



**UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA**

VITALIANO GOMES DE ARAÚJO NETO

**ADESÃO DO CIMENTO RESINOSO E TRANSMISSÃO DE LUZ ATRAVÉS
DA ZIRCÔNIA MULTICAMADA**

**ADHESION OF RESIN CEMENT AND LIGHT TRANSMISSION THROUGH
MULTI-LAYERED ZIRCONIA**

PIRACICABA-SP

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ADHESION OF RESIN CEMENT AND LIGHT TRANSMISSION THROUGH
MULTI-LAYERED ZIRCONIA

Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Mestre em Materiais Dentários.

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Orientador: Prof. Dr. Marcelo Giannini

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RESUMO

Avaliar a influência das cores nas multicamadas da zircônia na resistência de união por cisalhamento (SBS) do cimento resinoso, na transmissão de luz (LIT) e no grau de conversão (DCO) do cimento resinoso. Diferentes regiões da Katana Zirconia UTML foram identificadas como opaca (OPQ) ou translúcida (TNS). Um disco de zircônia foi seccionado com disco de diamante para obter amostras retangulares para análise de SBS LIT e DCO, após a sinterização. Os procedimentos adesivos foram realizados nas regiões OPQ e TNS com o sistema de cimentação Panavia V5 (*primers* e cimento resinoso) e o teste SBS foi realizado após 24 horas da preparação das amostras (n=12). Os dados do SBS foram analisados usando "teste T de amostras independentes" ($\alpha = 0,05$). Amostras da zircônia com espessura de 1 mm foram usadas para avaliação LIT com uma unidade de fotopolimerização LED *monowave* (Elipar Deep Cure-S). A LIT foi avaliada nas duas regiões de opacidade diferentes (OPQ e TNS) e dados de potência, irradiância, perda de irradiância, irradiância espectral e coeficiente de absorbância foram coletados com espectrômetro (n=5). Os dados foram analisados pela ANOVA (um fator) e Tukey HSD post-hoc ($\alpha=0,05$). Para analisar o DCO, o cimento resinoso foi fotoativado seguindo três condições: exposição direta à luz e através das duas regiões OPQ e TNS de zircônia. Os espectros do cimento resinado não polimerizado foram obtidos e após a ativação da luz, novos espectros foram obtidos 5 min, 1 h e 24 h pós-cura. Os dados (n = 5) foram analisados pela ANOVA (dois fatores e medidas repetidas) e teste de Tukey ($\alpha = 0,05$). A análise estatística não mostrou diferença significativa na SBS entre as regiões OPQ e TNS. Ambas as regiões da zircônia reduziram significativamente a potência, a irradiância e o DCO do cimento resinoso. OPQ causou maior perda de irradiância, maior coeficiente de absorbância e menor DCO que a região TNS. Diferentes regiões de opacidades/cores (OPQ e TNS) não influenciaram a SBS, entretanto ambas as regiões de zircônia afetaram a LIT através dela e OPQ produziu maior atenuação da luz e menor grau de conversão do cimento resinosos.

Palavras-Chave: Cerâmica. Cimentos Dentários. Polimerização. Resistência ao Cisalhamento.

ABSTRACT

To evaluate the influence of shades of a zirconia multilayer on shear bond strength (SBS) of resin cement, light transmission (LIT) and degree of conversion (DCO) of resin cement. Different shade/opacity regions of Katana Zirconia UTML were identified as opaque (OPQ) or translucent (TNS) groups. Zirconia disc was sectioned with a diamond blade to obtain rectangular samples for SBS, LIT and DCO analysis, after sintering. The bonding procedures were performed on OPQ and TNS regions with Panavia V5 cementing system (primers and resin cement) and SBS test conducted after 24 hours from specimen preparation (n=12). SBS data were analyzed using an "Independent Samples t-test" ($\alpha=0.05$). Zirconia samples with one mm thick were used for LIT evaluation with a monowave LED light-curing unit (Elipar Deep Cure-S). LIT was performed through different opacity regions (OPQ and TNS) and data of power, irradiance, irradiance loss, spectral irradiance and absorbance coefficient were collected using a spectrometer (n=5). Data was analyzed using one-way ANOVA and post-hoc Tukey HSD ($\alpha=0.05$). To analyze de DCO, resin cement was polymerized using one of three conditions: direct light exposure and through two OPQ and LIT regions of zirconia. Infrared spectra of the uncured resin cement were recorded and after light-activation, new spectra were obtained 5 min, 1 h and 24 h later. Data (n=5) were analyzed by two-way repeated measures ANOVA and Tukey's test ($\alpha=0.05$). Statistical analysis did not shown SBS significant difference between OPQ and TNS regions. Both regions of zirconia reduced significantly the power, the irradiance and DCO of resin cement. OPQ produced higher irradiance loss, higher absorbance coefficient and lower DCO than that through TNS. Different shade/opacity regions (OPQ and TNS) did not influence the SBS, however both regions of zirconia affected the LIT through the material and OPQ promoted the highest light attenuation and the lowest DCO of resin cement.

Key Words: Ceramics. Dental Cements. Polymerization. Shear Bond Strength.

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1 INTRODUÇÃO

As cerâmicas odontológicas têm amplo uso como materiais restauradores indiretos nos casos de reabilitação estética e funcional. A zircônia é um tipo de cerâmica muito utilizada e que passa por um processo de sinterização do óxido de zircônio e estabilizadores (óxido de ítrio) para manter sua forma cristalina mais resistente em temperatura ambiente (Tinschert et al., 2001). Após sinterização, a zircônia apresenta-se opaca, o que não favorece para o uso estético (Luthardt et al., 2002). A ausência da fase vítrea na estrutura da zircônia causa baixa translucidez, que resulta em um material pouco estético. Por isso no começo do seu uso em Odontologia ela era utilizada como estrutura interna para coroas e próteses fixas, sendo revestida com cerâmica feldspática mais translúcida (Herffeman et al., 2002; Sailer et al., 2015).

Algumas tentativas para melhorar a estética da zircônia foram utilizadas, entre elas a adição de materiais cerâmicos tradicionais na composição da zircônia, porém não obteve sucesso por não apresentar alta resistência ao lascamento (Rekow et al., 2011). Outra alternativa foi aumentar a porcentagem de ítrio em sua fase cúbica. Houve melhoras na estética da zircônia, porém diminuiu suas propriedades mecânicas (Zhang et al., 2016; Zhang Y., 2018).

Zircônias odontológicas mais atuais estão sendo formuladas com multicamadas de cores para melhorar as propriedades estéticas, simulando a naturalidade do dente de acordo com cada região, sendo a borda incisal mais translúcida e a região cervical opaca. Os pioneiros dessa tecnologia introduziram no mercado odontológico a zircônia Katana (Kuraray Noritake Dental Inc., Tóquio, Japão), que tem despertado o interesse de pesquisadores e clínicos nas restaurações monolíticas (Kolakarprasert et al., 2019). O presente estudo utilizou a “Zirconia Katana Ultra Translucent Multi Layered” (UTML), que contém quatro camadas: camada de esmalte (35%); camada de transição 1 (15%); camada de transição 2 (15%) e corpo/dentina (35%), de acordo com o fabricante.

Com relação ao material cimentante para as zircônias, o principal fotoiniciador, a canforquinona é o mais utilizado nos materiais resinosos restauradores, sendo sensível do espectro visível da luz azul (425-510 nm). Os fabricantes também usam co-iniciadores para melhorar o grau de conversão dos cimentos resinosos em regiões que não são atingidos pela luz (Vrochari et al., 2009). Aparelhos LED *monowave* ou *single*

peak emitem comprimento de onda no pico da ativação da canforquinona e possuem distribuição mais homogênea do feixe de luz para promover uma melhor abrangência da superfície a ser fotoativada. Assim, a eficiência da fotoativação depende da exposição adequada para garantir maiores propriedades físicas (Rocha et al., 2017). Pigmentos são usados para alterar as cores da zircônia multicamadas e hipoteticamente alteram a composição local, além de reduzir a transmissão da luz através do material protético. As consequências disso poderiam ser na adesão e polimerização do cimento resinoso. Para investigar os possíveis efeitos das multicamadas da zircônia, este estudo avaliou a influência das regiões opaca e translúcida da zircônia na resistência de união do cimento resinoso, na transmissão da luz através dessas diferentes regiões do material protético e seus efeitos no grau de conversão de um cimento resinoso.

2 ARTIGO:

Adhesion of resin cement and light transmission through multi-layered zirconia

Abstract

PURPOSE: To evaluate the influence of shades of a zirconia multilayer on shear bond strength (SBS) of resin cement, light transmission (LIT) and degree of conversion (DCO) of resin cement.

METHODS: Different shade/opacity regions of Katana Zirconia UTML were identified as opaque (OPQ) or translucent (TNS) groups. Zirconia disc was sectioned with a diamond blade to obtain rectangular samples for SBS, LIT and DCO analysis, after sintering. The bonding procedures were performed on OPQ and TNS regions with Panavia V5 cementing system (primers and resin cement) and SBS test conducted after 24 hours from specimen preparation (n=12). SBS data were analyzed using an “Independent Samples t-test” ($\alpha=0.05$). Zirconia samples with one mm thick were used for LIT evaluation with a monowave LED light-curing unit (Elipar Deep Cure-S). LIT was performed through different opacity regions (OPQ and TNS) and data of power, irradiance, irradiance loss, spectral irradiance and absorbance coefficient were collected using a spectrometer (n=5). Data was analyzed using one-way ANOVA and post-hoc Tukey HSD ($\alpha=0.05$). To analyze de DCO, resin cement was polymerized using one of three conditions: direct light exposure and through two OPQ and TNS regions of zirconia. Infrared spectra of the uncured resin cement were recorded and after light-activation, new spectra were obtained 5 min, 1 h and 24 h later. Data (n=5) were analyzed by two-way repeated measures ANOVA and Tukey’s test ($\alpha=0.05$).

RESULTS: Statistical analysis did not shown SBS significant difference between OPQ and TNS regions. Both regions of zirconia reduced significantly the power, the irradiance and DCO of resin cement. OPQ produced higher irradiance loss, higher absorbance coefficient and lower DCO than that through TNS.

CONCLUSION: Different shade/opacity regions (OPQ and TNS) did not influence the SBS, however both regions of zirconia affected the LIT through the material and OPQ promoted the highest light attenuation and the lowest DCO of resin cement.

1. Introduction

Development of CAD/CAM technology has allowed for dental zirconia ceramics to be increasingly used as prosthetic restorative materials, and recently even for anterior esthetic restorations (Almazdi et al., 2012). Yttria-stabilized tetragonal zirconia (3Y-TZP) was introduced in dentistry due to high mechanical strength; however, the absence of a glassy phase produced low translucency and therefore limited esthetic results, so it was used as framework for crowns and fixed dental prostheses that were coated with more translucent feldspathic ceramics (Herffeman et al., 2002; Sailer et al., 2015). The development of new zirconia ceramics has resulted in a material with better esthetic properties, because the formulations with either 4 mol% (4Y-PSZ) or 5 mol% (5Y-PSZ) of yttrium oxide, and present an increased amount of chemical element in their cubic phase.

The development of Y-PSZ also resulted in multi-layered presentations, where manufacturers intend to replicate the naturality of teeth according to each anatomical region, keeping translucency at the incisal edge and a higher opacity at the cervical area. To achieve that, during the manufacturing process, different amount of pigments are added to the zirconia powder and compacted in layers that may be more opaque or translucent (Kolakarnprasert et al., 2019), as a result of the pigment content. It has been reported that Katana UTML (Kuraray Noritake Dental Inc. Tokyo, Japan) has the highest translucency among commercially available translucent zirconia ceramics, resulting in a promising monolithic restorative material for aesthetic results (Inokoshi et al., 2018; Kolakarnprasert et al., 2019).

As a result of the better optical properties, Y-PSZ have been indicated for using as crowns and laminates on anterior teeth (Zhang Y., 2018), although it has been reported that modifications needed to produce Y-PSZ result in decreased mechanical properties of the zirconia (Zhang et al., 2016; Zhang Y., 2018). However, its composition still makes it difficult for resin cement to bond to zirconia ceramics (Caminaci et al., 2019). Despite the wide variety of protocols studied for adhesive cementation of zirconia restorations, the use of the 10-MDP monomer combined with aluminum oxide abrasion has been established as the gold standard for clinically successful cementation of zirconia ceramic with resin cements. The study of adhesion in

multi-layer zirconia is important because it has been indicated in cases of poor mechanical retention of the prepared tooth, such as laminates, where the weak point of adhesion would be between zirconia and resin cement (Inokoshi et al., 2018).

Besides adhesion to Y-PSZ zirconia, little is known about the attenuation of light emitted from the LED curing units that may affect polymerization of dual and light-cured cements. Insufficient polymerization of resin cements has been related to debonding and restoration fracture, marginal staining, secondary caries, monomers release and others (Faria-e-Silva et al., 2017; Soares et al., 2017). An efficient photoactivation depends on adequate light exposure to ensure optimal chemical and physical properties of resin cement (Rocha et al., 2017). For that reason, manufacturers have also used chemically initiators to increase the degree of conversion in regions receiving insufficient irradiance (Vrochari et al., 2009; Price et al., 2015).

The transmission of light in an appropriate wavelength range to activate dental photoinitiators remains to be evaluated to determine how does the translucency or opacity of Y-PSZ affects light transmission and consequently the polymerization of the resin cement. This study evaluated the influence of shades of a zirconia multilayer on shear bond strength of resin cement, light transmission through different regions of a highly translucent zirconia and degree of conversion of resin cement. The working hypothesis were that: (1) shear bond strength of a resin cement to translucent and opaque areas of a multi-layer Y-PSZ zirconia would be different between regions tested; (2) transmission of light from a monowave LED curing unit through different layers of a multi-layer Y-PSZ would be different, and (3) would influence on the degree of conversion of resin cement.

2. Material and Methods

Zirconia sample preparations

The tested materials are summarized in Table 1. One non-sintered, highly translucent, yttria partially stabilized zirconia (Y-PSZ, Katana UTML, Kuraray Noritake Dental Inc. Tokyo, Japan) disc was sectioned in slabs using a slow-speed diamond-wafering blade (Isomet 1000 Precision Saw; Buehler Co, Lake Bluff, IL, USA) under copious water irrigation. Specimens were sintered in a furnace (Dekema Austromat 674, Freilassing, Germany) at a heat rate of 10°C/min to a final temperature of 1500°C. After

sintering, the plates reached a final size of 11.4 mm length x 5 mm wide x 2 mm thickness.

Shear bond strength test

Zirconia plates were sandblasted with 50 μ m Al₂O₃ particles (BioArt, Sao Carlos, SP, Brazil), using a microetcher (Danville Engineering, Danville, San Ramon, CA, USA) for 15 s/cm² (Inokoshi et al., 2018) at 0.3 MPa. At each plate, the different opacity regions were identified as opaque or translucent, and marked to identify different regions for bond strength test. Black isolating tape (3M Scotch, Sumaré, SP, Brazil) with a 2 mm diameter aperture was placed over the zirconia to isolate the bonding areas. A single cylinder of the resin cement Panavia V5 (shade A2, Kuraray Noritake Dental Inc. Tokyo, Japan) was built (1.5 mm diameter x 1.5 mm height) using poly-vinyl siloxane matrixes (Virtual; Ivoclar Vivadent, Schaan, Liechtenstein) in each region (opaque or translucent). The exposed ceramic surface was primed (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental Inc. Tokyo, Japan) according to the manufacturer instructions of Panavia V5 and a thin layer of Tooth Primer (Kuraray Noritake Dental Inc. Tokyo, Japan) was applied before the resin cement to achieve the accelerator effect for chemical curing reaction. Resin cement cylinders were subjected to direct light exposure for 10 seconds using a monowave light-emitting diode (LED) curing unit (Elipar Deep Cure-S, 1,280 mW/cm² radiant emittance, 3M Oral Care, Saint Paul, MN, USA). Twelve cylinder-shaped cement specimens were prepared at the most translucent and opaque regions of zirconia plate. After matrix removal, the zirconia plates with resin cylinders were stored in a dark oven at 37°C (Fanen, São Paulo, SP, Brazil) in distilled water for 24 h before the bond strength test.

The diameter of each specimen was measured with a digital caliper (Mitutoyo Sul Americana Ltda, Suzano, SP, Brazil) to compensate for minor size variations in the bond strength by area. The ceramic plate with the resin cylinders was fixed to the testing device with a cyanoacrylate adhesive (Super Bonder Power Flex, Loctite, São Paulo, SP Brazil), and shear bond strength testing was performed using a Universal testing machine (EZ-test-500N, Shimadzu Co., Kyoto, Japan). A thin wire (diameter 0.20 mm) was looped around the resin cylinder and gently placed at the resin cement/ceramic interface, making contact through half its circumference. The wire loop, the resin–ceramic interface and the center of the load cell were aligned to ensure concentration of

shear force at the bonded area. Shear load was applied at a cross-head speed of 1 mm/min until failure occurred. Maximum SBS was recorded in N and transformed to MPa by dividing the result by the bonded area of each specimen (n= 12).

Fracture pattern analysis

The fractured surfaces of the tested specimens were sputter-coated with gold and examined using scanning electron microscopy (SEM) at $\times 35$ magnification (JSM IT 300; Jeol, Tokyo, Japan). The bonded areas were examined to classify the failure modes as follows (1) cohesive failure within resin cement, (2) adhesive failure at the zirconia-resin cement interface or (3) mixed failure.

Light transmission measurements

To determine the effect of different opacities of multilayer zirconia on the attenuation of light passing through the material, five plates with 1 mm thick were used. A flatted surface of the plates was completely covered with a black isolating tape (3M Scotch, Sumaré, SP, Brazil) except for a circular 5 mm diameter aperture at the most opaque and translucent regions, where measurements of light transmission were performed. The same LED curing unit was used to obtain the power, irradiance and spectrum of light passing through the 5 mm aperture were recorded using a spectrometer (STS-VIS-L-100-400 SMA, Ocean Optics, Dunedin, FL, USA) coupled to a software (SpectraSuite version 1.4.2, Ocean Optics, Dunedin, FL, USA) at 1.3 mm distance from the spectrometer. Baseline values of power, irradiance and emission spectrum without any material between the light guide and the sensor of spectrometer were also recorded for reference as a control. The spectral irradiance transmission ($\%T\lambda$), from 400 to 500 nm, was calculated using the following formula:

$$\%T\lambda = (I/I_0) \cdot 100$$

where "I" refers to the irradiance obtained at each wavelength through the zirconia and "I₀" was the baseline irradiance at each wavelength.

Absorbance coefficients were determined for wavelengths from 420 to 490 nm. Absorbance (AU) and absorbance coefficients (e) were calculated according to the formula, respectively:

$$AU: -\log(1/I_0)$$

$$\varepsilon = \frac{-\log(1/I_0)}{\text{mm}}$$

Where I was the irradiance value for each evaluated sample, I_0 was the irradiance for baseline, and mm was the sample thickness (in mm).

Degree of conversion measurements

The same zirconia plates used for the light transmission measurements were selected for degree of conversion analysis.. The resin cement (Panavia V5) was applied to the zirconia plate and the uncured resin cement paste was seated on a cover glass. Light activation of resin cement was performed for 10 seconds with Elipar Deep Cure through the zirconia plates at the OPQ and TNS regions, or through a glass slide (2 mm thick) that was the control. The samples were placed to a previously calibrated micro-Raman spectrophotometer (Xplora, Horiba JobinYvon, Paris, France) and the spectrum of each resin cement sample was recorded, using a HeNe laser with 3.2 mW power (532 nm wavelength, 1.5 μm spatial resolution and 2.5 cm^{-1} spectral resolution at 10 \times magnification lens, Olympus, London, UK) at post-curing times of five minutes, one hour and 24 hours. Samples were kept dry in darkness at 37°C during the whole experiment and in each one, two spectra were collected at different locations of cured resin cement (Miletic and Santini, 2012). The degree of conversion was calculated based in a previous investigation by means of the formula:

$$\text{DC} = [1 - (\text{peak area})_{\text{polymerized}} / (\text{peak area})_{\text{unpolymerized}}] \times 100$$

The absorption (peak area polymerized) at 1635 cm^{-1} aliphatic carbon-carbon double bond and 1610 aromatic carbon-carbon double bond (peak area unpolymerized) cm^{-1} were recorded.

Statistical analysis

Bond strength data were analyzed using an Independent Samples t-test ($\alpha = 0.05$). Light transmission data were analyzed for homogeneity with Levene's test and for normality with the Shapiro-Wilks test. After normality was corroborated, data was analyzed using one-way ANOVA and *post-hoc* Tukey HSD ($\alpha = 0.05$) to compare

group means using SPSS v.23 software (IBM SPSS Statistics, IBM Corp, Armonk, NY, USA).

For absorbance coefficient, a linear regression analyses were performed on the relationships between wavelength and absorption coefficients for a given material shade (Excel 2016, Data Analysis Tools: Regression Analysis, Microsoft Corporation, Redmund, WA, USA). The 95% confidence intervals for each slope value were compared to verify if there was an overlap. Degree conversion data were analyzed by two-way repeated measures ANOVA and *post-hoc* Tukey's test ($\alpha = 0.05$).

3. Results

Shear bond strength

The bond strength results are presented in Table 2. Statistical analysis did not shown significant bond strength differences between opaque (18.6 ± 8.4 MPa) and translucent (17.6 ± 6.2 MPa) areas. The percentages of each failure mode and representative images of each failure are presented in Figure 1. Mixed failure was the most frequent for both bonded areas and the incidence for each type of failure was similar for both ones.

Light transmission measurements

Results of power, irradiance and irradiance loss are presented in Table 3. There were significant differences for the power and irradiance at baseline and those transmitted through the different shades/layers. The power and irradiance were significantly lower ($p < 0.05$) at opaque area (26 mW and 132.2 mW/cm²) compared to translucent one (37.3 mW and 190 mW/cm²). The irradiance loss was significantly higher in the opaque region than that found at translucent one. For the absorbance coefficient, results showed no overlap between the verification intervals with higher absorbance for opaque area, with increasing absorption following increasing wavelength.

Degree of conversion measurements

Data of degree of conversion of resin cement through zirconia at five minutes, one hour and 24 hours are presented in the Table. 4. There were significant differences among control, TNS and OPQ groups ($p < 0,001$) and times ($p < 0,001$). The direct light exposure (control) showed the highest degree of conversion at all times, followed by TNS, and OPQ that caused the lowest degree of conversion. Degree of conversion of resin cement increased over time, with higher values being achieved after 24 hours.

4. Discussion

The first working hypothesis stating that shear bond strength of a resin cement to zirconia would be different between layers was rejected because the results showed that there were no statistical differences in the SBS either at the translucent or opaque layers. The second working hypothesis stating that light transmission from a monowave LED curing unit through different layers of a multi-layer Y-PSZ would be different was accepted because power, irradiance and absorbance coefficient were significantly different through translucent and opaque regions/layers. Third hypothesis was also accepted because transmission of light through different layers of zirconia influenced the degree of conversion of resin cement.

Bond strength results can be explained because the zirconia powder formulation at the different layers is the same and the only changes are related to the pigment content. Thus, results showed that bond strength was not influenced by the contained opaque and pigments of the layers. The multilayer zirconia used in this study presents four layers, according to its manufacturer: enamel layer (35%); transition layer 1 (15%); transition layer 2 (15%) and body layer (dentin) 35%) (Yamada et al., 2016). It was decided that two most peripheral layers of each zirconia plate would be used, because both are larger and show the greater differences in opacity, allowing performing the bond strength test in a circular area of 7.065 mm^2 . The two-intermediate or transition layers are not easily distinguished to the naked eye, and therefore could not be evaluated individually and correctly.

The composition of self-cured and light-cured resin cements varies among manufacturers, and the sensitivity of the polymerization activation systems may affect the polymerization of them under conditions with less than ideal light exposure. In this

study, the commercial dual-cure cementing system used (Panavia V5, Kuraray Noritake Dental Inc.) contains, besides resin cement pastes, two primers to be specifically applied to zirconia (Clearfil Ceramic Primer Plus) and tooth (Tooth Primer). According to its instructions, manufacturer recommends 5 seconds of light activation, or 5 minutes for self-curing. Its self-curing mechanism is dependent on the presence of catalysts from the Tooh Primer that is used to activate a slower chemical reaction of the resin cement.

Thus, to replicate the clinical scenario for cementation of a zirconia ceramic indirect restoration and yield better mechanical properties for the resin cement, Tooth Primer was applied over the primed zirconia with the ceramic primer. Clinically, the Tooth Primer is mixed with the cement and this mixture comes into contact with primed zirconia. For some cementing systems, light-cure and chemical-cure activation mechanisms are not integrated (Leprince et al., 2013), therefore each component of the cementing system has a specific function and must be used in order to reach the best curing conditions during the cementation procedures. Although curing light tends to increase the degree of conversion of resin cements, the early vitrification promoted by light-activation can impair the autopolymerization reaction (Manso et al., 2017).

The bonding of resin cement to zirconia occurs with the 10-MDP monomer from cementing system and is based on chemical reaction between the hydroxyl chains of the monomer to zirconium oxide (Ozcan M., 2015). 10-MDP monomer is present in both primers (Clearfil Ceramic Primer Plus and Tooth Primer) that are used in the pretreatment of zirconia and prepared tooth, respectively (Butler et al., 2018). Unlike previous versions, 10-MDP is not present in the resin cement paste formulation. Studies have reinforce that the use of 10-MDP is more favorable and beneficial when incorporated into primers (Chen et al., 2017; Inokoshi et al., 2014).

The adhesive failure implies rupture of the cement-zirconia interface and showed approximately 30% of the failures (Figure 1B). The mixed failure was the most observed in the samples and corresponded to approximately 60% (Figure 1C). The presence of resin cement on the zirconia surface may indicate a good adhesion of the resin cement promoted by the primer. Less than 15% of the samples failed within resin cement structure (Figure 1D). This may indicate a good adhesion or poor polymerization, which reduced the resistance of the resin cement becoming it the weak link during shear bond strength test.

Increasing of sorption and solubility has been observed in self-adhesive resin cements because acid-hydrophilic monomers are not neutralized during the curing reaction. Under acidic conditions, the incomplete polymerization reaction might increase water sorption and solubility at tooth-resin cement-zirconia interface (Marghalani HY, 2012). In this the study use of the Tooth Primer in the absence of dental structure may lead to the same problem reported for self-adhesive resin cements. The consequences with use of this primer or not on polymerization reaction of resin cement and bonding stability can be detected by resin cement monomer conversion analysis and the water-storage of the bonded samples for a long time, following the bond strength test and new failure pattern investigation.

Clinician and readers should carefully consider that in this study the bond strength results were not influence by light attenuation that might affect polymerization of the resin cement, because resin cement inside the mold was directly exposed to LED. The translucency in zirconia remains a challenge because of the presence of crystals and pigments attenuate the light transmission. To increase transmittance of Y-PSZ zirconia, grain size was reduced to allow the passage of visible light and improve material aesthetics (Zhang Y 2018). However, the composition and microstructure of each layer material may influence translucency due to differences between the refractive index of yttrium crystals, the size of the filler particles and the wavelength of the incident light, all factors that influence light scattering (Lise et al., 2018).

This study showed that the light transmission was higher for translucent region and with lower total irradiance loss compared to opaque one, using zirconia plates of 1 mm thick. Besides the chemical composition and cubic microstructure of the material, the thickness of the prosthetic material is another determining factor for light transmission, (Kolakarnprasert et al., 2019). Although the manufacturer indicates the presence of Ti and Fe pigments at the layers to explain for translucency differences, the pigment content could explain the higher light transmission in the translucent layer (Sedda et al., 2015). Differences in composition have allowed for better aesthetics as a result of the increased translucency, while acceptable physical properties are maintained (Sulaiman et al., 2015).

It has been proposed that effective activation of photoinitiators in the resin cements produce better mechanical properties, higher degree of conversion and longer

clinical durability of indirect restorations (Rueggeberg et al., 1994). On the other hand, inadequate polymerization may cause early monomer degradation, that besides affecting the mechanical properties, clinically can cause color alterations (Stansbury JW, 2012). In this study, the curing unit emits light in a wavelength range that matches the absorption spectra of camphorquinone (425–490 nm) (Price et al., 2010), which is the most used dental photoinitiator and is present in the resin cement.

Regarding the curve graphic observed in Figure 3, the zirconia opacities and wavelengths strongly influenced linear equations. It was noted that shorter wavelengths, such as violet, showed higher absorbance values for both areas studied. Opaque region showed higher absorbance coefficient values for all wavelengths (420 to 490 nm), which can be explained by different nature of the material in terms of pigments used for increasing the opacity.

Differences in the degree of conversion of resin cement were observed between the evaluated regions, as result of the different absorbance of the blue curing light by the zirconia layers. The high absorbance in the opaque region reduced the degree of conversion of the resin cement. Because the opaque region is located at the cervical margin of the indirect restoration, inadequate polymerization of the resin cement may reduce the longevity of the luting material. Therefore, it has been recommended to apply complementary light curing cycles to conclude light activation procedures (Price et al., 2015), where the light transmission is very low. Translucent area also reduced the light transmission, but was significantly higher than that at opaque areas, resulting in higher degree of conversion. Finishing and adjust occlusion procedures must consider that degree of conversion of resin cement increases over time, achieving higher values at 24 hours,

5. Conclusions

Within the limitations of the present in vitro study, it can be stated that:

1. There is no difference in the bond strength of dual cure resin cement to the different layers (translucent and opaque) of Katana UTML zirconia.

2. The differences in translucency of zirconia produce significant differences in the transmission of light emitted by a LED-curing unit. Transmission of light was significantly higher at the more translucent layer.

3. Despite presenting the same composition and microstructure, modifications in the different layers to achieve different optical properties and esthetics also increase the absorbance at opaque areas of the multi-layering zirconia.

4. Opaque area reduced the degree of conversion of resin cement, which increases until 24 hours.

6. Acknowledgment

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Attached:

Table 1. Materials used and respective manufacturer information.

Material	Product name	Composition	Lot number
Zirconia Ceramic	Katana Zirconia UTML	Zirconium oxide, yttrium oxide, pigments	DOQCO
Primer	Clearfil Ceramic Primer Plus	3-Methacryloxypropyl trimethoxysilane, 10-MDP, ethanol	A40030
Adhesive	Tooth Primer	10-MDP, 2-HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water	BL0046
Resin Cement	Panavia V5	Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroalminosilicate glass filler Paste B: Colloidal silica, Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminum oxide filler, accelerators, dl-camphorquinone, pigments	1R0010

Abbreviations: 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP); Hydroxyethyl methacrylate (HEMA); bisphenol A-glycidyl methacrylate (Bis-GMA); Triethylene glycol dimethacrylate (TEGDMA).

Table 2. Mean and standard deviations (SD) of shear bond strengths (in MPa) of resin cement to zirconia.

Group	Mean SBS
OPQ	18.6 (8.5) A
TNS	17.6 (6.2) A

Abbreviations: Opaque (OPQ); Translucent (TNS).

Table 3. Baseline and tested Power, Irradiance and Irradiance Loss means through different layers of zirconia.

Power (mW)			Irradiance (mW/cm ²)			Irradiance Loss (%)	
Baseline	TNS	OPQ	Baseline	TNS	OPQ	TNS	OPQ
523.7 (2.2) A	37.3 (4.9) B	26.0 (3.8) C	2668.4 (11.3) A	190.0 (25.1) B	132.2 (19.4) C	92.9 (0.9) B	95.1 (0.7) A

Means followed by different letters indicate statistical difference ($p < 0.05$).

Abbreviations: milliWatts (mW); Translucent (TNS), Opaque (OPQ).

Figure 1. Percentage of failure patterns (%) obtained at Opaque (OPQ) and Translucent (TNS) regions. after SBS. Bar graph of failure patterns percentage (Fig. 1A). SEM image represents an adhesive failure (Fig. 1B). Mixed failure was predominant (Fig. 1C) and the cohesive failure within resin cement (Fig. 1D) showed low incidence.

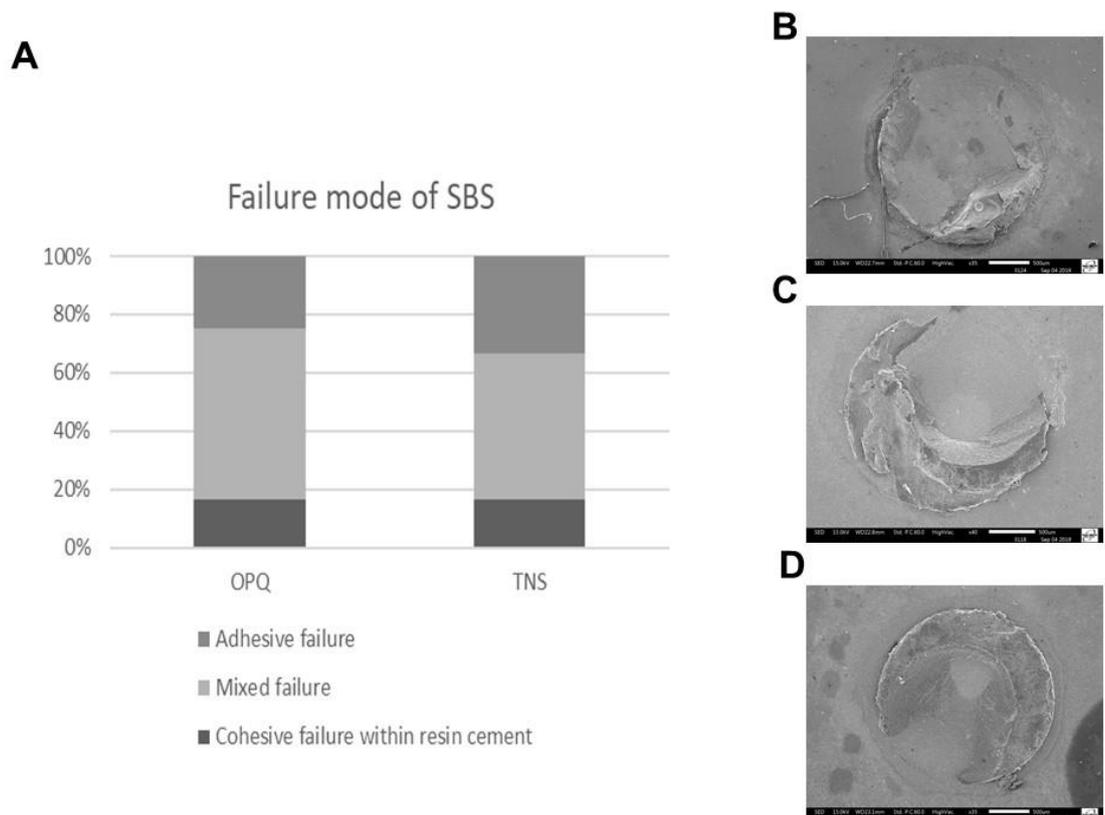


Figure 2. Spectral irradiance ($\text{mW}/\text{cm}^2/\text{nm}$) through Translucent (TNS) and Opaque (OPQ) regions of zirconia. BASELINE: direct exposure (without interposition of indirect material between the tip of light-curing unit and the spectrometer).

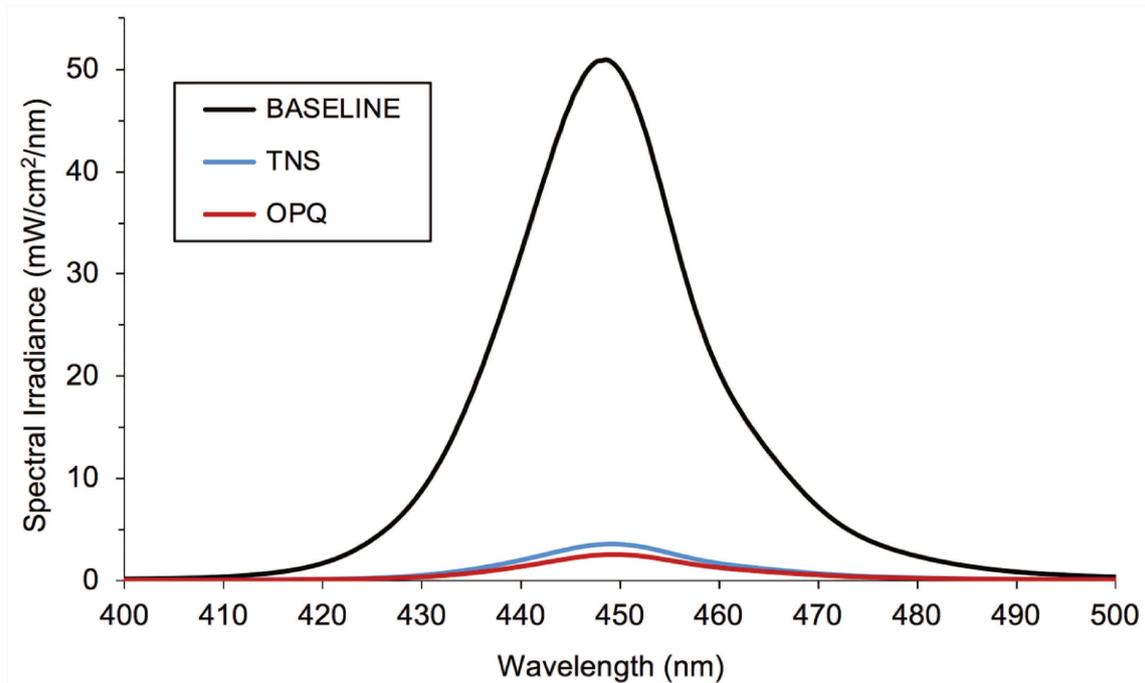


Figure 3. Absorbance coefficient promoted by Translucent (TNS) and Opaque (OPQ) regions of the zirconia.

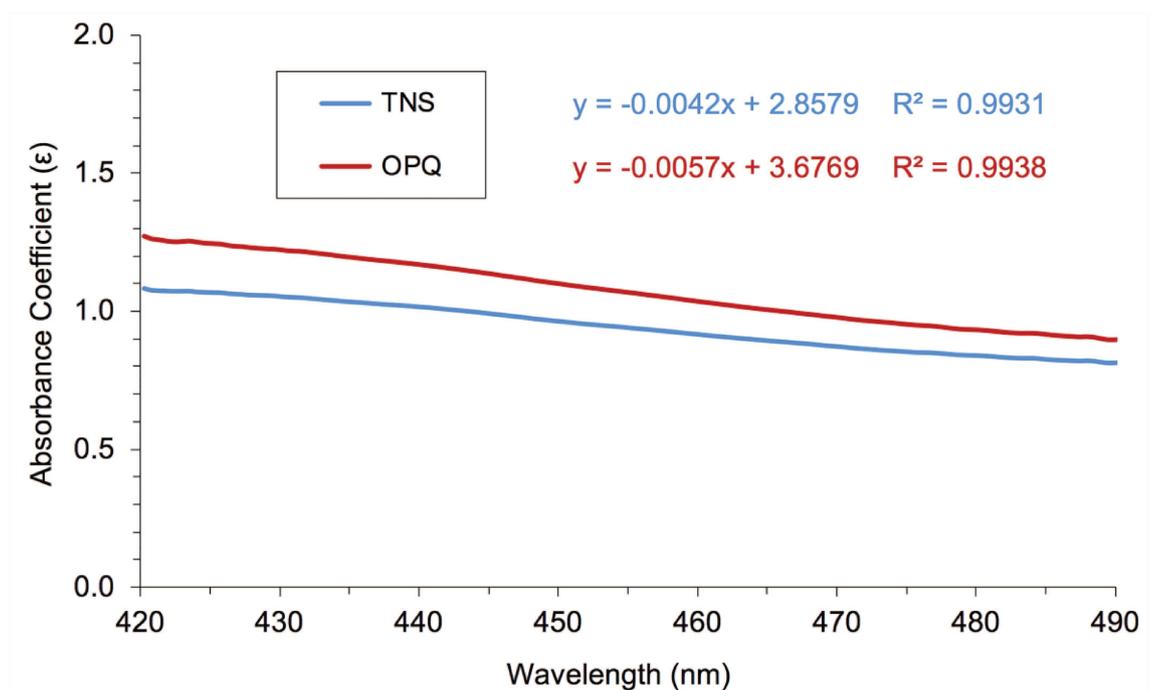


Table 4. Degree of conversion results (%) of resin cement, according to the evaluation times post light activation.

<i>Groups</i>	<i>Evaluation time</i>		
	5 minutes	1 hour	24 hours
<i>OPQ</i>	18.5 (4.6) C c	39.0 (5.8) B c	52.4 (9.5) A c
<i>TNS</i>	48.7 (5.5) C b	59.3 (6.6) B b	71.2 (9.8) A b
<i>Direct light exposure (control)</i>	61.8 (6.8) C a	71.2 (5.6) B a	83.1 (3.5) A a

Means followed by different letters indicate statistical difference ($p < 0.05$). Capital letters indicate significant ($p < 0.05$) differences among times and for the same group. Small case letters depict statistical differences ($p < 0.05$) among groups within the same evaluation time. Abbreviations: Translucent (TNS), Opaque (OPQ).

3 CONCLUSÃO

Diferentes regiões de opacidades (opaca e translúcida) da zircônia não influenciaram a resistência de união do cimento resinoso. Ambas as regiões de zircônia afetaram a transmissão de luz e a área mais opaca promoveu maior atenuação da luz e consequentemente menor grau de conversão do cimento resinoso.

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