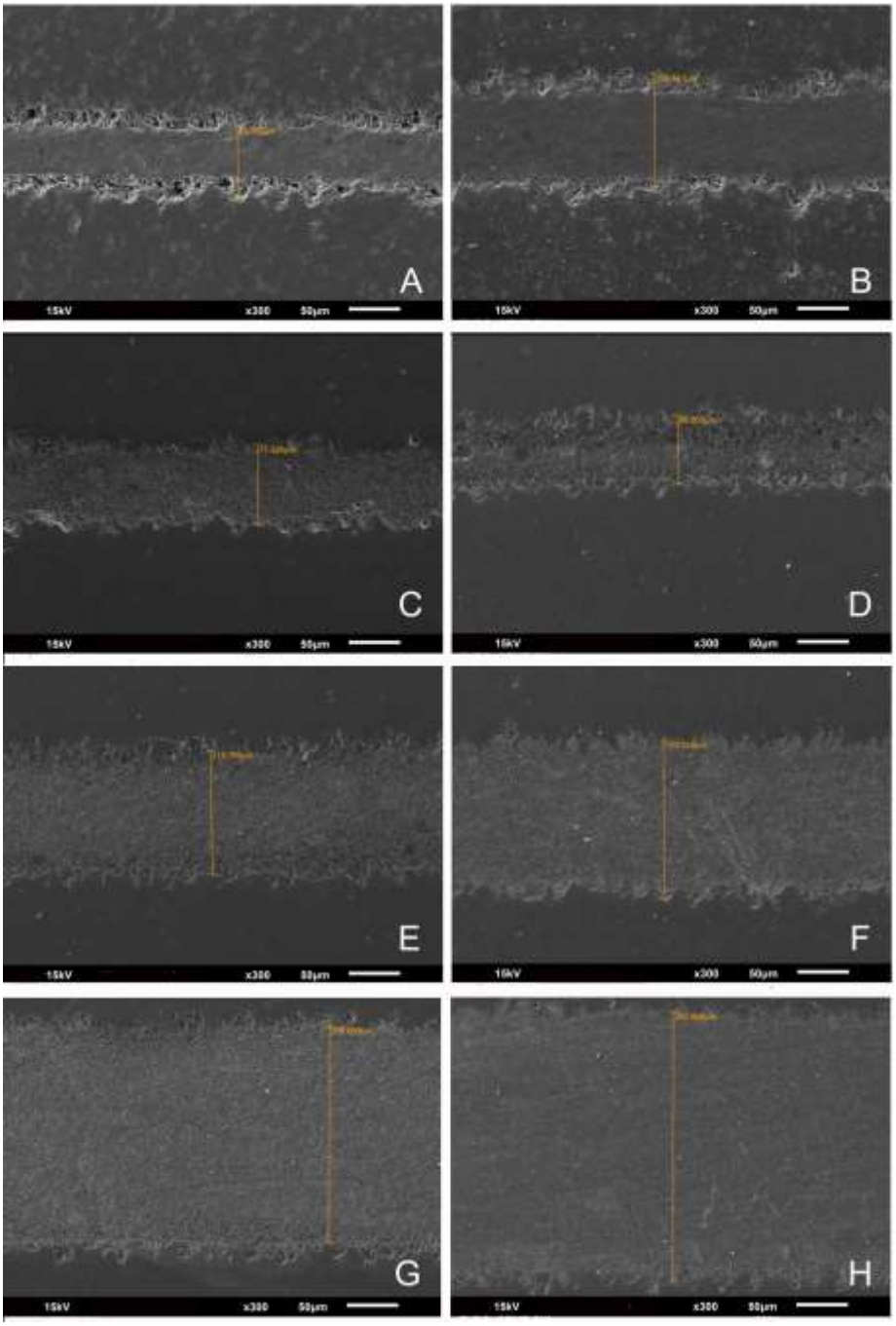

Figure 6. *SEM* images of the bonded interface showing film thickness originated, from resin-based luting agent using or not adhesive, observed at × 300 magnification: C (A); C-Ad (B); Low (C); Low-Ad (D); Intermediate (E); Intermediate-Ad (F);High (G); High-Ad(H)

5 Artigo 2[§]

Title: **Effect of inorganic filler content of resin-based luting agents on color of ceramic laminate veneers**

Running Title: **Effect of inorganic filler content of resin-based luting agents on color**

Keywords: Ceramics, Dental Cements, Color, Aesthetic, Translucency.

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Title: **Effect of inorganic filler content of resin-based luting agents on color of ceramic laminate veneers**

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SUMMARY

Objectives: To evaluate the influence of inorganic filler content of resin-based luting agents (RBLAs) on color change (ΔE00), CIEL*a*b* (individual color coordinates) and translucency parameters (TP) of simulated ceramic laminate veneer (CLV).

Methods: RBLAs with low, intermediate and high inorganic filler content (55%, 65% and 75% of mass fraction, respectively) were prepared. The ΔE_{00} was calculated by CIEDE2000 color difference metric for each feldspar ceramic (Vitablocks Mark II) specimens (1.2 mm × 0.8 mm, A1C shade) bonded to simulated composite resin substrate (1.6 mm × 1.2 mm, A2D shade) using three experimental and a commercial (RelyX Veneer) RBLA (translucent shade), under three conditions (before, immediately and 24h after luting). The TP was calculated using CIEL*a*b* color coordinates measured over white and black backgrounds. Surface morphology of RBLAs and composition of commercial also were analyzed. The one-way and two-way analyses of variance with a post-hoc Tukey test were used respectively to calculate TP and ΔE_{00}. For CIEL*a*b* coordinates, each pair of variable was compared using the Student t-test ($\alpha = 0.05$).

Results: Overall the RBLAs tested presented clinically visible ΔE_{00} values under the three conditions evaluated. For all RBLAs, higher ΔE_{00} values were observed between measurements obtained before and immediately after luting. Different inorganic filler content did not significantly increase the opacity of the set ceramic-luting agents-resin composite.

Conclusion: The variation of inorganic filler content did not influence significantly the TP of simulated CLV; although all experimental RBLAs tested yielded ΔE_{00} above perceptibility threshold. The L*, a*, and b* individual color coordinates were cementation dependent.

Clinical Relevance Statement: The use of luting agents with different inorganic filler content can influence final color of CLV. Nonetheless, it is unlike that the optical performance of a luting agent can be predicted based solely on the commercial available compositional information.

Keywords: Ceramics, Dental Cements, Color, Aesthetic, Translucency.

1. Introduction

Restorative procedures involving ceramic laminate veneer (CLV) are often used due to minimum removal of dental structure and adequate reestablishing of dental aesthetic and anatomic patterns.[1,2-5] Several all-ceramic systems are currently available for fabrication of CLV. Among these, the Vitablocs Mark II feldspar ceramic (Vita Zahnfabrik) is widely used due to its significant aesthetic properties attributed to high content of vitreous phase in its composition.[6,7] However, feldspar ceramic presents limited mechanical strength,[3] and its clinical success is based on proper quality of the interfacial bond between ceramic and dental substrate. The quality of this bonding depends adhesive procedures that are controlled in part by the ceramic surface treatment and the materials used for adhesive cementation,[8] including the resin-based luting agent.[8,9]

The influence of the shade[4,10-14] and color stability of light or dual-cured[14-17] resin-based luting agent on final color of thin ceramic restorations has been widely studied. Indeed, studies have suggested that the inorganic filler content of resin-based luting agents can influence its adequate penetration on the grooves produced by ceramic surface treatment, acting on quality and durability of the adhesive interface.[9,17,18] However, there is a lack of studies considering the role of the luting agent inorganic filler content on final color of CLV; while few studies have investigated the influence of the resin composite inorganic filler content on restoration brightness,[19] and shape of filler and particle size on color change of resin composite restorations.[20-23]

Since the color of thin CLV can be influenced by several factors, and achieving natural tooth-like restoration is still one of the greatest challenges for restorative dentistry[11,15,24-26] this study evaluated the influence of low, intermediate

and high inorganic filler content (55%, 65% and 75% of mass fraction, respectively) on the translucency parameters (TP), color change (ΔE_{00}) and CIEL*a*b* individual color coordinates of simulated CLV. The hypotheses tested were that the inorganic filler content would influence the ΔE_{00} and TP of simulated CLV.

2. Materials and Methods

2.1 Study design

This *in vitro* study evaluated the response variables ΔE_{00} and TP, based on CIEL*a*b* color coordinates measured with a spectrophotometer (Easyshade Advanced 4.0, Vita Zahnfabrik, Bad Säckingen, Germany), of experimental resin-based luting agents loaded with low (55%/wt.), intermediate (65%/wt.), high (75%/wt.) mass fraction (translucent shade), and a commercial reference (RelyX Veneer, translucent shade, 3M ESPE, St Paul, MN, USA) with 66% wt. mass fraction of inorganic filler content (n=10/group).

The feldspar ceramic CAD/CAM blocks (I14 A1 Vitablocs Mark II for Cerec; Vita Zahnfabrik, Bad Säckingen, Germany) were evaluated over a simulated dental substrate (A2D shade) and the ΔE_{00} was calculated by CIEDE2000 color difference metric[25] under three conditions: *(i)* (B × IL), before *versus* immediately after luting; *(ii)* (B × IW) before *versus* 24 h after luting and water immersion; and *(iii)* (IL × WI) immediately after luting *versus* 24 h in water immersion. The TP was calculated using CIEL*a*b* color coordinates measured over white and black backgrounds.

2.2. *Formulation of experimental resin-based luting agents*

The experimental resin-based luting agents were formulated combining 50% of mass fraction of the monomers urethane dimethacrylate (UDMA) and

triethyleneglycol dimethacrylate (TEGDMA) (Esstech Inc., Essington, PA, USA). For the luting agents, 0.4% mass fraction of camphorquinone (Sigma-Aldrich, St. Louis, MO, USA) was used as photosensitizer, and 0.8% mass fraction of ethyl 4-dimethylaminobenzoate (Sigma-Aldrich, St. Louis, MO, USA) was used as coinitiator. The monomers and mass fractions of the luting agents were tuned in pilot studies. Therefore, three different resin-based luting agents were prepared using different inorganic filler contents (% mass fraction), 55% wt. (low), 65% wt. (intermediate) and 75% wt. (high). Barium-borosilicate glass particles (2 μm average size) coated with 1% mass fraction of silane coupling agent (V-119-4120; Essington, PA, USA) were used as inorganic filler content. The materials were mechanically mixed using a centrifugal mixer (SpeedMixer DAC150; FlackTek, Landrum, SC, USA) at 1500 RPM during 20 s to produce homogeneous materials.[27]

A light-cured resin-based luting agent (RelyX Veneer; 3M ESPE, St Paul, MN, USA) was used as commercial reference, widely used for the adhesive cementation of CLV. This luting agent presents 66% wt. mass fraction of inorganic filler content according to the manufacturer,[28] which is similar to the intermediate experimental luting agent tested.

2.3. *Preparation of the feldspar ceramic specimens*

Feldspar ceramic CAD/CAM blocks (I14 A1 Vitablocs Mark II for Cerec, A1C shade; Vita Zahnfabrik, Bad Säckingen, Germany) were milled under water-cooling originating cylindrical shape (12 mm diameter x 18 mm thickness). The cylinders were cut into 0.8 mm thickness discs (n=10/group)[27] using a diamond saw under water-cooling (Isomet1000; Buehler, Lake Bluff, IL, USA), simulating monolayer restorations.[27,29] All discs had both sides sequentially polished using 600 and 1200-

grit SiC papers (Norton SA, Guarulhos, SP, Brasil) under running water. The final dimensions of each specimen were measured using a digital caliper with 0.001 mm accuracy (Mitutoyo, Tokyo, Japan).

2.4. *Preparation of simulated dental substrate*

Cylinders (12 mm diameter × 18 mm thickness) from dentin resin composite shaded A2 (Llis, FGM, Joinville, SC, Brazil) were fabricated incrementally using polydimethylsiloxane molds (Clonage; New DFL, Rio de Janeiro, RJ, Brazil). Each increment was light-cured using an LED unit (Radii; SDI Limited, Bayswater, Victoria, Australia) at 1200mW/cm^2 irradiance following the manufacturer's recommendations. The cylinders were cut into 1.6 mm thickness discs using a diamond saw under water-cooling (Isomet1000; Buehler, Lake Bluff, IL, USA) originating a total of 40 resin discs (n=10/group)[11] prepared to evaluate ΔE_{00} and TP. The top surface of specimens was polished with 600 and 1200-grit SiC abrasive papers under running water.

2.5. Luting procedures for cementation of ceramic discs to simulated dental substrate

The polished ceramic disc surfaces were etched with 10% hydrofluoric acid for 60 s (Condac Porcelain 10%; FGM, Joinville, SC, Brazil), washed for 60 s, and dried with water- and oil-free compressed air for 30 s.[29] For cleaning, the specimens were etched with 37% phosphoric acid (Condac Porcelain 10%, FGM, Joinville, SC, Brazil) for 30 s, washed and dried as previously described. Two layers of silane coupling agent (RelyX Ceramic Primer; 3M ESPE, St Paul, MN, USA) were applied,

and after 60 s, they were dried with compressed air for 30 s and a thin layer of adhesive Single Bond 2 (3M ESPE, St Paul, MN, USA) was applied[15,27] .

A resin-based luting agent was applied to the center of the disc, and two matrix strips were lightly pressed to extrude the luting agent and create a film thickness between 100 to 250 μm suggested as ideal film thickness.[30-32] The ceramic discs were luted to resin composite discs with the different resin-based luting agents tested. The resin composite disc was centrally orientated on a leveled loading platform, and its top ceramic surface was loaded with 750 gF load for 2 min.[15] Luting agent excess was removed using a microbrush, and light-cured for 40 s using LED unit (Radii; SDI, Bayswater, Victoria, Australia) with irradiance of 1200 mW/cm^2.[15] on all interfaces. Therefore, four groups were originated according to resin-based luting agent: C, commercial reference of resin-based luting agent; Low, 55% mass fraction of inorganic filler content; Intermediate, 65% mass fraction of inorganic filler content; High, 75% mass fraction of inorganic filler content. The specimens were dry-stored at 37°C for 24 h in lightproof containers.

The experimental luting agent with high inorganic filler content was heated, until maximum temperature in order not to occur monomer conversion (60ºC), for 30 min[15,33] prior to cementation, due to its high viscosity increasing the wetting in the ceramic surface.

*2.6. Evaluation of CIEL*a*b* color coordinates*

The ΔE_{00} was estimated by calculating the CIEDE2000 color variation between the feldspar ceramic bonded to simulated dental substrate discs (n=30) using the experimental resin-based luting agents tested, under three conditions*: (i)* (B × IL), before *versus* immediately after luting; *(ii)* (B × IW), before *versus* 24 h after

luting and water immersion; and *(iii)* (IL × WI), immediately after *versus* 24 h after luting and water immersion, according to the following equation:[26,34]

$$\Delta E_{00} = \left[(\Delta L'/K_L S_L)^2 + (\Delta C'/K_C S_C)^2 + (\Delta H'/K_H S_H)^2 + R_T(\Delta C'/K_C S_C)\left(\Delta H'/K_H S_H\right)\right]^{1/2}$$

where Δ*L′*, Δ*C′*, and Δ*H′* are the differences in lightness, chroma, and hue between two sets of color coordinates. R_T is the rotation function that accounts for the interaction between chroma and hue differences in the blue region. S_L, S_C, and S_H are the weighting functions used to adjust the total color difference for variation in perceived magnitude with variation in the location of the color coordinate difference between two color readings. K_L, K_C, and K_H are the correction terms for the experimental conditions. The perceptibility and acceptability thresholds were set at ΔE_{00}= 0.8 and ΔE_{00}= 1.8, respectively.[35] All measurements were made using glycerin as liquid coupling medium between the background and the specimens.[36]

2.7. Evaluation of TP

The TP of the ceramic-luting agent-resin composite set was estimated by the difference between color coordinates measured over a white background (*L*= 90.9, a*= 0.3, b*= 4.9*) and a black background (*L*= 0.5, a*= 14.6, b*= -21.5*) using the following equation:[26]

$$\mathrm{TP} = [(L*_W - L*_B)^2 + (a*_W - a*_B)^2 + (b*_W - b*_B)^2]^{1/2}$$

where subscript W and subscript B refer to the color coordinates measured on the white and black backgrounds. All measurements were also made using glycerin as liquid coupling medium.[36]

2.8. *Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) analyses*

Four discs (6 mm × 2 mm) were prepared (n=4) to evaluate the surface morphology of all luting agents and composition of the commercial.[37] The specimens were embedded in epoxy resin, ultrasonically cleaned with distilled water for 30 min, dried at 37 °C and polished using 600, 1200, 2000 and 2500-grit SiC abrasive papers followed by diamond suspensions of 3 and 1 µm. After they were cleaned, dried, sputter-coated with gold-palladium, and examined using SEM (SSX-550, Shimadzu, Tokyo, Japan). The analysis of the composition of the groups was performed. Mappings were generated using *EDS* at 20 Kv.32 Representative images and spectra were recorded.[38]

2.9. Statistical analysis

The Shapiro-Wilk test was used to assess the normality of data. One-way and two-way analyses of variances with a post-hoc Tukey test were used respectively to calculate TP and ΔE_{00} (conditions and groups). CIEL*a*b* individual color coordinates values were analyzed considering pair of variables for each resin-based luting agents under three conditions using the Student t-test for equal variance data (α= 0.05). Confidence intervals for the means (95% CI) were also calculated.

3. Results

3.1 ΔE_{00}

Table 1 shows the results of ΔE_{00} for comparisons between groups and conditions. All experimental resin-based luting agents yielded ΔE_{00} values above 0.8 under the three conditions, which is the perceptibility threshold color difference for CIEDE2000 method.[35] For all resin-based luting agents, significantly higher and lower ΔE_{00} values were observed in the B × IL and IL × WI conditions, respectively ($p < 0.001$). Statistically significant differences were found for groups comparisons ($p = 0.039$) in the three conditions; the C group showed significantly lower ΔE_{00} values in the IL × WI condition; while in the B × IL and B × IW conditions the High group yielded significantly higher ΔE_{00} values.

3.2 CIEL a* b* color coordinates*

Table 2 shows the results for the CIEL*a*b* color coordinates (L*, a*, and b*) measured for all groups under the three conditions. Significant differences in b* values were observed for all groups ($p < 0.001$) evaluated over white and black backgrounds; except for C group in the IL × WI condition, which presented the highest b* values before luting for all conditions. The L* was lower for CLV after luting; and the highest L* values were found after luting for C group (86.82) in the IL × WI condition. Regarding a* coordinate, statistical significant differences were observed for Low and Intermediate groups in the B × IL condition, and for Low, Intermediate and High in the IL × WI condition. Only positive a*, and b* values were found.

3.3 TP

Figure 1 shows that the inorganic filler content did not influence significantly the opacity of the experimental resin-based luting agents tested (ceramic-luting agent-resin composite); however, the C group showed significantly higher TP.

3.4 SEM and EDS analyses

Representative SEM images of resin-based luting agent surfaces are shown in Figure 2. Experimental luting agents revealed similar fillers distribution according to resin matrix available, and larger and irregular fillers particles. The commercial luting agent showed better homogenous distribution between organic and inorganic matrixes, and smaller and more spherical fillers particles. For the C group, the EDS analysis confirmed the presence of modified monomers (Figure 3) and the initial composition was C (K) 50.08%; O (K) 19.19%; Si (K) 1.91% and Ca (K) 0.93% in the #1 and #3 points. In the #2 point, which represent the inorganic particles, the initial composition was C (K) 5.2%; O (K) 36.04%; Na (K) 0.31%; Al (K) 0.09%; Si (K) 29.9%; Ti (K) 0.37%; Zr (K) 16,39% and Tc (K) 0.35%.

4. Discussion

This study evaluated the influence of the inorganic filler content on optical properties of simulated CLV. Nonetheless, the final esthetic appearance of ceramic restorations results from a complex association of factors, which are not restricting to those evaluated in this study. Nevertheless, some variables that could interfere on color of simulated ceramic restoration, such as the ambient light conditions,[39] ceramic composition, thickness[24] glazing[40] and shade,[39] luting agent shade[11,14] and thickness,[24,30-32,41] background shade,[11,14,24] and adhesive cementation

procedures[42,43] were controlled. Luting agent thickness around 0.1 mm, suggested as clinically ideal,[30-32] was used to reproduce typical clinical scenario avoiding overestimated results regarding its effect on the color changes.[44]

All experimental luting agents were prepared with TEGDMA and UDMA resinous monomeric matrix since previous studies reported higher color stability and translucency than Bis-GMA.[4,45] Materials with translucent shade were prepared and/or selected due to their highest translucency values and refraction index than opaque and colored shades;[45] thus the influence of inorganic filler content would be better evaluated. Finally, color measurements were made with a spectrophotometer for accuracy and reproducibly.[36]

In this study all luting agents tested yielded ΔE_{00} values greater than 0.8 in the B × IL and B × WI conditions which is the threshold for perceptibility of color differences according to previous publication,[35] confirming the first hypothesis of the study. These results are in agreement with previous studies reporting the influence of the commercial[13,16,42,46] and experimental luting agent[47] on optical properties of translucent ceramic restorations. Overall, amongst the groups tested, the High group showed more pronounced effect on the final color of the specimens in the B × IL and B × IW conditions. These results indicated that the mass fraction of filler content tested influenced the ΔE_{00} of the experimental luting agents. This result is in line with previous study reporting that the inorganic content of resin composite should be controlled for the best color reproduction of natural tooth color.[21] Nevertheless, Low and Intermediate groups yielded similar ΔE_{00} values in the three conditions; whereas the better shade matching (i.e. lower ΔE_{00} values) was obtained for specimens luted with commercial luting agent (ΔE_{00}= 0.85±0.27) in the IL × WI condition. Probably the lower ΔE_{00} values obtained for C group is due to the different refractive indexes of

commercial and experimental fillers tested in this study.[20,22] Therefore, it seemed that differences in ΔE_{00} values among material could be dominated by organic matrix and filler properties such as particle size and shape, in addition to filler content, since the light scattering and absorption between matrix influence the optical properties.[21,48] In fact, the size and distribution of filler particles into resin matrix seems to correlate directly with the results of this study; the experimental luting agents that presented larger filler particles dimension (average size of approximately 2 μm) with irregular shape and less homogeneous distribution in resin matrix showed higher ΔE_{00} values compared to the commercial reference in which the filler particles dimension were smaller (average size of approximately 0.6 μm), spherical and better distributed. Thus, it seems that the size, shape and volume fraction of fillers should be controlled for the best color reproduction considering the refractive indices of filler and resin matrix.[20,22]

Considering the conditions evaluated, the highest ΔE_{00} values were observed in the B × IL and B × IW for all groups. It probably occurred because in these conditions the specimens were evaluated before and after luting. These results are in agreement with previous studies reporting that the luting agent play an important role on final color of thin CLV.[11,16,42] Under B × IW condition the specimens were water immersed during 24 h and lowest ΔE_{00} values were observed as compared with that values obtained in the B × IL condition, in which the specimens were not water stored. An explanation for changes in ΔE_{00} values due to water exposure might be found in the materials' composition. It is known from literature that resin-based composition allows water to penetrate the matrix or filler-matrix interface producing color changes.[48,49] This result shows the hydration influence of the set, even during a relatively short period (24 h), similar to clinical situation with oral fluid hydration.[50]

Within the limitations of this study, this finding suggests that the clinicians do not need to wait for long time of hydration for better color matching of CLV with adjacent teeth. Nevertheless, statistical significant differences with lower ΔE_{00} values were observed for C group, in the IL × WI condition, where only the hydration of the ceramic-luting agent-resin composite set was evaluated immediately after luting *versus* 24 h in water immersion. From this finding, the amount of inorganic filler content (75%, 65%, 55% experimental; 66% commercial) by itself could not be directly associated with the degree of color change found after storage. This is in accordance with author who stated that color changes could also be related to mass fraction of resin matrix[48] (UDMA and TEGDMA for experimental; BisGMA and TEGDMA for commercial) and properties of fillers.[21]

Regarding CIEL*a*b* individual color coordinates measured after luting, decreased L* values were observed for Low group in the B × IL and B × IW conditions. Our findings are in line with a previous study that evaluated resin composites with different particle sizes. This study reported decreased L* values for low particle sizes,[20] while a* coordinate showed positive values indicating a tendency toward a reddish color at all conditions. Low group presented lower a* values after luting and Intermediate and High increased a* values. However, the low values (0.11-1.21) found in this coordinate are clinically irrelevant.

Lower b* values were found in the B × IL and B × IW conditions, indicating that after luting the experimental luting agents originated less tendency to yellow color on ceramic specimens. Nonetheless, in the IL × WI condition, increased b* values were noted, indicating yellowish color including for C group, but without statistical significant difference. For the coordinates a* and b* the data have some similarities and counterpoints with other study, that showed a tendency of reddish (a*

positive) and bluish (b* negative) tones after immersion in water.[20] Our findings found positive values in both coordinates yielding yellowish (b*) and reddish (a*) tones. It is important to note that the time points observed in these studies were not similar, which could originate different results.

This study hypothesized that the TP would be affected according to the inorganic filler content since it could produce higher opacity. Nonetheless, the amount of inorganic filler content did not yield statistically significant differences among the experimental luting agents tested; while the commercial reference showed significantly the highest TP values, rejecting this hypothesis (Figure 1). An explanation for TP values found among experimental luting agents is due to even the luting agent loaded with high inorganic filler content was properly homogenized using the mechanical mixing device (Figure 2). However, the C group presented better homogeneous organic and inorganic matrixes compared to experimental groups. It is known from literature that homogeneous matrix phase provided lower color changes[20,49] due to lower light reflection and dispersion coefficients, which could change the refractive index.[19,20,21,49] The higher the refractive index difference between inorganic fillers and matrix phase of resin composites, the greater the opacity of the materials, due to multiple reflection and refraction at the matrix filler interfaces.[21,51] In fact, the color perception is directly connected with scattering since the interface can modify the way in which the light is scattered by the particles. The interface between resin and fillers is one of the weakest points of the composite material, and materials with a higher mass fraction of organic filler may have modifications in the way the light is punctured by the particles,[48,52] which may lead to mutation in resin-based luting agents opacity. This statement may explain the highest TP values found for High among experimental (Low and Intermediate)

groups, and the highest TP values found for C group among all groups tested, according to surface morphology, Figure 2.

The great difficulty of comparisons between products is that manufacturers disclose few details about proportion of monomers and filler particles, nor describe type and concentration of silane, pigments, and opacifiers. The knowledge about material composition is important since not only the inorganic filler content, but also the shape of filler and particle size could influence the light transmittance characteristics.[22] Our results indicated that by itself the filler content no significantly affected the TP of the experimental luting agents tested. Thus, it is unlike that the optical performance of a commercial luting agent can be predicted based solely on the available compositional information.[19] According to the manufacturer, the commercial luting agent used in this study is composed of BisGMA and TEGDMA monomers, zirconia and silica filler particles (average size of approximately 0.6 μm), and a patented dimethacrylate polymer that modifies the material rheology and provides a unique handling characteristic allowing the luting agent to flow easily under pressure, providing uniform film, regardless of the inorganic filler content,[28] as shown by the EDS image (Figure 3). In fact, the RelyX Veneer composition differs from experimental luting agents tested in some aspects regarding filler properties, including the average of particle size of approximately 0.6 μm and 2 μm and spherical and irregular shape, respectively for commercial and experimental luting agents (Figure 2). This result corroborates previous study reporting that materials containing smaller and irregular-shaped fillers showed higher light transmittance and diffusion angle distribution with a sharper peak, as compared with those containing larger and spherical-shape fillers.[22,48] Because of this, the highest TP values found for RelyX Veneer as compared to experimental luting agents with similar amount of

filler (Intermediate) could be linked to the scattering coefficient of the particle diameter and the wavelength of the incident visible light.[21]

One of the strengths of this study was the accuracy and reliability of the shade records. Only one calibrated examiner performed all the readings, avoiding inter-examiner variability. However, our study is not free of limitations, this is an *in vitro study* with short-term aging period; long-term aging effect on color stability of CLV luted with resin-based luting agents are a complex phenomena. Indeed, these findings should be interpreted with caution since there are few studies addressing the influence of inorganic filler content on optical properties of thinner CLV; nonetheless, this confirms that our data could help to achieve success of aesthetic treatment. It is important point out that greater color variation is desirable in clinical scenario when a shade match between darker and lighter adjacent tooth substrates is required. In this situation a more opaque luting agent shade should be used to mask the underlying color providing appropriate making ability, and consequentially shade matching with adjacent teeth. Then, since the scattering and absorption properties might be influenced by the difference in refractive indices between filler and resin matrix, further studies are recommended to evaluate the optical properties of resin-based luting agents loaded with different inorganic filler content, shape of filler and particle size, as well as, the long-term effect of aging on color change of luted CLV.

5. Conclusion

The variation of inorganic filler content evaluated did not influence significantly the translucency parameters of simulated ceramic laminate veneer; although all experimental luting agents tested presented color change above

perceptibility threshold. Finally, the L*, a*, and b* individual color coordinates were cementation dependent.

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Table 1. Means and standard deviations (SD) for color change (ΔE_{00}) for group and conditions comparisons.

Luting agents	**Before *versus* immediately luting**	**Before *versus* 24h after luting**	**Immediately *versus* 24h after luting**
	Means (SD)	**Means (SD)**	**Means (SD)**
RelyX Veneer	3.33 (1.52) B, a	2.74 (1.20) B, b	0.85 (0.27) B, c
Low	3.40 (0.71) B, a	2.60 (1.14) B, b	1.42 (0.34) A, c
Intermediate	3.50 (1.27) B, a	2.59 (0.94) B, b	1.47 (0.76) A, c
High	4.19 (0.69) A, a	3.50 (0.99) A, b	1.25 (0.17) AB, c

Different capital letters in the same column and lower letters in the same line show significant differences $p \leq 0.05$, respectively for groups and conditions. ΔE_{00} values (conditions and groups) were compared using two-way analysis of variance and Tukey post-hoc test;

Luting agents: RelyX Veneer, commercial group; Low, 55% mass fraction; Intermediate, 65% mass fraction; High, 75% mass fraction.

Table 2. Mean, standard deviation (SD), and 95% confidence interval (CI) for individual CIE L*, a*, and b* color coordinates for groups measured under three conditions.

	N	L*	95% CI	*P*	a*	95% CI	*P*	b*	95% CI	*P*
Before *versus* immediately luting										
RelyX Veneer Before	10	86.05 (1.01)	85.33 – 86.78	0.440	0.96 (0.25)	0.78 – 1.15	0.119	22.22 (1.31)	21.28 – 23.16	< 0.001*
RelyX Veneer After	10	86.40 (0.93)	85.73 – 87.06		1.21 (0.40)	0.92 – 1.50		16.40 (2.71)	14.46 – 18.35	
Low Before	10	85.88 (0.60)	84.45 – 86.32	0.003*	0.97 (0.26)	0.78 – 1.16	0.023*	20.89 (1.58)	19.75 – 22.02	< 0.001*
Low After	10	84.77(0.85)	84.16 – 85.38		0.11 (0.38)	0.34 – 0.88		14.95 (0.81)	14.37 – 15.53	
Intermediate Before	10	85.94 (0.45)	85.62 – 86.27	0.438	0.90 (0.28)	0.70 – 1.10	< 0.001*	21.09 (2.12)	19.57 – 22.60	< .001*
Intermediate After	10	86.26 (1.19)	85.41 – 87.12		0.73 (0.23)	0.27 – 0.60		14.91 (1.29)	14.00 – 15.83	
High Before	10	85.76 (0.52)	85.39 – 86.14	0.542	0.71 (0.29)	0.51 – 0.92	0.078	21.96 (1.39)	20.97 – 22.96	< 0.001*
High After	10	85.61 (0.51)	85.25 – 86.00		0.46 (0.31)	0.24 – 0.69		14.37 (0.72)	13.86 – 14.88	

Before *versus* 24h after luting and water immersion										
RelyX Veneer Before	10	85.94 (0.45)	85.62– 86.27	0.955	0.90 (0.28)	0.70 – 1.10	0.715	21.08 (2.12)	19.57 – 22.60	< 0.001*
RelyX Veneer After	10	85.97 (1.44)	84.94 – 87.00		0.94 (0.25)	0.77 – 0.12		16.67 (0.77)	16.12 – 17.23	
Low Before	10	85.88 (0.60)	84.45 – 86.32	0.001*	0.97 (0.26)	0.78 – 1.16	0.847	20.89 (1.58)	19.75 – 22.01	< 0.001*
Low After	10	84.04 (0.47)	82.98 – 85.10		0.94 (0.13)	0.64 – 1.23		16.80 (0.97)	16.10 – 17.50	
Intermediate Before	10	85.94 (0.45)	85.62 – 86.27	0.955	0.90 (0.28)	0.70 – 1.10	0.715	21.09 (2.12)	19.57 – 22.60	< 0.001*
Intermediate After	10	85.97 (1.43)	85.00 – 87.00		0.94 (0.24)	0.77 – 1.11		16.68 (0.77)	16.12 – 17.23	
High Before	10	85.76 (0.53)	85.39 – 86.14	0.045	0.71(0.29)	0.51 – 0.92	0.163	21.96 (1.39)	20.97 – 22.96	< 0.001*
High After	10	86.50 (0.94)	85.83– 87.17		0.91(0.31)	0.68 – 1.13		15.71 (1.01)	15.00 – 16.44	
Immediately after *versus* 24h after luting and water immersion										
RelyX Veneer Immediately	10	86.40 (0.96)	85.73– 87.06	0.275	1.21 (0.40)	0.92 – 1.50	0.813	16.40 (2.71)	14.46 – 18.35	0.339
RelyX Veneer After	10	86.82 (0.76)	86.28– 87.37		1.26 (0.47)	0.92 – 1.60		17.52 (2.34)	15.84– 19.20	

Low Immediately	10	84.77 (0.85)	84.16 – 85.38	0.199	0.61 (0.38)	0.34 – 0.88	0.007*	14.95 (0.81)	14.37 – 15.53	< 0.001*
Low After	10	84.05 (1.48)	82.98 – 85.11		0.13 (0.41)	0.65 – 1.23		16.80 (0.97)	16.10 – 17.50	
Intermediate Immediately	10	86.26 (1.20)	85.41 – 87.12	0.626	0.44 (0.23)	0.27 – 0.60	< 0.001*	14.91 (1.29)	14.00 – 15.83	< 0 .001*
Intermediate After	10	85.97 (1.44)	84.94 – 87.00		0.94 (0.24)	0.77 – 1.12		16.68 (0.77)	16.12 – 17.23	
High Immediately	10	85.62 (0.51)	85.25 – 85.99	0.018*	0.46 (0.31)	0.24 – 0.69	0.005*	14.37 (0.72)	13.86 – 14.88	0.003*
High After	10	86.50 (0.94)	85.83– 87.17		0.91 (0.31)	0.68 – 1.13		15.71 (1.01)	15.00 – 16.44	

Data in columns and lines not interrelated. *Statistically significant difference $p \leq 0.05$; CIEL*a*b* individual color coordinates were compared for each pair of variables by using Student *t* test;

Luting agents: RelyX Veneer, commercial group; Low, 55% mass fraction; Intermediate, 65% mass fraction; High, 75% mass fraction.

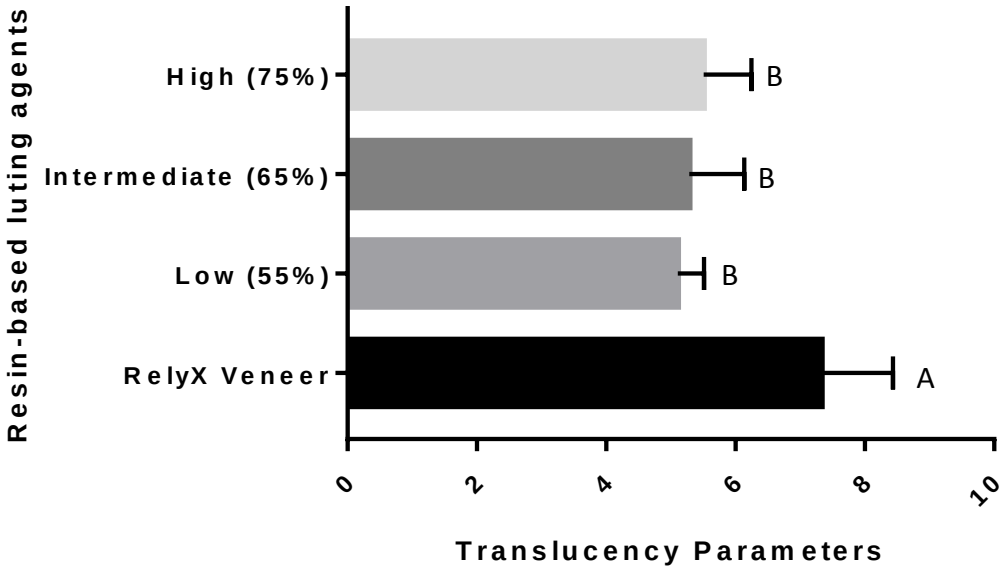

Figure 1. Resin-based luting agents comparisons using one-way analysis of variance with a Tukey post-hoc test for translucency parameter (mean and standard deviation).

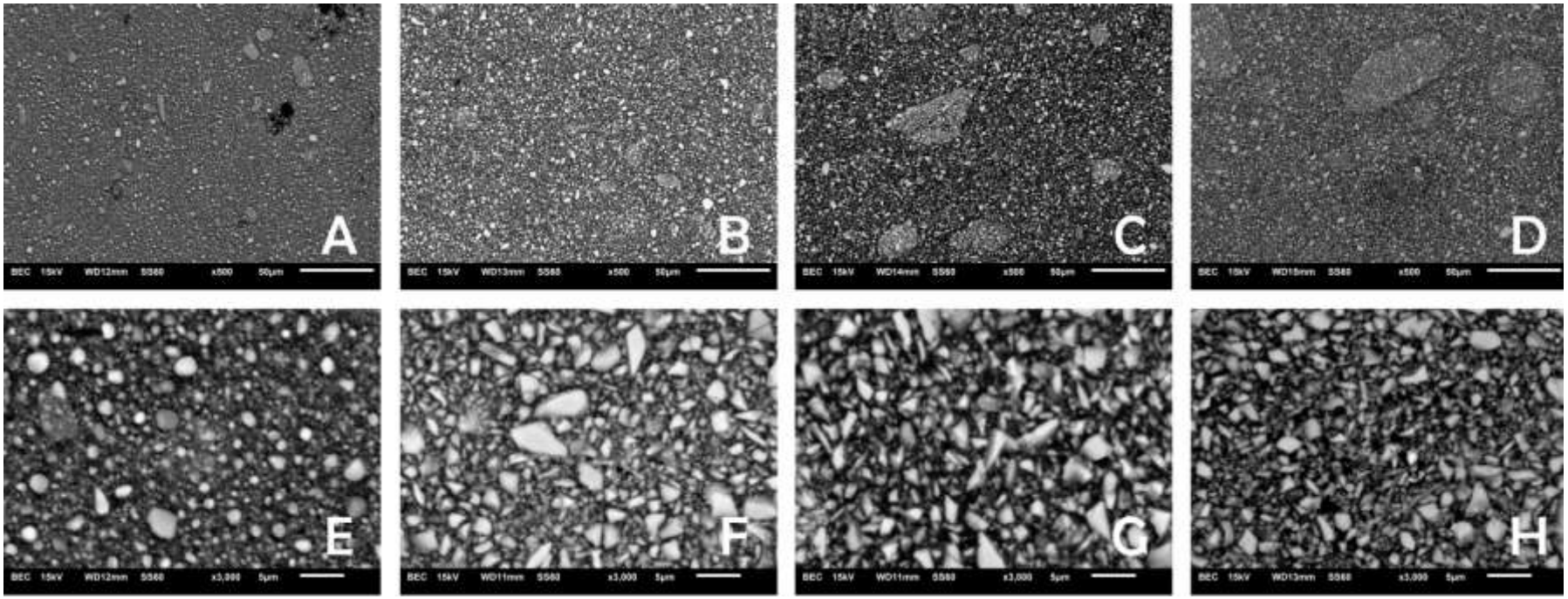

Figure 2. *SEM* images show information about the distribution of the inorganic fillers in the organic matrix of the resin-based luting agents observed at × 500 (A to D) and × 3000 (E to H). The A and E images, show the commercial luting agent with more homogeneous organic and inorganic matrix distribution and spherical filler particles; the B and F (Low), C and G (Intermediate) and D and H (High) images show the experimental resin-based luting agents.

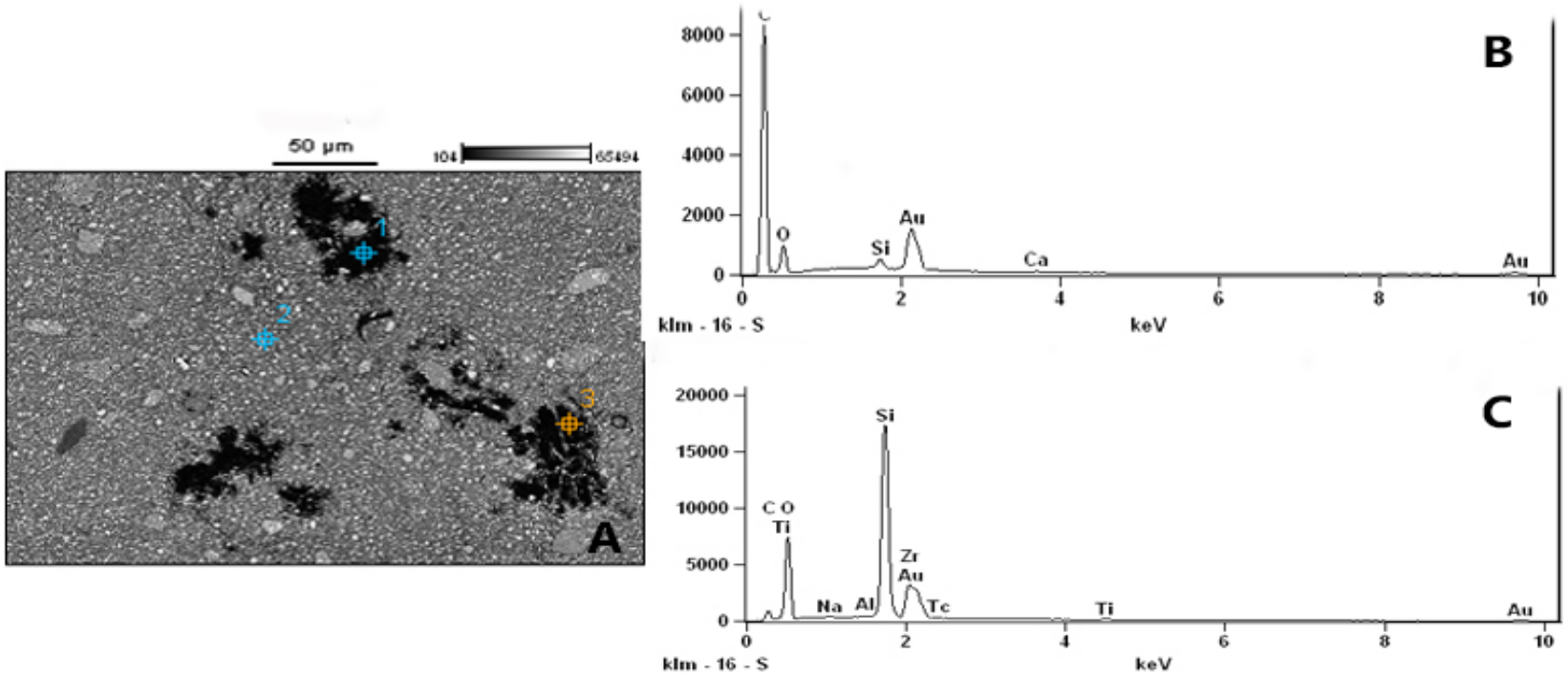

Figure 3. (A) Representative *SEM* image of commercial luting agent showing organic (#1 and #3 points) and inorganic (#2 point) matrixes. Graphics of the organic (B) and inorganic (C) matrix composition.

6 Considerações Finais

Este estudo avaliou a influência do conteúdo de matriz inorgânica de agentes de cimentação resinosos sobre as propriedades mecânicas e ópticas de simulados laminados cerâmicos. O Artigo 1 mostrou que o aumento do conteúdo inorgânico foi associado com aumento da viscosidade dos agentes de cimentação experimentais, enquanto a viscosidade e o grau de conversão não foram influenciados. O uso do adesivo melhorou a resistência à microtração e resistência característica do comercial e agente de cimentação experimental com alto conteúdo de carga.

O artigo 2 mostrou que a variação da quantidade do conteúdo inorgânico dos agentes de cimentação experimentais não influenciou significativamente o parâmetro de translucidez dos simulados laminados cerâmicos; embora todos os agentes de cimentação experimentais testados tenham produzido alteração de cor acima dos limites de perceptibilidade. As coordenadas de cor individuais L*,a* e b* foram dependentes da cimentação.

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Apêndices

Apêndice A – Nota da Dissertação

Influência do conteúdo de carga inorgânica nas propriedades ópticas e mecânicas de agentes de cimentação resinosos

A presente dissertação de mestrado foi dividida em dois artigos originados a partir de dois estudos *in vitro.* Tais estudos permitiram observar que a quantidade de matriz inorgânica inserida nos agentes de cimentação resinosos experimentais tem influência nas propriedades mecânicas, bem como na viscosidade e espessura da película de cimentação. Adicionalmente foi observado que o uso do adesivo é imprescindível quando cimentos resinosos com maior conteúdo de carga inorgânica são usados. Finalmente foi notado que a variação do conteúdo inorgânico não influenciou significativamente o PT dos LCs; embora todos os agentes experimentais de cimentação testados tenham produzido ΔE_{00} acima dos limites de perceptibilidade. As coordenadas de cor individuais L*,a* e b* foram dependentes da cimentação.

Campo da pesquisa: Clínica Odontológica, Odontologia Estética, Prótese Dentária, Materiais Odontológicos.

Candidato: Fabíola Jardim Barbon, Cirurgiã-dentista pela Faculdade Meridional IMED (2014)

Data da defesa e horário: 27 de Julho de 2017, às 9:00 horas

Local: Auditório do Programa de Pós-graduação em Odontologia da Universidade Federal de Pelotas. 5º andar da Faculdade de Odontologia de Pelotas. Rua Gonçalves Chaves, 457.

Membros da banca: Prof. Dr. Ataís Bacchi, Prof. Dr. César Dalmolin Bergoli e Profª. Drª Giana da Silveira Lima (suplente)

Orientadora: Profª. Drª. Noéli Boscato

Co-Orientador: Prof. Dr. Aloísio Oro Spazzin

Informação de contato: Fabíola Jardim Barbon – email: fabi_barbon@hotmail.com

Apêndice B – Súmula do currículo do candidato

Súmula do currículo

Fabíola Jardim Barbon nasceu em 20 de janeiro de 1993, em Passo Fundo, Rio Grande do Sul. Completou o ensino fundamental na Escola Estadual de Ensino Fundamental Monte Castelo e médio na escola privada Garra em Passo Fundo. No ano de 2010 ingressou na Faculdade de Odontologia da Faculdade Meridional IMED, tendo sido graduada cirurgiã-dentista em 2014. Durante o período de graduação foi bolsista FAPERGS/PROBIT desde o terceiro semestre da faculdade até o último. Trabalhou durante três anos na área epidemiológica com a Prof.ª Dr.ª Paula Wietholter, e durante dois anos e meio na área de materiais dentários com o Prof. Dr. Aloísio Oro Spazzin. Graduou-se como aluna destaque da turma no ano de 2014, sendo contemplada com uma bolsa de Pós Graduação Lato Sensu em Dentística, curso que concluiu no ano de 2016. No ano de 2015 ingressou no Mestrado do Programa de Pós-graduação em Odontologia da Universidade Federal de Pelotas (UFPel), área de concentração Prótese Dentária, sob orientação do Profª. Drª. Noéli Boscato e co-orientação do Prof. Dr. Aloísio Oro Spazzin. Durante o período de mestrado foi bolsista da Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) e desenvolveu trabalhos nas áreas de materiais dentários, revisões sistemáticas, ensaios clínicos e pesquisas *in vitro*. Em fevereiro de 2017, foi aprovada no doutorado em Clínica Odontológica com ênfase em Prótese Dentária na Universidade Federal de Pelotas (UFPel).

Publicações:

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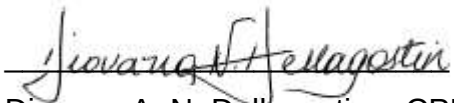
Anexos

Anexo A – Certificado da revisão de inglês do Artigo 1

ATESTADO

Atesto para os devidos fins que o artigo intitulado "**The role of inorganic filler content of resin-based luting agent and use of adhesive underlying feldspar ceramic bonding",** dos autores Fabíola Jardim Barbon, Rafael Ratto de Moraes, Aloisio Oro Spazzin e Noéli Boscato passou por revisão de inglês feita por mim com relação a seu conteúdo gramatical.

Pelotas, 26 de junho de 2017.



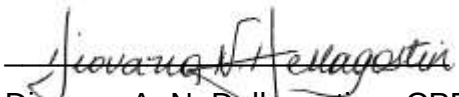
Diovana A. N. Dellagostin – CPE

Anexo B – Certificado da revisão de inglês do Artigo 2

ATESTADO

Atesto para os devidos fins que o artigo intitulado “**Effect of inorganic filler content of resin-based luting agent on color of ceramic laminate veneers”,** dos autores Fabíola Jardim Barbon, Rafael Ratto de Moraes, Josiane Viccan Calza, Ana Paula Perroni, Aloisio Oro Spazzin e Noéli Boscato passou por revisão de inglês feita por mim com relação a seu conteúdo gramatical.

Pelotas, 26 de junho de 2017.



Diovana A. N. Dellagostin – CPE