Helena Letícia Quirino de Oliveira

Caracterização mecânica e óptica de resinas compostas de cor única utilizadas em dentes posteriores

Mechanical and optical characterization of single-shade resin composites used

in posterior teeth

Dissertação apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia como requisito parcial para obtenção do título de Mestre em Odontologia na Área de Clínica Odontológica Integrada.

Uberlândia, 2022

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Orientador: Prof. Dr. Carlos José Soares

Banca examinadora: Prof. Dr. Carlos José Soares Prof. Dr. Hugo Lemes Carlo Profª Drª Larissa Silveira de Mendonça Fragoso

UNIVERSIDADE FEDERAL DE URERLÂNDIA

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Sumário

Resumo

OBJETIVO: Este estudo teve como objetivo avaliar o desempenho óptico e mecânico de duas resinas compostas de cor única em comparação com uma resina composta convencional para a técnica de preenchimento incremental em dentes posteriores.

MÉTODOS: Duas resinas de cor única, Omnichroma (Tukoyama) e Vittra Unique APS (FGM) e um Resina Composta convencional, Filtek Z350XT cor A2 (3M Oral Care) foram testados neste estudo. O desempenho óptico envolveu a análise da correspondência de cores verificada usando espectrofotômetro. A transmissão da luz foi realizada utilizando o método de perfil de feixe do VALO Grand (Ultradent) e da luz transmitida através dos 2,0mm de cada amostra de resina. O desempenho mecânico foi expresso pelo cálculo da dureza Knoop (KH, N/mm2), grau de conversão (DC, %) na parte superior e inferior usando espectroscopia Raman, a resistência à flexão (FS, MPa) e o módulo de elasticidade (E, MPa). Foi realizada microscopia eletrônica de varredura para caracterização da carga. Os dados de FS, E, DTS, CS e Shr foram analisados por ANOVA de 1 via e KH e DC por ANOVA de 1 vias repetidas seguidas pelo teste de Tukey (α = 0,05), e a tensão residual foi analisada quantitativa e qualitativamente pelo critério de von Mises modificado. Os valores de tensão de von Mises modificados, a transmissão de luz e as imagens MEV foram analisados qualitativamente.

RESULTADOS: Os valores de KH e DC reduziram significativamente em relação a base para o topo para todas as resinas (P < 0,001), porém os valores de razão sempre foram superiores a 80%. Houve diferença significativa entre as resinas para $E (P < 0.001)$, DTS $(P = 0.008)$, valores maiores para Filtek Z350XT; CS (P<0,001) Vittra Unique APS e Filtek Z350XT tiveram valores menores que Omnichroma. As resinas de cor única obtiveram tensão de contração semelhante ao dente durante o carregamento oclusal. O Filtek Z350XT resultou em um pouco mais de tensão de contração dentro das resinas testadas. Não houve diferença para FS (P = 0,083) e Shr (P = 0,144). Omnichroma mostrou menor diferença de cor do que Vittra Unique APS, independentemente da tonalidade. Todas as resinas de cor única apresentaram maior variação de cor em comparação com a resina controle. A luz transmitida através de todas as resinas diminuiu significativamente. Omnichroma e Vittra Unique APS aumentou a transmissão de luz durante o processo de polimerização.

CONCLUSÃO: Omnichroma e Vittra Unique APS possuem propriedades mecânicas semelhantes ou superiores a Filtek Z350XT. As resinas de cor única tendem a aumentar a transmissão de luz durante o processo de fotoativação. A Omnichroma apresentou menor diferença de cor do que o Vittra Unique APS independente das cores dos dentes.

Palavras-chave: Resina composta monocromática; propriedades mecânicas; cor; tensão.

Abstract

OBJECTIVES: This study aimed to evaluate the optical and mechanical performance of two single-shade resin-based composites (RBCs) compared with a conventional RBC for incremental filling technique in posterior teeth.

METHODS: Two single-shade , Omnichroma (Tukoyama) and Vittra Unique APS (FGM) and a conventional RBC, Filtek Z350XT shade A2 (3M Oral Care) were tested in this study. Optical performance involved analysis of shade matching checked using spectrophotometer. The Light transmission was performed using beam profiling method of the VALO Grand (Ultradent) and of the light transmitted through the 2.0mm of specimen of each RBC. The mechanical performance was expressed by calculation of the Knoop hardness (KH, N/mm²), degree of conversion (DC, %) at top and bottom using Raman spectroscopy, the flexural strength (FS, MPa) and elastic modulus (E, MPa) using 3-point bending test, diametral tensile strength (DTS, MPa), compression strength (CS, MPa) using axial compression test, post-gel shrinkage (%) using strain-gauge test, shrinkage and residual stress using finite element analysis. Scanning electronic microscopy was performed for filler characterization. Data of FS, E, DTS, CS and Shr were analyzed by 1-way ANOVA and KH and DC by 1-way ANOVA repeated measurement followed by Tukey's test (α = 0.05), and residual shrinkage was analyzed quantitatively and qualitatively by modified the von Mises criteria. FS, DTS, CS, E were analyzed using a 1-way analysis of variance and KH and DC data were analyzed using a 1-way analysis of variance for repeated measures followed by Tukey post-hoc test ($α = 0.05$). The modified von Mises stresses values, light transmission and the SEM imagens were analyzed qualitatively.

RESULTS: KH and DC values reduced significantly from the top to the bottom of the specimens for all RBCs (*P* < 0.001), however the ratio values were always higher than 80%. There was significant difference among RBCs for E (*P* < 0.001), DTS (*P* = 0.008), higher values for Filtek Z350XT; CS (*P* <0.001) Vittra Unique APS and Filtek Z350XT had lower values than Omnichroma. The singleshade RBCs obtained similar shrinkage stress to the tooth during occlusal loading. Filtek Z350XT resulted in slightly more shrinkage stress within the

RBCs. There was no difference for FS $(P = 0.083)$ and Shr $(P = 0.144)$. Omnichroma showed less color difference than Vittra Unique APS irrespective of shade. All single-shade RBCs showed more color variation compared to the control RBC. The light transmitted through all RBCs decreased significantly. Omnichroma and Vittra Unique APS increased light transmission during the polymerization process.

CONCLUSION: Omnichroma and Vittra Unique APS have similar or superior mechanical properties than Filtek Z350XT. The single-shade RBCs tend to increase light transmission during the photoactivation process. The Omnichroma showed less shade difference than Vittra Unique APS independent of tooth shades.

Keywords: resin composite; mechanical properties; color; stress.

1. Introdução e referencial teórico

A demanda por resinas compostas com melhor estética, procedimentos menos demorados (Hussain & Khan, 2016; Soto-Montero *et al*., 2022), procedimentos mais confiáveis e previsíveis tem aumentado. (Cidreira *et al*., 2019) No entanto, a satisfação do paciente não é preenchida apenas com o desempenho mecânico da restauração, eles tendem a se concentrar inicialmente nos parâmetros mais estéticos. (Ismail & Paravina, 2022)

O dente humano é policromático, o que torna a seleção de cores para a restauração com resinas compostas um procedimento desafiador para os clínicos. (Iyer *et al*., 2021) Os fatores que influenciam o potencial de ajuste de cor das resinas compostas são classificados em três categorias: (1) tipo de material, (2) desenho da cavidade e (3) o substrato ao redor da restauração. Cada fator pode ser otimizado para melhorar a correspondência de cores e o resultado restaurador. (Ismail & Paravina, 2022) A determinação da cor do dente está relacionada a vários fatores, como condições de iluminação, características como translucidez, opacidade, dispersão da luz e brilho. (Joiner, 2004) O esmalte permite maior transmissão de luz que a dentina devido a sua estrutura prismática altamente mineralizada, baixo teor orgânico e pequena quantidade de água, sendo substrato mais translúcido. (Schmeling *et al*., 2010) A dentina possui maior quantidade de água, sendo menos translúcido quando comparado ao esmalte. (Schmeling *et al*., 2010)

Procedimentos restauradores com resinas compostas são comumente realizados pela técnica de estratificação. (da Costa *et al*., 2010; Zhang *et al*., 2022) Este é um procedimento complexo que incorpora a dificuldade de escolha da cor e espessura de cada incremento, principalmente em áreas mais extensas. (Ismail, 2021)

Os dentes naturais têm variações de tonalidade complexas, o que levou à fabricação de resinas em diferentes tonalidades. (Dietschi, 2016) As resinas compostas também estão disponíveis em diferentes opacidades, as resinas de maior opacidade são chamadas de dentina ou corpo e opacidade mais baixa são chamadas de esmalte ou translúcida, a combinação é importante para mimetizar as características ópticas do substrato do dente. Os profissionais também devem considerar características como valor, matiz e croma, além de outras propriedades ópticas, como translucidez, opacidade e brilho da superfície. (Lee, 2015)

Uma tecnologia recente lançada no mercado odontológico envolve resinas compostas universais, também chamadas de resinas monocromáticas ou de cor única. Resinas monocromáticas têm um efeito "camaleão" que pode imitar a cor da estrutura do dente. Essas resinas são capazes de interagir com a cor circundante do esmalte e da dentina, mimetizando as características ópticas independente das variações do ambiente e do operador. (de Abreu *et al.*, 2021)

O sucesso das restaurações dentárias é atribuído às suas propriedades estéticas e mecânicas. (Bicalho *et al*., 2014; Ismail & Paravina, 2022) A caracterização mecânica das resinas é muito importante e tem grande relevância clínica para compreender o comportamento biomecânico durante a função mastigatória causadas por forças oclusais. (Pontes *et al*., 2013; Rosatto *et al*., 2015; Cidreira *et al*., 2019) Propriedades como resistência à tração, dureza e módulo de elasticidade são propriedades mecânicas previstas que expressam a capacidade das resinas compostas de resistir as forças oclusais em dentes posteriores. (Pontes *et al*., 2013; Oliveira *et al*., 2022)

No conhecimento do autor, não há relato na literatura sobre a avaliação do desempenho óptico e mecânico das resinas compostas universais em comparação com uma resina composta convencional. Considerando a relevância clínica do processo de simplificação de procedimentos restauradores e a literatura limitada sobre as propriedades mecânicas e ópticas de resinas monocromáticas, o objetivo deste estudo foi avaliar a correspondência de cores, propriedades mecânicas, e tensão residual de resinas monocromáticas em comparação com uma resina convencional. As hipóteses nulas foram de que o tipo de resinas, monocromáticas ou convencionais: 1) não influenciaria nas propriedades mecânicas, contração pós-gel e tensão residual da restauração posterior; 2) não influenciaria o

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desempenho óptico expresso pela correspondência de cores, percepção de cores de restauração e a luz transmitida através das resinas.

2. CAPÍTULO 1 ARTIGO 1

Mechanical and optical characterization of single-shade resin composites used in posterior teeth

*Artigo a ser enviado para o periódico Operative Dentristy

Mechanical and optical characterization of single-shade resin composites used in posterior teeth

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Running title: Characterization of resin composites single-shade.

Keywords: Resin composite single-shade; mechanical properties; color; stress.

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Mechanical and optical characterization of single-shade resin composites used in posterior teeth

Abstract

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CLINICAL RELEVANCE: Single-shade RBCs demonstrated appropriated physical, mechanical and optical properties. They can be considered as an option for restoring posterior teeth for public and private services, sparing clinicians from purchasing multi shades RBCs.

INTRODUCTION

The demand for resin based composites (RBCs) with better esthetic and also less time-consuming procedures has increased.^{1,2} New formulations of RBCs provide better mechanical properties and make the restorative procedure more reliable and predictable. 3 However, the patient satisfaction is not only filled with mechanical performance of the restoration, they tend to focus initially more on the esthetic parameters.⁴

The human tooth is polychromatic, which makes the colors selection for RBCs restoration a challenging procedure for clinicians.⁵ Factors that influence the color adjustment potential of RBCs are classified into three categories: (1) material type, (2) cavity design, and (3) the substrate surrounding the restoration. Each factor can be optimized to enhance color matching and the restorative outcome.⁴ Tooth color determination is related to several factors such as lighting conditions, characteristics such as translucency, opacity, light dispersion and brightness.⁶ Enamel permit greater light transmission than dentin due its highly mineralized prismatic structure, low organic content and small amount of water, being more translucent substrate.⁷ Dentin has a lower mineral content, an organic tubular structure and greater amount of water, being less translucent when compared to enamel.⁷

Restorative procedures using RBC are commonly performed using the layering technique. $^{\rm 8,9}$ This is a complex procedure that incorporates the difficulty for choosing the shade and thickness of each increment, especially in more extensive 4

Natural teeth have complex shade variations, which led to the manufacture of RBCs in different shades. 10 RBCs are also available in different opacities, higher opacity RBCs are called dentin or body and lower opacity are called enamel or translucent, the combination is important to mimic the optical characteristics the tooth substrate. Clinicians should also consider characteristics such as value, hue, and chroma, in addition to other optical properties, such as translucency, opacity, and surface gloss. $^{\rm 11}$

The recent technology launched RBCs in the dental market involves universal, also called single-color or single-shade RBCs. Single-shade RBCs have a "chameleon" effect that can mimic the color of the tooth structure. These

RBCs are able to interact with the surrounding color of enamel and dentin, mimicking the optical characteristics.¹² Simplified restorative techniques allowed the clinician to reduce office time and also minimize errors related to color selection, the latter being dependent on environment and operator variations.¹³

The success of dental restorations is attributed to their esthetic and mechanical properties.^{4,14} The mechanical characterization of RBCs is very important and has great clinical relevance to understand the biomechanical behavior during function of mastication caused by occlusal forces.^{3,15,16} Properties such as tensile strength, hardness and elastic modulus are predicted mechanical properties that express the ability of the RBCs to withstand more occlusal forces in posterior teeth. 16,17

On the best of the author's knowledge, there is no report in the literature regarding the evaluation of the optical and mechanical performance of singleshade compared with conventional RBC. Considering the clinical relevance of the process of simplifying restorative procedures and the limited literature on the mechanical and optical properties of single-shade RBCs, the aim of this study was to evaluate the correspondence of colors, mechanical properties, shrinkage and residual stress of single-shade s compared to a conventional RBC. The null hypotheses were that the type of RBCs, single-shade or conventional: 1) would not influence the mechanical properties, post-gel shrinkage and residual stress of posterior RBC restoration; 2) would not influence the optical performance expressed by color matching, restoration color perception and the light transmitted through the RBCs;.

METHODS AND MATERIALS

The materials used in this study are shown in Table 1. Two single-color composite resins were used: Omnichroma (Tokuyama, Tokyo, Japan) and Vittra Unique Aps (FGM, Joinville, SC, Brazil) and an A2B-colored resin, Filtek Z350 (3M Oral Care, St Paul, MN, USA).

Light transmission - Beam profiling

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The light transmitted through 2.0 mm thick RBC was measured during real-time polymerization. Specimens were prepared using a 2.0 mm thick, 6.0 mm diameter mold. A mylar strip was used between the mold and a glass plate, the RBC was inserted into the mold, then another mylar strip of was used over the top of the RBC. The dental light-curing unit (LCU) tip was placed on the top side of the RBC and the transmitted light beam was examined on the other side using a profile camera with a 50 mm focal length lens (SP928, Ophir-Spiricon, Logan, UT, USA) with two blue filters (HOYA UV-VIS colored glass bandpass filter, Edmund Industrial Optics, Barrington, NJ, USA) and two neutral density filters (1.0 and 3.0, Edmund Optics). They were used to flatten the spectral response of the CCD camera. All materials were light cured using VALO Grand LCU (Ultradent). To capture the images during the 20 seconds of light curing, the number of frames and the exposure time were calculated with the same scale.

Light transmission – Integrating sphere

A 6-inch integrating sphere (Labsphere, North Sutton, NH, USA) connected to a fiber optic spectrometer (USB 4000, Ocean Insight, Largo, FL, USA) was used to measure total radiant power (mW) and irradiance, spectrum (mW/cm2/nm) of the LCU and the light transmitted through samples of the three RBCs with a thickness of 2 mm. The test started with the uncured sample and data was captured during the 20 seconds of light curing. The LCU light tip was placed into the 8 mm diameter inlet of the integrating sphere and all light from the LCU tip was captured. The measurement system, composed of the spectrometer, optical fiber and integrating sphere, was calibrated before use.

Color matching - spectrophotometer

The lower first molars of the prototype with class I cavity (# 6 mm in diameter and # 3mm in depth) produced in acrylic resin (P-Occlusal, São Paulo, SP, Brazil) were made in colors A2, B2, C2 and D3 (Figure 1A) . The color measurement of the models was performed with a visible/ultraviolet reflection spectrophotometer (Ci64UV, X-Rite, Grand Rapids, MI, USA) with an aperture diameter of 4 mm and the readings were carried out with a 2° observer angle

and illuminant D65. The color system established by the Commission Internationale de L'Eclairage (CIE), which is based on the dimensions L^* (white to black), a* (red to green), and b* (yellow to blue) were recorded. The tooth color was measured at baseline and 24 hours after restoration, at the same position for both measurements. Three RBCs were used to restore teeth, being 2 single-shade RBCs: Omnichroma (Tokuyama, Tokyo, Japan) and Vittra Unique APS (FGM, Joinville, SC, Brazil) and one multi-shade RBC, control group, Filtek Z350XT (shade A2, 3M Oral Care, St Paul, MN, USA). Teeth were restored using a single increment (6 mm in diameter x 2mm in depth) with a flat surface (Figure 1B, 1C and 1D). Restorations were light-cured using VALO Grand (Ultradent, South Jordan, UT, USA for 20 s).

Simple specimens of composites were made using a plastic matrix with 6-mm of internal diameter and a depth of 2 mm ($n = 5$). The composites were covered with a polyester strip before the light-curing which was made using the same photoactivation protocol described above. Color coordinates were read with the specimens placed against a white background (Color Checker grayscale, X-Rite, Grand Rapids, MI, USA - L*white = 95.2, a*white = 21.2, $b*$ white = 50.3).

The CAP (color adjustment potential) of the single-shade composites Vittra Unique APS (FGM, Joinville, SC, Brazil) and Omnichroma (Tokuyama, Tokyo, Japan) and multi-shade composite (Control group, Filtek Z350XT, shade A2, 3M Oral Care, St Paul, MN, USA) was measured by the following formula:

$$
CAP = 1 - (\Delta E^{\text{ restored}}/\Delta E^{\text{simple}})
$$

Where ΔE simple refers to the color difference between unrestored tooth (shades A2, B1, C2, and D3) and single-shade composites simple specimen. And ΔE restored is the color difference between CI I restored with a single-shade composite and unrestored tooth color. The CIELAB color difference (∆E simple and ΔE^{restored}) equation was calculated as follows: ∆E= (∆L² + ∆a² + ∆b²)^{1/2} where ∆L^{*}, ∆a^{*}, and ∆b^{*} refers to lightness, green-red, and blue-yellow differences.

The CIEDE2000 color difference was calculated with the following equation: ΔE_{00} = [($\Delta L/K_L S_L$)² + ($\Delta C/K_C S_C$)² + ($\Delta H/K_H S_H$)² + R_T ($\Delta C/K_C S_C$) $(\Delta H/K_H S_H)$] ^{1/2}. Where $\Delta L'$, $\Delta C'$, and $\Delta H'$ are the changes in luminosity, chroma, and hue, respectively. SL, SC, and SH are the weighted functions for each component. KL, KC, and KH are the weighted factors for Lightness, Chroma, and Hue, respectively (KL = $KC = KH = 1$). RT is the interactive term between chroma and hue differences.

Knoop Microhardness (KH)

Knoop hardness (KH, N/mm²) was performed on the top and bottom of RBC specimens after 24 hours of light curing, after measuring the degree of conversion. Before reading the Knoop hardness, the samples were polished with diamond metallographic pastes of 6, 3, 1 and 0.25 μm (Arotec, São Paulo, SP, Brazil). The RBC specimens ($n = 10$) were placed in a microhardness tester (FM700; FutureTech Corp., Kawasaki, Japan) and ten measurements were taken at different positions (ten indentations were made in the middle of each surface with an interval of 1 mm between them to obtain an average value) on the upper and lower surfaces, applying a load of 50 gf for 15s.

Degree of convertion - Raman spectroscopy

A LabRam HR Evolution Raman spectrometer (Horiba LabRam, Villeneuve d'Ascq, France) was used operating at an excitation power of 20 mW with radiation emitted by a He-Ne laser (633 nm). The Raman signal was acquired using a 600 lines/mm grid centered between 1000 and 2000 cm^{-1} with a confocal hole of 400 μm. OriginPro 7.5 software (OriginLab Corporation, Northampton, MA, USA) was used to analyze the acquired Raman data. An initial spectrum was collected for each uncured RBC. Ten samples from each group were analyzed to calculate the degree of conversion. The aromatic peak, observed at 1608 cm^{-1} , was used as the reference peak and the vinylic peak, observed at 1638 cm^{-1} , as the aliphatic peak. The degree of conversion was calculated using the ratio of the reaction to the internal reference peak areas as the ratio of polymerized to unpolymerized RBC. The formula used to calculate the degree of conversion was: DC $(\%) = (1 - P/NP) \times 100$.

Flexural strength (FS) and Elastic modulus (E)

A three-point bending test was performed to obtain the flexural strength (MPa) and Elastic modulus (MPa) of all groups. The specimens ($n = 10$) were made using stainless steel mold (25.0 mm \times 2.0 mm \times 2.0 mm) prepared in a dark room under yellow light and at temperature controlled (25 \pm 1^oC). The presence of bubbles was minimized and a smooth surface was obtained, placing a mylar strip between the glass plate and the mold. The RBCs were inserted into the mold and another mylar strip was placed over the top surface that was covered by a second glass plate. The RBC specimens were light activated for 20 s at three light exposures covering the entire length of the specimen using a multi-peak LCU (VALO Grand; Ultradent, South Jordan, UT, USA) with 916.19 mW/cm² checked using an integrating sphere. The LCU tip was positioned as closed as possible to the mylar strip and stabilized using a metallic support.

The test was performed using a universal testing machine (ElectroPuls™ E3000, Instron, High Wycombe, UK) and assembled using Bluehill software (Instron Training Center, Norwood, MA, USA) with a crosshead speed of 0.5 mm/min until failure occurred. The FS was determined using the formula: α = 3FL / 2w*t*, where F is the maximum applied force (N), L is the distance between the supporting beams (mm), w is the width of the specimen (mm), and *t* is the thickness of the specimen (mm). $^\mathrm{18}$

The E (MPa) was determined using a bending deflectometer (W-E401-J, E-Series Deflectometer, Instron, Norwood, MA, USA). During the test, the tip of the deflectometer was positioned at the center of the specimen bottom, measuring the deflection during the load application. The E was determined using the following formula: $E = FL^3 / 4BH^3d$, where F is the maximum load (N), L is the length of the specimen (mm), B is the width of the specimen (mm), H is the height of the specimen (mm) and d is the deflection (mm) corresponding to the load F. 18

Compressive strength (CS) and diametral tensile strength (DTS) calculation

Compressive and diametral tensile strengths ($n = 10$) of RBCs were calculated. The RBC was inserted into a cylindrical metallic mold for the compressive strength test (6 mm height, 3 mm diameter) or the diametral tensile strength test (2 mm height, 4 mm diameter). The specimens were light activated in three 2.0 mm increments by using VALO Grand (Ultradent). The specimens were stored for 24 h at 37°C, afterwards were submitted to compressive strength and diametral tensile testing in a universal testing machine (ElectroPuls™ E3000, Instron) at a crosshead speed of 0.5 mm/min until failure occurred. Compressive strength values were calculated by dividing the fracture load (F) by the cross-sectional area and converting it into MPa. Diametral tensile strength values were calculated using the equation: DTS = $2F/\pi dt$, where *d* is the specimen diameter, and *t* is the height of the specimen. DTS and CS values were converted into MPa.

Post-Gel Shrinkage

Post-gel shrinkage (Shr) was calculated using the strain-gauge test method.¹⁹ The tests were performed in a dark room with yellow light and temperature control (22 \pm 1 °C). The materials were cast into hemi-spheres on a biaxial strain gage (Excel sensors, Embú, São Paulo, Brazil) that measured shrinkage stresses in two perpendicular directions. A strain conditioner (ADS05000IP, Lynx Tecnologia Eletrônica, São Paulo, Brazil) converted electrical resistance variations in the strain gage into voltage variations through a quarter-bridge circuit with an internal reference resistor (120 Ω). The strain values measured along the two axes were calculated, since the material properties were considered homogeneous and isotropic on a macroscale. All materials were light cured using an LED LCU (VALO Grand; Ultradent, South Jordan, UT, USA) with the light tip held at a distance of 1 mm from the surface of the RBC. Stress values were collected for five minutes after the sample was photoactivated. The five minute maximum shrinkage stress, moment that expressed the stabilization of the post-gel curves, was used as the post-gel linear shrinkage and converted to volumetric percentage by multiplying by 3 and 100%.

Scanning electron microscopy

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To evaluate the filler content, 5 disc-shaped specimens of each RBC were prepared using a metallic mold with 2 mm thick and 4 mm in diameter. The samples were light cured for 5s each, using the VALO Grand LCU (Ultradent). The RBCs disc was immersed for 1 week in 2 mL of acetone, which was changed daily. The filler content extracted from each specimen was fixed on metallic stubs. The samples were metallized by a sample coating equipment (Leica EM SCD050, Wetzlar, Germany) and observed with a scanning electron microscope (Zeiss EVO MA10, Oberkochen, Germany). Representative areas showing filler particles were photographed at 10000x magnification. 20

Shrinkage stress and residual stress - Finite Element Analysis

Shrinkage and residual tooth stress were calculated using a twodimensional finite element analysis. A MOD cavity of a maxillary premolar was created. The geometric model was based on a buccolingual cross-sectional model. A simplified boundary condition was applied to the root cutting plane (fixed zero offsets in the horizontal and vertical directions). The modulus of elasticity of enamel was 84 GPa (main direction, perpendicular to the pulp surface) and 42 GPa in transverse directions, with a Poisson ratio of 0.30. The dentin elastic modulus was 18 GPa and Poisson's was 0.23. An oblique layering of the composites was performed. For the simulation of polymerization shrinkage and modulus development, three mechanical properties were required: the linear shrinkage coefficient (α), Poisson's Ratio (u) and the elastic modulus. The elastic modulus of the three RBCs were obtained from experimental data. The Poisson ratio was chosen to be the same for all composites at 0.24.²¹ Polymerization shrinkage was simulated by thermal analogy, in which a virtual temperature change in the composite is used to create material shrinkage. Using this method, the linear contraction value was entered as the coefficient of linear thermal expansion and the temperature was reduced by one degree. Finite element stress analysis was performed using MSC.Mentat (pre- and post-processor) and MSC. Marc Software (solver) (MSC Software; Santa Ana, CA, USA). Modified von Mises equivalent stresses were used to express the stress conditions. Modified von Mises equivalent stress was

used to express the stress conditions using compressive-tensile strength ratios for enamel 37.3 and dentin 3.0. 22

The compressive-tensile strength ratios for each RBC was calculate experimentally. Stress values were recorded at the integration points of each element and at nodes along material interfaces in any aspect. Mean values of the highest 5% stresses were determined for enamel, dentin and composite structures and for enamel/composite and dentin/composite interfaces.

Statistical Analysis

The KH, DC, Shr, FS, E, DTS, CS, color difference, and CAP data were tested for normaldistribution (Shapiro–Wilk) and equality of variances (Levene's test), followed by parametric statistical tests. One-way analysis of variance (ANOVA) was performed for each mechanical property. KH and DC data were analyzed using one-way analysis of variance (ANOVA) with repeated measurement. Multiple comparisons were made using Tukey' test. Color outcomes were analyzed using two-way ANOVA. Pair-wise comparisons were performed with Tukey's test. Also, One-way ANOVA and Dunnett's test were to compare the control group (multi- shade RBC) with experimental groups (single-shade RBC). All tests employed α = 0.05 significance level and all analyses were carried out with the statistical package Sigma Plot version 13.1 (Systat Software Inc., San Jose, CA, USA). The modified von Mises stresses values, light transmission, and the SEM imagens were analyzed qualitatively.

RESULTS

Light transmission - Beam profiling

VALO Grand beam profiles distribution across the no RBC light tip (control) and across 2.0 mm of all RBCs are reported in Figure 3. The light was homogeneously distributed across the LCU tip (control). The light transmitted through the RBCs decreased significantly. The beam profiles distribution across the 2.0 mm specimen of all RBCs during the light curing process are reported in Figure 4. The Omnichroma (Figure 4A) and Vittra Unique APS (Figure 4B) increased the light transmission during the polymerization process, on the other hand the Filtek Z350XT showed almost the same light transmission during the light curing activation process (Figure 4C).

Light transmission – Integrating sphere

The spectral radiant powers emitted by VALO Grand LCU and through the RBCs are shown in Figure 5. The LED LCU delivered 550 mW and the radiant power decreased significantly through the RBCs (*P* <0.001), reducing to 85 mW (15%) through the 2.0 mm thick Vittra Unique APS, 78 mW (14%) through the Omnichroma and 25 mW (5%) through the Filtek Z350XT. The single-shade RBCs tend to increase the light transmission during the polymerization process (Figure 5B).

Color matching - spectrophotometer

Color difference between restored and unrestored teeth according to shade (A2, B2, C3 and D3) and single-shade composites are displayed in Table 4. Two-way ANOVA showed that ΔEab and ΔE00 were affected by both factors single-shade RBCs (*P* <0.001) and tooth shade (*P* <0.001), as well as by the interaction between the factors (*P* <0.001). Omnichroma showed less color differences than Vittra Unique APS, regardless of the RBC shades. However, the color difference increased toward more chromatic tooth (from A2 to D3) for both outcomes (ΔEab and ΔE00). All single-shade RBCs showed greater color variation than the control RBC.

Regarding the values of CAP, the factors 'single-shade composite' (*P* <0.001) and 'tooth shade' (*P* <0.001) affected the results, but not the interaction between these factors (*P* =0.054) (Figure 2).

Irrespective of the tooth shade, the highest PAC was observed to singleshade composites when the Omnichroma was used. When the tooth was restored with Vittra Unique APS in B2 and A2 tooth shades and with Omnichroma in C3 tooth shade, a similar PAC was observed compared to the control group. The Omnichroma used to restore A2, B2 and D3 tooth shade showed higher PAC than the control RBC, and Vittra Unique APS the lower in C3, and D3 tooth shade.

Mechanical properties

The KH and DC mean values and standard deviations of three RBCs measured at top and bottom are shown in Table 2. ANOVA repeated measurement showed a significant difference between the RBC factor (KH *- P* <0.001; DC*- P* <0.001), for location of measurement factor (KH *- P* <0.001; DC*-P* <0.001), however no significant difference for interaction between RBC and location of measurement factors (KH - *P* =0.413; DC - *P* =0.126). Vittra Unique APS had significantly higher DC than Omnichroma and Filtek Z50XT. Vittra Unique APS and Filtek Z50XT had similar and significantly higher KH values than Omnichroma. The KH and DC were significantly higher at the top than the bottom irrespective of RBCs (*P* <0.001); however, the variation of KH and DC values were always higher than 80% for tested parameters and RBCs.

The FS, E, Shr, DTS, CS and CS/DTS ratio mean values and standard deviations of three RBCs are shown in Table 3. ANOVA showed a significant difference between the RBC for E (*P* <0.001), DTS (*P* =0.008), CS (*P* <0.001) and CS/DTS ratio (*P* <0.001). However, ANOVA showed no significant difference for FS (*P* =0.083) and Shr (*P* =0.144). Omnichroma had significantly lower and Filtek Z50XT significantly the highest E value. Both single-shade RBCs had similar DTS value and significantly lower than Filtek Z50XT. Vittra Unique APS and Filtek Z50XT had similar and significantly lower CS and CS/DTS ratio values than Omnichroma.

Stress analysis

The stress distributions during restoration (modified von Misses stress) are shown in Fig. 6 A, B and C. The use of the single-shade RBCs resulted in similar shrinkage stress in the tooth structure during the restoration than when conventional RBCs inserted incrementally were used.

The stress distributions at 100N occlusal loading (modified von Misses stress) are shown in Fig. 6 D, E and F. The use of the single-shade RBCs resulted also in similar shrinkage stress in the tooth structure during the occlusal loading than when conventional RBCs inserted incrementally were used. The Filtek Z350XT resulted in little higher shrinkage stress than both single-shade RBCs during the restoration and at 100N occlusal loading.

DISCUSSION

The single-color composite resins showed satisfactory results, similar or superior to the conventional resin in most of the mechanical tests, post-gel shrinkage and residual stress, therefore the first null hypothesis was rejected regarding the mechanical properties, except in hardness, modulus of elasticity and diametrical traction. However, the second null hypothesis about the optical performance was rejected, since the single-shade RBCs showed higher color variation than the control.

RBCs have been extensively studied and improved over time.²³ In this study, new RBCs, named single-shade RBCs, present recently in the market were evaluated and compared to a consolidated nanoparticle RBC. Flexural strength is correlated with composite wear. $24,25$ The standard test for flexural strength analysis is the three-point bending test. 26 In the present study, there was no significant difference between RBCs regarding the flexural strength, all tested RBCs had above the allowed value of 80 MPa.²⁶

The E values expresses capacity of the accumulation of stresses at the interface between the tooth and the restorative material, which are generated by polymerization shrinkage of the RBC and cusp deflection.²⁷ Filtek Z350XT showed higher elastic modulus compared to single-shade RBCs. Polymerization shrinkage values are directly influenced by the amount of organic and inorganic content of the RBCs. 28 This result is linked to the high filler content of Filtek Z350XT compared with both single-shade RBCS. The amount of filler content plays an important role in mechanical properties and wear resistance, and also influences the shrinkage stress of the RBC. 29 The benefits of high filler content are enhanced mechanical and physical properties, decreased thermal expansion and shrinkage, and improved workability.^{29,30} In contrast, lower filler content causes greater shrinkage in the RBCs. 31 The RBCs analyzed in this study have silica, zirconia (Filtek Z350XT and Omnichroma) and glass-aluminum-silicate in their compositions e a Vittra Unique APS boronaluminium-silicate glass.

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One of the consequences of the polymerization of RBC is the volumetric contraction. The volumetric contraction is a consequence of the polymerization of RBC during the formation of a polymeric network, causing volumetric changes.³² There was no statistical difference between RBCs for post-gel shrinkage of all tested RBCs. The similar performance of all RBCs is due the similarity balance created on the monomer types and filler content of these materials. The manufactures of different RBCs has different strategies to formulate the RBCs, however in this study we can confirm that all tested materials presented low post-gel shrinkage and also E values with values that allowed the deformation during the shrinkage process, reducing the shrinkage stress generated.

Another important factor to consider when evaluating the RBC is the degree of conversion (DC). It directly influences the mechanical and physical properties and also the resistance to RBC degradation over time.³³ The DC is influenced by the RBC composition, expressed by organic components, initiator concentration, type, size and amount of filler particles, color, degree of translucency/opacity and also by factors related to photoactivation.^{34,35,36,37} A low DC can result in reduced mechanical properties such as wear resistance, as well as decreased color stability, increased water sorption and chance of secondary caries.³⁸ In this study, we used Raman spectroscopy to determine the degree of conversion and the result showed that Filtek Z350XT, which is a nanoparticulate RBC, showed the lowest DC value at the top and bottom than single-shade RBCs. Vittra Unique APS showed the highest DC value when compared to Filtek Z350XT and Omnichroma. There are no reports in the literature comparing Vittra Unique APS. In a previous study, Omnichroma RBC was evaluated for the effect of repeated heating and cooling and showed that without heating, similar values were found as in the present study. 39 Probably the higher DC values are related to higher translucency and consequently higher light that reach the entire volume of the material determining more energy for polymerization process.

In our findings, the analyzed erythrocytes have similar percentages of filler particles, but a statistical difference was found in relation to Knoop hardness. The polymeric mechanical properties depend on the initial

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composition and the polymerization network created with RBC polymerization.⁴⁰ The most popular base monomer used is Bis-GMA with a molecular weight (MW) of 512 g/mol. The compound comprises a rigid core of bisphenol A and hydroxyl groups capable of forming strong hydrogen bonds.⁴¹ Bis-EMA has a similar structure to Bis-GMA, being based on a rigid bisphenol A core; however, since it lacks two pendant hydroxyl groups and has longer ethoxylate bonds, Bis-EMA (MW = 540 g/mol) is more flexible with lower viscosity than Bis-GMA. Therefore, it demonstrates a higher overall conversion.⁴² The TEGDMA (MW = 286 g/mol) is a flexible and low viscosity dilute monomer, which is used to achieve a higher degree of conversion and homogenization of the filler particles. Importantly, the final properties result not only from the characteristics of the individual monomers, but also from the interactions that arise in the mixing of the monomers and the characteristics of the resulting polymer network. 43 Filtek Z350XT has combination of Bis-GMA, UDMA, Bis-EMA and TEGDMA, while Omnichroma and Vittra Unique APS features UDMA and TEGMA. This combination may explain the higher degree of surface hardness attributed to the Filtek Z350XT.

Two-dimensional beam profiling is a technique used to characterize the beam homogeneity of dental light-curing units.⁴⁴ In this study, the single-shade Vittra Unique APS and Omnichroma RBCs showed higher light transmission during the photoactivation process indicating that these materials become more translucent during light activation when compared to Filtek Z350XT. The transmission of the light through the integrating sphere confirmed that there was a trend of single-shade RBCs towards greater translucency during the polymerization process. There are no reports in the literature that analyzed this methodology to compare with this type of RBC. However, the Filtek Z350XT showed stable light transmission through during the light polymerization process, demonstrating that this is a mechanism that can explain the capacity of the color matching verified for single-shade RBCs.

The stress analysis was performed using finite element analysis. The stress is determined by the strength properties of the material, typically the interaction between elastic modulus and post-gel shrinkage. ⁴⁵ Different location and moments were considered for analyzing the shrinkage stress in the tooth structure during restoration and also the residual stress during occlusal loading. The single-shade and conventional RBCs tested showed similar performance, demonstrating properly balance between elastic modulus and post-gel shrinkage.

Tensile strength is examined indirectly by the diametral tensile strength property. The tensile strength value is directly linked with the filler content of the materials. ⁴⁶ The diametral tensile strength test may reveal different values for apparently similar materials. However, this variation has been explained by the difference between the polymer matrix, size of the fillers, and bonding between the fillers and the matrix.⁴⁷

In this study, it was possible to observe that after polymerization, the composite Omnichroma becomes more translucent, boosting its capacity to color adjustment. Furthermore, pigments, sizes, and forms of filler particles used in the composition of the RBCs have an effect on the color of the materials.⁴ They selectively reflect certain bands of light wavelengths altering light propagation which contributes to the color of the material. The Omnichroma has been developed with smart chromatic technology, with supranano-spherical fillers of silicon dioxide (SiO2) and zirconium dioxide (ZrO2) that produce a red-to-yellow color, making it easier to match the color of teeth⁴⁸. This characteristic was confirmed by SEM analysis performed in this study (Fig 7). When teeth with A2 or B2 shades were restored, Vittra Unique APS the CAP values were similar to those of the control group (multi-shade composite), which was also reported in a prior study. 49 The great translucency of Vittra Unique APS tend to be caused by the RBC matrix being more important than its color-shifting capabilities.

The increases in the ΔEab and ΔE00 with a decrease in tooth shade value from A2 to C3 showed that these single-shade RBCs can better matches the color of the teeth with higher shade values. It also appears that when a grey component of color is added to the tooth, the color differences increase (VITA shades C and D). However, these data may imply that for teeth with low value and high esthetic needs, a multi-shade composite system is preferable to achieve the best potential esthetic result.⁵ Another parameter that must be evaluated is the perceptibility threshold. The use of acrylic teeth increases the

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color difference values greater than the clinical threshold (2.66 to ΔEab and 1.77 to ΔE00).⁵⁰ However, Omnichroma and Vittra Unique APS used in A2 shade teeth was the only RBC that came closest to this limit possibly due to the characteristics described above.

Single-shade RBCs demonstrated properly mechanical and optical properties that make them suitable and safety for restoring posterior teeth. It is important to emphasize that these materials can replace conventional RBC of multiple colors, reducing the purchased materials, and indirectly reducing the capacity of expiration of the material, that is very frequently in private and public dental health services. Also, the absence of shade selection and the multiple layering shades and opacities can simplify the restorative process reducing the time. However, when high esthetic demand are present the clinicians should considered the use of multi-shade RBCs.

CONCLUSION

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

- Single-shade RBCs, Omnichroma and Vittra Unique APS, have similar or superior mechanical properties to the conventional resin tested;
- The shrinkage and residual stress was similar for posterior teeth restored with single-shade RBCs, Omnichroma and Vittra Unique APS, or conventional Filtek Z350XT;
- Single-shade RBCs increased light transmission during the photoactivation process;
- Vittra Unique Aps tends to perform better shade matching for B2 and A2 tooth shades than C3 and D3 tooth shades.

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TABLES

Table 1. Resin composites composition.

dimethacrylate; TEGDMA,triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Resin composite	Knoop Hardness $(N/mm2)$			Degree Conversion (%)		
	Top	Bottom	Variation Top		Bottom	Variation
Filtek Z350 XT	77.1 ± 2.1^{Aa} 61.9 \pm 3.5 ^{Ab} 87%				57.4 \pm 3.7 ^{Ba} 49.0 \pm 6.0 ^{Bb} 85%	
Vittra Unique APS 66.1 ± 4.5^{Ba} 57.0 ± 2.1^{Bb} 86%					72.4 ± 3.6 Aa 68.2 \pm 2.6 ^{Ab} 94%	
Omnichroma		59.8 ± 5.7 ^{Ca} 52.8 ± 3.5 ^{Cb} 89%			56.9 ± 3.6 Ba 46.3 ± 2.6 Bb 81%	

Table 2. Experimentally determined mean (and standard deviation) Knoop hardness and degree of conversion on top and bottom of tested RBCs.

Different letters mean significant difference; upper caser letter used for comparison between RBCs, and lower caser letter for comparison between location for each mechanical property provided by Tukey test (*P* <0.05).

Table 3. Experimentally determined mean (and standard deviation) flexural strength, elastic modulus, volumetric post-gel shrinkage, compressive strength, diametral tensile strength and compression/tensile strength ratio.

Different letters mean significant difference between RBCs for each mechanical property provided by Tukey test (*P* <0.05).

Table 4 - Color differences (± standard deviation) between restored and unrestored teeth according to shade and RBCs.

For each outcome (ΔEab and ΔE00), different letters (uppercase comparing shades, lowercase comparing RBCs) indicate statistical difference at Tukey test (*P* <0.05). *Indicate statistically significant difference between each experimental group compared with control group (Dunnett's test, p<0.05).

Figures

Figure 1. A. Color. A. Teeth without restoration; B. Teeth with Omnichroma restorations; C. Teeth with Vittra Unique APS restorations; D. Teeth with Filtek Z350XT restorations.

Figure 2. Means (± standard deviation) of color adjustment potential of the composites according to the tooth shade. Different letters (uppercase comparing single-shade RBCs and lowercase comparing tooth shades to each RBC) indicate statistical difference at Tukey test (P <0.05). *Indicate statistically significant difference between each experimental group compared with the control group (Filtek Z350XT, shade A2, by Dunnett test, *P* <0.05).

Figure 3. Two-dimensional and three-dimensional beam profile images in the same scale captured with no RBC light tip (control) and across 2.0 mm of all RBCs. All materials are showed in the same scale compared to the VALO Grand beam profile image through the 60-degree screen.

Figure 4. Three-dimensional beam profile images in the same scale captured across 2.0 mm of all RBCs during the light activation process. A. Omnichroma, Tokuyoma; B. Vittra Unique APS; C. Filtek Z350XT, 3M Oral Care.

Figure 5. Radiant power (mW) emitted during 20 s; A. VALO Grand on standard mode; B tested through specimens of the RBCs. Note the image control are in the different scales.

Figure 6. Modified von Mises stress distribution generated by shrinkage during the restoration and at occlusal loading for different RBCs tested at the maximum intercuspidation of the molar teeth.

Figure 7. SEM images of tested RBCs: A. Omnichroma; B. Vittra Unique APS; C. Filtek Z350XT (10 000 X magnification).

Anexo

Link para acesso as normas da **Operative Dentistry**:

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