

UNIVERSIDADE DE UBERABA
MESTRADO ACADÊMICO EM ODONTOLOGIA

LARISSA MARTINS DA COSTA

EFEITO DE DENTIFRÍCIOS CLAREADORES NA RECUPERAÇÃO DA COR E
NA RUGOSIDADE SUPERFICIAL DE RESINAS COMPOSTAS
NANOPARTICULADAS MANCHADAS COM VINHO TINTO

UBERABA – MG
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Dissertação apresentada ao Programa de Pós-Graduação em Odontologia- Mestrado Acadêmico da Universidade de Uberaba, como requisito para obtenção do título de Mestre em Odontologia, na área de concentração em Clínica Odontológica Integrada.

Orientador: Prof. Dr. Vinícius Rangel Geraldo Martins

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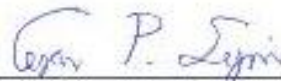
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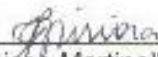
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DEDICATÓRIA

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RESUMO

RESUMO

O objetivo deste estudo foi avaliar o efeito de dentifrícios clareadores na recuperação da cor e na rugosidade da superfície de resinas compostas nanoparticuladas submetidas ao manchamento com vinho tinto. Foram obtidos quarenta discos de cada compósito nanoparticulado (Filtek™ Z350 XT e Filtek™ One Bulk-Fill). A leitura de cor inicial foi realizada através do Espectrofotômetro Vita Easyshade Compact Advanced (CIELab) e a rugosidade da superfície com microscopia confocal de varredura a laser (LEXT 4000, Olympus Co., Hamburg, Germany). Posteriormente, as amostras foram manchadas com vinho tinto, e tiveram sua cor e rugosidade superficial reavaliadas. Após o manchamento, os espécimes foram divididos em 8 grupos (n = 10): G1 a G4 foram compostos por amostras da resina composta Z350 e G5 a G8 foram compostos por amostras da resina Bulk Fill. Em seguida, as amostras passaram pelo desafio abrasivo, onde: G1 e G5 foram escovados com água destilada (AD); G2 e G6 Colgate Máxima Proteção Anticáries (CMPT), G3 e G7 o Colgate Luminous White Advanced (CLWA) e G4 e G8 com o Curaprox Black is White (CBW). A escovação foi realizada com escova de dentes elétrica (1,96N; 30 minutos). A cada 30 segundos 1,0 mL do slurry foi injetado entre as cerdas da escova e a amostra. Após o ensaio de escovação, as amostras tiveram sua cor e rugosidade reavaliadas. A diferença de cor (ΔE) entre cada fase foi analisada pelo ANOVA seguido pelo pós teste de Tukey ($\alpha = 5\%$). Os resultados mostraram que a resina Z350 foi mais suscetível ao manchamento do que a resina Bulk Fill. Exceto no grupo 3 (CLWA), todas as alterações de cor permaneceram abaixo de 3,3 após a escovação. O desafio abrasivo não foi capaz de recuperar a cor dos compósitos, mas alterou a rugosidade da superfície dos compósitos. Concluiu-se que os dentifrícios clareadores não conseguiram remover a mancha de vinho tinto dos compósitos nanoparticulados, mas aumentaram a rugosidade da superfície desses materiais dentários.

PALAVRAS- CHAVE: Resina Composta, Dentifrícios Clareadores, Rugosidade, Cor.

ABSTRACT

ABSTRACT

The aim was to evaluate the effect of whitening toothpastes on color recovery and surface roughness of red-wine stained nanofilled composite resins. Forty discs of each nanofilled composite (Filtek™ Z350 XT and Filtek™ One Bulk-Fill) were obtained. The baseline color Spectrophotometer Vita Easyshade Compact Advanced (CIELab) and surface roughness LEXT 4000, Olympus Co., Hamburg, Germany (confocal laser scanning microscopy) of resin discs were evaluated. Afterwards, samples were staining by red wine, had their color and surface roughness reevaluated, and were divided in 8 groups (n=10): G1 to G4 were formed by Z350 samples, and G5 to G8 were formed by Bulk-Fill samples. Subsequently, samples were brushed with the following dentifrices: distilled water (control); Colgate Maximum Cavity Protection (CMCP) (G2 and G6), Colgate Luminous White Advanced (CLWA) (G3 and G7) and Curaprox Black is White (CBW) (G4 and G8). Brushing was performed with an electric toothbrush (1,96N; 30 minutes). Every 30 seconds, 1.0 mL of the slurry was injected between the bristles of the brush and the specimen. After abrasive challenge, samples had their color and roughness reevaluated. The color difference (ΔE) between each stage were analysed by ANOVA and Tukey's test. The Two-Way ANOVA and Tukey's test were used to compare the ΔE obtained for baseline x after staining and baseline x after brushing. Two-Way ANOVA followed by post hoc Tukey's test analyzed the differences between the initial and final surface roughness of each group ($\alpha=5\%$). Results showed Z350 was more susceptible to staining than Bulk Fill. Excepting group 3 (CLWA), all color changes remained below 3.3 after brushing. No abrasive challenge was able to recover the color of the composites, but all abrasive challenges changed the surface roughness of the composites. It was concluded that whitening toothpastes failed to remove red wine staining from nanofilled composites, and the abrasive challenge increased the surface roughness of that dental materials.

KEY WORDS: composite resins, whitening dentifrices, roughness, color.

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1. INTRODUÇÃO

1. INTRODUÇÃO

O sorriso influencia diretamente no estado psicológico e na vida social de adultos jovens. Fatores como alinhamento e cor dos dentes são as principais queixas relacionadas a estética, aproximadamente 25% a 55% dos adultos são insatisfeitos com a cor dos dentes (DAUD A et al., 2018).

Existem vários fatores que podem causar o manchamento dos dentes. Essa condição pode ocorrer de maneira intrínseca ou extrínseca. As manchas extrínsecas são resultado do acúmulo de substâncias cromóforas exógenas que se aderem na superfície externa do dente devido à má higiene oral, uso de tabaco, ingestão de alimentos e bebidas cromatogênicas. Com o passar do tempo se essas manchas não forem removidas com a escovação de rotina ou profilaxia dentária, elas adquirem resistência e se difundem através do esmalte dental. Quando esse processo ocorre a mancha passa a ser intrínseca, não podendo mais ser removida pela escovação dentária ou profilaxia. (ABDALLAH et al., 2014).

Assim como os dentes naturais, materiais restauradores como as resinas compostas também podem ter sua cor alterada devido a exposição a alimentos e bebidas cromatogênicas. Essa suscetibilidade ao manchamento é a principal razão pela qual alguns cirurgiões-dentistas optam por restaurações cerâmicas em vez de restaurações diretas de resina composta. Devido a grande maioria dos pacientes esteticamente exigentes não aceitam alterações de cor de suas restaurações ao longo do tempo (ARDU et al., 2018). Um estudo realizado por D. Manojlovic et al em 2015, avaliou a alteração de cor em uma resina composta após a imersão em quatro bebidas diferentes: chá, café, Coca-Cola e vinho tinto. Foi observado que chá e o café alteraram o croma do material restaurador, enquanto o vinho tinto além de alterar o croma modificou a propriedade de valor da resina composta.

As resinas compostas são classificadas de acordo com o tamanho das partículas de carga presente no seu conteúdo, essa característica tem influência direta no desgaste do material, que ocorre de maneira semelhante ao esmalte e a dentina. Porém o desgaste promove alterações superficiais nos compósitos devido a lixiviação das partículas de carga, o que pode ser influenciado por alguns fatores, dentre eles: parafunções orais, e escovação com dentifrícios abrasivos (ALKHURAIIF et al., 2014). Uma pesquisa realizada por Roselino et al., em 2015, avaliou os efeitos da escovação com dentifrícios

abrasivos em uma resina composta nanoparticulada e foi comprovado que a escovação prolongada com dentifrícios abrasivos pode influenciar na alteração de cor do material.

No que diz respeito às características físicas dos materiais restauradores, a nanotecnologia foi introduzida na odontologia com o objetivo de incorporar nos materiais restauradores características que melhorem suas propriedades estéticas e mecânicas. A presença de nanopartículas proporcionam aos compósitos propriedades que os tornam superiores a resinas compostas micro- híbridas, como por exemplo um polimento mais eficaz, a capacidade óptica, permitindo assim uma mimetização da estrutura dental, além da resistência a abrasão (MANSOURI, ZIDANI et al., 2018).

Além da nanotecnologia, outras modificações nos materiais restauradores foram feitas dentre elas a introdução no mercado das resinas compostas bulk fill. Elas foram criadas com o intuito de simplificar procedimentos restauradores otimizando o tempo clínico gasto em restaurações diretas, possibilitando a técnica incremental única. Poucos são os estudos que avaliaram a estabilidade de cor e a rugosidade superficial desse material (ALZRAIKAT H et al., 2018).

Com o aumento da demanda da odontologia estética, houve um grande destaque de tratamentos que visam alterar a cor dos dentes de maneira conservadora e eficaz. Dentre as opções terapêuticas o clareamento dental tem apresentado grande destaque por ser uma técnica relativamente segura e precisa para resolver problemas como o manchamento dental. Existem diferentes métodos de clareamento dental. A técnica pode ser realizada através da remoção física de manchas extrínsecas ou por reação química de oxidação de pigmentos intrínsecos. (VIEIRA-JUNIOR et al., 2016).

No final da década de 80, principalmente nos Estados Unidos várias empresas introduziram no mercado produtos de clareamento dental para serem utilizados em casa que prometiam melhorias “milagrosas” na cor dos dentes. Dentre eles as fitas dentais clareadoras e os dentifrícios com ações clareadoras, que atualmente, têm apresentado grande destaque nas áreas de marketing comercial voltado a venda de produtos odontológicos de consumo caseiro (CLIFTON CAREY, 2014).

As pastas dentais clareadoras apresentam em sua formulação umectantes, surfactantes e alguns tipos de abrasivos como a sílica, carbonato de cálcio, bicarbonato de sódio, pirofosfato de cálcio, alumina entre outros materiais. Em algumas formulações estão presentes também componentes dos clareamentos dentais utilizados em consultório

como peróxido de hidrogênio que tem como objetivo prevenir e remover manchas extrínsecas presentes nas superfícies dos dentes. (TAO et al., 2017, JOINER 2010). Além desses oxidantes, alguns dentifrícios apresentam em sua composição polímeros e pigmentos que atuam de maneira química ou óptica. Ainda é desconhecido o efeito deles sobre a superfície de materiais restauradores (VAZ et al., 2019).

Recentemente, foi introduzido no mercado dentifrícios a base de carvão vegetal destinados a remoção de manchas extrínsecas e ao clareamento do dental. Esses produtos estão ganhando a popularidade por parte dos fabricantes e dos pacientes. O carvão atua sobre a superfície dental adsorvendo pigmentos e manchas responsáveis por alterar a cor dos dentes. Porém, existem poucas pesquisas sobre este material, ainda são desconhecidos os efeitos do seu uso sobre a estrutura dentária ou materiais restauradores. Pouco se sabe sobre o seu efeito clareador. (BROOKS, BASHIRELAHI, REYNOLDS 2017; VAZ et al., 2019).

Considerando a recente introdução da resina Bulk Fill nas práticas clínicas e o uso rotineiro de resinas compostas nanoparticuladas pelos cirurgiões dentistas, o consumo de bebidas cromatogênicas e a grande influência do marketing comercial sobre pacientes voltado ao uso de dentifrícios clareadores, ainda são escassos os estudos que avaliam o efeito desses dentifrícios clareadores sobre a superfície de resinas compostas como alterações de cor e da rugosidade superficial. Portanto este trabalho teve como objetivo avaliar *in vitro* o efeito de dentifrícios clareadores presentes no mercado sobre a superfície de resinas compostas nanoparticuladas submetidas ao manchamento artificial.

2. PROPOSIÇÃO

2. PROPOSIÇÃO

O objetivo deste estudo foi avaliar os efeitos de dentifrícios clareadores, sobre a superfície de duas resinas compostas nanoparticuladas previamente submetidas ao manchamento artificial. Foi avaliado a alteração de cor e a rugosidade superficial do material restaurador.

3. CAPÍTULO 1

3. CAPÍTULO 1

Effect of whitening toothpastes on color recovery and surface roughness of red wine stained nanofilled composite resins

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Short Title: Whitening dentifrices alter the surface of stained nanoparticulate composite resins

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Key words: composite resins, whitening dentifrices, roughness, color

4. ABSTRACT

The aim was to evaluate the effect of whitening toothpastes on color recovery and surface roughness of red-wine stained nanofilled composite resins. Forty discs of each nanofilled composite (Filtek™ Z350 XT and Filtek™ One Bulk-Fill) were obtained. The baseline color (CIELab) and surface roughness (confocal laser scanning microscopy) of resin discs were evaluated. Afterwards, samples were staining by red wine, had their color and surface roughness reevaluated, and were divided in 8 groups (n=10): G1 to G4 were formed by z350 samples, and G5 to G8 were formed by Bulk-Fill samples. Subsequently, samples were brushed with the following dentifrices: distilled water (control); Maximum Cavity Protection (G2 and G6), Colgate Luminous White Advanced (G3 and G7) and Curaprox Black is White (G4 and G8). Brushing was performed with an electric toothbrush (1,96N; 30 minutes). Every 30 seconds, 1.0 ml of the slurry was injected between the bristles of the brush and the specimen. After abrasive challenge, samples had their color and roughness reevaluated. The color difference (ΔE) between each stage were analysed by ANOVA and Tukey's test. The Two-Way ANOVA and Tukey's test were used to compare the ΔE obtained for baseline x after staining and baseline x after brushing. Two-Way ANOVA followed by post hoc Tukey's test analyzed the differences between the initial and final surface roughness of each group ($\alpha=5\%$). Results showed Z350 was more susceptible to staining than Bulk Fill. Excepting group 3, all color changes remained below 3.3 after brushing. No abrasive challenge was able to recover the color of the composites, but all abrasive challenges changed the surface roughness of the composites. It was concluded that whitening toothpastes failed to remove red wine staining from nanofilled composites, and the abrasive challenge increased the surface roughness of that dental materials.

KEY WORDS: composite resins, whitening dentifrices, roughness, color.

5. INTRODUCTION

Composite materials are increasingly used in clinical offices due to their favorable characteristics, such as esthetic appearance and good physical and mechanical properties. Composite restorations present high longevity in the oral cavity, if their clinical indications are correct ¹. Esthetic appearance and mechanical properties of resin composites have been shown to be influenced by their composition and microstructure. Nanofilled composites, that use nanosized particles (1-100 nm) throughout the resin matrix, are indicated for both anterior and posterior restorations, mainly due to its high initial polish and gloss, as well as its mechanical strength suitable for use in high-stress-bearing areas ². Nevertheless, despite advances in this material, it needs to be placed in the cavities using the incremental filling technique in order to minimize adverse effects caused by the polymerization shrinkage of the composite. This technique, although effective, increases the clinical time for tooth restoration ³.

In an attempt to reduce clinical time, bulk-fill resin composites have been introduced to be employed as a bulk restorative material when performing direct restoration, with the possibility to build up increments up to 4 or 5 mm, while decreasing the typical problems regarding depth of cure and polymerization contraction showed by conventional resin composites when used in these same conditions. This composite may be of high and low viscosity depending on its clinical indication ⁴. Likewise the conventional composites, bulk fill composites are subject to degradation and staining, which severity depends on the individual's diet.

When staining of the restorations occurs, several treatment options may be proposed for patients. The most effective would be the replacement of pigmented restorations ¹. However, this option is considered invasive, because dentist also remove, besides the restoration, sound hard dental tissue on the margins of the old restoration, so that a new one can be satisfactory positioned. In addition, this option has a higher cost than re-polishing the pigmented restorations.

Sometimes extrinsic pigmentation also occurs in natural teeth. In this case, tooth color recovery can be done by home and at-office dental bleaching, micro-abrasion techniques and, more recently, by the use of whitening toothpastes ⁵. Although controversial, the use of whitening toothpastes has been gaining prominence. These

category of dentifrices have in their formulation humectants, surfactants and some types of abrasives such as silica, calcium carbonate, sodium bicarbonate, calcium pyrophosphate, alumina, hydrogen peroxide, optical pigments among other components⁶. Previous studies have shown that the cleaning action is mainly promoted by the abrasive particles, which are insoluble minerals designed to disorganize the bacterial biofilm, removing microorganisms and stains, giving a whitened appearance. Notwithstanding, the use of whitening dentifrices may results in higher tooth wear⁷.

Recently, charcoal-based toothpaste has been introduced to the market and, because of that, limited information has been published. The whitening effect of activated charcoal is based on its high capacity to adsorb and retain chromophores in the oral cavity. It promises to remove discoloration using activated carbon without abrading, since its relative relative dentin abrasion (RDA) is 50. The whitening effect of activated carbon is supplemented by optical means, where a blue filter reduces yellow discoloration. This helps to make teeth appear whiter without the use of chemical agents⁸. However, it is not known whether carbon-based toothpastes have any effect on composite resin restorations.

Therefore, the objective of the present study was to evaluate the color stability and the surface roughness of different composites subjected to brushing with whitening toothpastes. The null hypothesis is that the roughness and the color of the composite are not changed after abrasive challenge.

6. MATERIALS AND METHODS

6.1 Sample preparation

The composite resins (Table 1) were manipulated following manufacturers' instructions. Each material was inserted into the cylindrical stainless steel metal mold (6.0 mm diameter × 2.0 mm thickness) in one increment with an appropriate instrument to obtain 40 disc of each composite. Immediately after insertion of the material, a polyester strip and a glass slide were placed over the mold/resin under axial load of 500g during 1 minute to obtain a flat surface. The composite was light cured for 20s, according to manufacturer's instructions, using a visible light-curing unit with 1,200 mW/cm² power output (Radii-cal, SDI Limited, Vitoria, Australia). The surfaces opposite to the glass slide were finished and polished with Sof-Lex Pop On sequential discs (3M ESPE, St Paul-

MN, USA) from the coarsest to the finest granulation. To standardize the color and surface roughness analyses, a small identification was done at the bottom of the specimen, so the measurements could be done always in the same position. Prior to baseline measurements, the specimens were washed with distilled water for 30 seconds, dried with absorbent paper and immersed in distilled water at 37°C.

6.2 Baseline analysis

The baseline surface roughness measurements were assessed using a 3D Laser Confocal Microscope (LEXT 4000, Olympus Co., Hamburg, Germany) at a magnification of 40×. All data were documented at a resolution of 1024x1024 pixels. The baseline color of the specimens were measured using the CIE-Lab color system, which is defined as a 3-dimensional (3D) measurement system, was applied. In this system, “L” indicates the brightness, “a” the red-green, and “b” the yellow-blue proportion of the color. Three measurements were done with the active point of the spectrophotometer in the center of each specimen to obtain an average of each specimen. Spectrophotometric measurements were made using a white background, as previously described. (Pecho et al., 2019) Before testing, the spectrophotometer (Vita Easyshade Compact Advance 4.0, VITA Zahnfabrik H. Rauter GmbH & Co. KG - Bad Säckingen – Germany) was calibrated according to manufacturer recommendations using the supplied calibration standards.

6.3 Exposure to red wine

All samples were exposed to red wine (Cantina da Serra[®] - COMARY- Arbor Brasil- Teresópolis, RJ- Brasil; 10.5% alcohol by volume, pH 3.36), Specimens were placed into 24-well cell culture plates, with each well containing a single specimen. Two milliliters of red wine were then added to the cell culture wells, and the specimens were fully submerged in the liquid. The specimens were exposed for 4 h per day for 30 days at 25 ± 1 °C⁸. Subsequently, surface roughness measurements were assessed using the same procedure done at baseline.

6.4 Abrasive challenge

The composite discs were allocated in eight groups, according to the type of composite and dentifrice used (Table 2) (n=10). Groups 1 and 5 were brushed with distilled water (no dentifrice), Groups 2 and 6 were brushed with Colgate Cavity Protection (Colgate Oral Pharmaceuticals, New York, USA), Groups 3 and 7 were brushed with Colgate Luminous White Advanced (Colgate Oral Pharmaceuticals, New York, USA) and groups 4 and 8 were brushed with Curaprox® Black is White (CURADEN International AG, Kriens). Each composite disc was brushed at standardized abrasion force (1,96N). The dentifrice slurries were made immediately before use and consisted of 1-part dentifrice (100 mL) to 2-parts distilled water (200 mL) and hand-mixed for 2 min, following ISO #14569-1 specification. An automatic tooth brushing device (Oral-B Pro 5000, Procter and Gamble, Cincinnati-OH, USA) with standardized soft bristled toothbrushes (Oral-B Precision Clean, Procter and Gamble) was used. Each composite disc was brushed during 1800 seconds. Considering that a person brushes each tooth 3 times a day during 5 seconds on each face of the tooth, the present brushing protocol simulated 120 days of toothbrushing⁹. During the abrasive challenge, 1.0 mL of the respective slurry (or distilled water for groups 1 and 4) was injected sideways to the specimen every 30 seconds, between the restorative material and the toothbrush. At the final of the abrasive challenge, the composite discs were cleaned in an ultrasonic cleaner device during 1 minute.

6.5 Final Surface roughness and color change analyses

The surface roughness of each specimen was evaluated at the end of the brushing challenge under the same conditions described for the baseline and after staining roughness analysis. The baseline and the final analysis were made exactly at the same position and area. The response variable was the difference between the baseline, the roughness after red wine staining and after brushing.

The final color analysis was done with the same colorimeter used for the baseline and after staining measurements. The color difference (ΔE) between the color coordinates was calculated by applying the formula $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ in order to compare values at baseline and after staining, after staining and after the abrasive challenge, and at baseline and after the abrasive challenge. Three measurements were

done, with the active point of the spectrophotometer in the center of the specimen to obtain an average of each specimen. As for the surface roughness measurement, the color analysis were made exactly at the same position and area.

6.6 Statistical analyzes

Based on a previous study, a sample of 10 per group would allow 90% power at an alpha level of 0.01 to detect the expected differences between groups ⁵.

The data were tabulated and subjected to statistical analysis (Sigmastat 3.01, Systat, USA). First of all, the data were subjected to the D'Agostino normality test. The primary outcome measure was the mean color difference (ΔE) of composite resins before and after exposure to red wine. The means were compared using analysis of variance (ANOVA) and post hoc Tukey's test. The same method was used to compare ΔE obtained in all groups after staining and after the brushing procedures. Finally the Two-Way ANOVA and Tukey's test were used to compare the ΔE obtained for baseline x after staining and baseline x after brushing. The level of significance adopted in all cases was set at 5%. The differences between the initial and final surface roughness of each group were analyzed by Two-Way ANOVA followed by post hoc Tukey's test. The level of significance adopted was set at 5%.

7. RESULTS

8.1 Color Change

The mean (\pm standard deviation) color difference (ΔE) of composite resins before and after exposure to red wine is shown in Table 3. According to statistical analysis, the nanofilled Filtektm Z350 composite resin (24.48 ± 0.72) was more susceptible to red wine staining than Bulk Fill ($12.43 \pm 1,15$) ($p < 0.01$).

Figure 1 shows the mean (\pm standard deviation) ΔE (after staining x after brushing) found in each experimental group. Excepting group 3 (4.81 ± 3.85), all color changes remained below 3.3, which is the threshold value for the unaided visual perception of color changes. However, the value found for group 3 was statistically similar to those found for the other groups ($p > 0.05$).

Table 4 shows the comparison between ΔE obtained for baseline x after staining and baseline x after brushing in all groups. Similar to Table 3, red wine had greater effects on nanofilled composites than on bulk fill composite samples ($p < 0.05$). These data were the same when comparing ΔE after abrasive challenge. This table also shows that brushing with or without bleaching dentifrices were not able to reduce the red wine staining.

8.2 Surface Roughness Analysis

The baseline, after red wine staining and after brushing surface roughness of the nanofilled and the bulk fill composites are shown in Table 5. Results pointed out the initial (baseline) roughness was similar for both composites. Surface analysis after staining with red wine showed the alcoholic beverage did not change the roughness of the composites when compared to their baseline values. In addition, Z350 and Bulk Fill composites presented similar surface roughness after staining when compared to each other ($p > 0.05$). This table also shows that brushing with or without bleaching have changed the surface roughness when compared to baseline and after staining values ($p < 0.05$). However, when comparing the effect of brushing with different dentifrices on the surface roughness of each composite, it was observed that the roughness of the Bulk Fill composite brushed with Colgate Cavity Protection ($2.129 \pm 0.12 \mu\text{m}$) was lower than the values found for Filtek Z350 samples brushed with Colgate® Luminous White Advanced ($2.489 \pm 0.16 \mu\text{m}$) and with Curaprox® Black is White ($2.645 \pm 0.30 \mu\text{m}$).

8. DISCUSSION

This study showed that brushing nanofilled or bulk fill composite discs with the whitening dentifrices used here did not remove red wine staining on the surface of those composites. On the other hand, the three dentifrices changed the surface roughness of the composites. Thus, the null hypotheses can be refuted for the surface roughness but not for the color change.

The present study aimed to evaluate if whitening dentifrices are able to remove severe staining from the composite surfaces, returning the original color of the restorative material. The stain chosen was red wine because it is widely consumed worldwide and

known to cause tooth discoloration^{10, 5}. The staining protocol used here was based on previous studies and was intended to simulate a severe discoloration of composite restorations^{10, 8, 5}. The discoloration promoted by red wine is related to its low pH (3.36), which may affect the surface integrity of dental composites, enable easier penetration of pigments into the resin matrices, leading to internalized discoloration that makes it difficult to be removed⁵.

Red wine used here contained 10.5% alcohol by volume. Alcohol in beverages acts on bisphenol A-glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA)-based polymers, that may increase surface roughness and may reduce the hardness of the composite resins since it works as a plasticizer of the polymer matrix, leading to soften and dislodge filler particles¹¹.

Table 3 showed that the composite Filtek™ Z350 XT was more susceptible to red wine staining when compared to Filtek™ One Bulk-Fill composite. Previous studies also showed Bulk-fill resin composite yielded better color stability and similar water sorption and solubility values than nanofilled composites^{12,13}. Discoloration is a multifactorial problem, and water sorption is one factor associated with the ability of composites to discolor. The composite Z350 XT contains a Bis-GMA/TEGDMA (triethylene glycol dimethacrylate) copolymer system, and previous research demonstrated that the presence of TEGDMA makes the restorative material more vulnerable to water sorption, resulting in a higher level of staining^{14, 15}. The bulk fill restorative material used here contains two methacrylate monomers (aromatic urethane dimethacrylate - AUDMA and addition fragmentation monomers – AFM) that, in combination, act to lower polymerization stress. During polymerization, AFM reacts into the developing polymer as with any methacrylate, including the formation of cross-links between adjacent polymer chains¹⁶. The BisGMA monomer that is used in our other composites has been replaced with a dimethacrylate that does not use Bisphenol A in its synthesis. Additionally, DDDMA (1, 12-Dodecanediol dimethacrylate) has a hydrophobic backbone that increases its molecular mobility and compatibility with nonpolar resins and aids in adjusting viscosity^{16, 17}. As previously stated, the presence of hydrophobic monomers favors the color stability of the restorative material, which could explain the lower staining caused in this composite when compared to Z350 XT.

The color change of the composites was measured with a portable digital colorimeter (Vita Easyshade Advance 4.0), which is used to verify the color of composite restorations in vitro and in vivo. (Klotz et al., 2018) The color is measured according to the CIE L*a*b* system. The definition of L*, a*, and b* follows the recommendations of the Commission Internationale de l'Eclairage (CIE; International Commission on Illumination) and enables three-dimensional description in the colorspace (L*=lightness; a*= red-green and b*=blue-yellow.) The use of CIELAB-based spectrophotometers is the most effective method used for assessment of the color and color changes over time because it is more objective and accurate than using a visual scale or photographs¹⁸.

Figure 1 showed, except for group 3 (Z350 XT brushed with Colgate Luminous White), that the color change (ΔE) between stained composite and brushed samples of the nanofilled and bulk-fill composites remained below 3.3. Three different intervals are used for distinguishing color differences: ΔE values of 1 are regarded as not appreciable by the human eye; ΔE values between 1.0 and 3.3 mean that this change is noticeable only by a qualified person (i.e. the color of the composite is clinically acceptable); and ΔE values over 3.3 indicate that the color change of the material can be easily observed¹⁹. In this way, the color change after brushing could only be noticed through a colorimeter, which demonstrates the ineffectiveness of the dentifrices in removing the discoloration of the composites.

In accordance with Table 4, Table 5 showed that the abrasive challenge, with or without toothpaste, was not able to remove red wine staining from the composite resins tested here and did not return the initial/baseline color of the restorative materials.

One method proposed to remove extrinsic staining of teeth is brushing with whitening toothpastes. In this context, electrical brushing is adequate to simulate daily oral hygiene procedures in vitro, since some parameters must be standardized, such as the time, frequency, amount of toothpaste used during brushing and the force applied on the specimen during abrasive challenge²⁰. In the present research, an electric toothbrush was used, with soft bristles and a standard brushing force of 1.96N, during 1800 seconds uninterruptedly^{21,22}.

Although initially developed to remove extrinsic staining from teeth, whitening toothpastes have been studied to assess whether they can remove extrinsic staining from direct restorative materials^{23,20,2,7,6}. The action of whitening toothpastes is based on the

presence of active ingredients, such as proteases, and phosphate-based compounds (pyrophosphates) which displace anions or negatively charged macromolecules associated to the acquired enamel pellicle and help to degrade the stained pellicle. In addition, oxidizing agents, like hydrogen peroxide and sodium chlorite, are able to penetrate into teeth and to remove extrinsic stains⁷. Besides these components, there are also optical agents, like blue covarine, a pigment that emulates the enamel properties regarding scattering wavelengths in the blue extension, changing the yellowish to a bluish appearance. Whitening toothpastes may also contain more abrasive agents than the conventional dentifrices, optimizing the mechanical removal of biofilm and the control of extrinsic pigments⁶.

Colgate Cavity Protection is a minimally abrasive conventional fluoride toothpaste and was chosen because it has been widely applied for caries control⁷. Colgate Luminous White contain tetrasodium pyrophosphate and hydrated silica as abrasive, being considered one of the most abrasive dentifrices, since the combination of those components is known to contribute in enhancing the abrasivity effect of the whitening toothpastes^{6,24}. Also, it has 1% hydrogen peroxide in its composition. However, despite having a chemical bleaching agent, which could potentiate the whitening effect, the ΔE in groups 3 and 7 were similar than that of the other groups. Thus, the whitening effect of this dentifrice may be more related to the abrasive action of hydrated silica and other agents that assist in the removal of extrinsic stains present in the formulations.

The use of charcoal-based dentifrices and powders are believed to be increasing in many countries across the world. Those fashionable oral hygiene products promise to remove extrinsic stain removal without abrasion of the dental hard tissue^{25,26}. There is a lack of studies regarding the charcoal-based dentifrice used here but, according to Curaprox[®] Black is White manufacturer's, this whitening toothpaste removes stains using activated carbon without abrasive agents and without bleach. The RDA is correspondingly low (about 50), which is lower than in many conventional toothpastes, and the activated carbon particles simply absorb pigments particles "like a vacuum cleaner". Furthermore, sparkling blue aesthetically supports the whitening effect as a physical brightener and emphasizes the whiteness of teeth. However, a previous study showed that charcoal-based dentifrice is less effective than abrasives and blue covarine dentifrices to remove tooth pigmentation²⁶.

Although previous researches have shown that toothpastes have been able to remove stains caused by coffee, tobacco or soy sauce, the abrasive challenge used here could not remove red wine staining on composites^{24, 5}. The results obtained here also showed that the stain removal brushing only with distilled water was similar to that observed in the presence of toothpastes. This may have occurred here due to the staining severity and the staining depth of the red wine. In addition, it is likely that the bonding of the wine pigments with the resin is strong enough to resist the action of the chemical components the conventional or of the whitening toothpastes, and to be removed by the mechanical action of the brush.

One of the goals of toothbrushing is to polish teeth and restorations superficially to remove stain and to achieve surfaces that are smoother and less susceptible to staining. Since completely flat restorations can not be achieved, such procedures can have a direct influence on the longevity of the restoration. In the present research, although staining with red wine did not make the surface rougher, the confocal laser scanning microscopy demonstrated that abrasive challenge changed the surface roughness of both composites, which is in agreement with past studies^{20, 27}.

The surface roughness of a composite is influenced by a number of factors, such as the size of the filler particles, the percentage of surface area filled by the inorganic particles, the hardness, the degree of conversion of the composite and the interaction between the organic and inorganic matrices²⁸. According to manufacturer, both composites used here are nanofilled. Filtek™ One Bulk Fill contains agglomerate 100 nm ytterbium trifluoride and a combination of a non-agglomerated/non-aggregated 20 nm silica filler, a non-agglomerated/non-aggregated 4 to 11nm zirconia filler, an aggregated zirconia/ silica cluster filler (comprised of 20nm silica and 4 to 11nm zirconia particles), making the total inorganic filler loading approximately 76.5% by weight (58.4% by volume). The fillers of Filtek™ Z350 XT are a combination of non-agglomerated/non-aggregated 20 nm size silica, 4-11nm zirconia unclustered/unaggregated and clusters of zirconia/silica aggregate particles (combination of 20 nm silica particles and zirconia at 4-11 nm), making the total inorganic filler loading approximately 78.5% by weight (by 63.3% by volume).

Changes on surface roughness after abrasive challenge are related to the degradation of the polymer matrix or the matrix/filler interface, and the release of filler

particles from the resin matrix²⁹. A previous research reported that the nanoparticles were detached from the organic matrix after automated brushing using low abrasive dentifrice and soft bristle brushes and brushing strength of 0.2N, suggesting that due to their size and regularity, nanoparticles can be more easily removed from the composite surface than larger and irregular filler particles²⁹.

The abrasivity of the dentifrices is measured by the relative dentin abrasivity (RDA), which is the abrasivity of a dentifrice in relation to a standard paste set at 100. RDA is a reasonably robust method considered a useful tool for the determination of the relative abrasive level of dentifrices and abrasive powders³⁰. RDA ranges from 0 to 250, and low abrasive dentifrices have RDAs between 0-70, medium abrasive have RDAs of 71-100, high abrasives have RDAs of 101-150, and those considered potentially damaging to dentin have a RDA of 151-250. According to that classification, the dentifrices used here had low (Colgate Cavity Protection – RDA=70; and Curaprox® Black is White – RDA=50), and high RDA (Colgate Luminous White - RDA = 175)³⁰.

Gel toothpastes containing silica as its abrasive agent are considered of low abrasives. However, when silica is combined with other abrasives, such as calcium carbonate, sodium pyrophosphate, titanium oxide or sodium phosphate, it is considered as a high abrasive dentifrice. Abrasive wear increases linearly as the particle size increases up to a critical size. Silica, when used in fine particles and with regular forms, preserves its characteristic of low abrasive mineral. Nevertheless, when thick and irregular particles are incorporated, the dentifrice becomes highly abrasive. Thus, only the formulation or type of abrasive present in a dentifrice is not sufficient to characterize its abrasiveness to the composites^{6, 30, 7}.

In the present research it was decided to carry out experimental groups where the abrasive challenge was performed without dentifrice (G1 and G5). Results indicated the action of the soft-brush bristles was sufficient to change the surface roughness of the two composites in the same magnitude observed by brushing with conventional and whitening toothpastes. Although the softer bristles lead to a less degradation of the surface of the composite, the time and the brushing force contribute to increase the surface of the restorative material³¹.

Table 5 also showed that immersion in red wine did not change the surface roughness of the composites used here. There is no consensus in the literature about the

effect of red wine on the superficial smoothness of composites. What is known is its effect depends on the composition, pH, alcohol content and temperature of the beverage. In this way, conflicting results are expected to be found, as the drinks have different compositions around the world^{32, 33, 10}.

According to the data found here, it was observed that whitening toothpastes were not able to remove the staining caused by red wine on conventional and bulk fill nanofilled composite resins. Furthermore, the surface roughness of the composites after brushing was similar in the presence or absence of the dentifrice, which showed that the action of the bristles on the composites seems to be more aggressive than the abrasiveness of the dentifrices. As previously mentioned, the methodology was applied to simulate severe staining by a beverage consumed worldwide and with a large quantity of dark pigments. Thus, it is not yet known whether the charcoal-based dentifrices would be able to remove less severe pigments on teeth and on composites stained with other substances such as juices, foods or tobacco. Additionally, further studies are needed to see if brushing with these toothpastes while using dark beverages or pigmented foods could prevent staining of composites.

9. CONCLUSIONS

According to the results obtained in the present research, it was observed that, due to its organic matrix, Z350 was more susceptible to staining than Bulk-Fill composite. Besides that, the whitening toothpastes used here failed to recover the color of the red-wine-stained composites. Additionally, brushing with the dentifrices tested here increased the surface roughness of the nanofilled composites. However, the whitening dentifrices did not cause any alteration different from those observed in groups where the abrasive challenge was performed without dentifrice

10. APPENDIX

Table 1. Composition of restorative materials used in this study*

Composite	Composition			
	Matrix	Filler Size	Filler (weight/volume)	Load
Filtek™ Z350 XT	Bis-GMA, UDMA, TEGDMA, BIS-EMA, PEGDMA	20-75nm	78.5%/63.3%	
Filtek™ One Bulk-Fill	AUDMA, AFM, DDDMA, UDM	4-100 nm	76.5%/58.4%	

*Product information according to the manufacturer 3M ESPE (St Paul-MN, USA) (AUDMA, aromatic urethane dimethacrylate; AFM, addition fragmentation monomers; BIS-EMA, bisphenol A-polyethylene glycol diether dimethacrylate; BIS-GMA, bisphenol A-glycerolate dimethacrylate; DDDMA (1, 12-Dodecanediol dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, tetraethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.)

Table 2. Composition of dentifrices used in this study*

Groups	Dentifrice	Composition	RDA
1 and 4	None	Distilled Water	0
2 and 5	Colgate Cavity Protection ¹ (L3290CO1014)	Sodium Monofluorophosphate, Dicalcium Phosphate Dihydrate, Water, Glycerin, Sodium Lauryl Sulfate, Cellulose Gum, Flavor, Tetrasodium Pyrophosphate, Sodium Saccharin	70
3 and 6	Colgate Luminous White Advanced (9103BR12B)	Water, hydrated silica, sorbitol, glycerin, pentasodium triphosphate, PEG-12, tetrapotassium pyrophosphate, sodium lauryl sulphate, aroma, flavor, cellulose gum, polyethylene, cocamidopropyl betaine, xanthan gum, saccharin sodium, sodium hydroxide, titanium dioxide, D & C blue in 1 aluminum lacquer (CI 42090) and 0.243% sodium fluoride (1100 ppm fluorine)	175
4 and 8	Curaprox Black is White (98MHDEXP022)	Water, sodium monofluorophosphate (0.723%) potassium thiocyanate (0.02%), sorbitol, glycerin, hydrated silica, charcoal powder, aroma, argilla, decyl glucoside, cocamidopropyl betaine, sodium monofluorophosphate, tocopherol, mica, xanthan gum, hydroxyapatite (nano), titanium dioxide, microcrystalline cellulose, maltodextrin, potassium acesulfame, sodium benzoate, potassium chloride, potassium sorbate, menthyl lactate, methyl diisopropyl propionamide, ethyl menthane carboxamide, zea mays starch, stearic acid, cetearyl alcohol, citrus limon peel oil, citric acid, lactoperoxidase, glucose oxidase, amyloglucosidase, tin oxide, sodium bisulfite, hydrogenated lecithin, limonen, CI75810, CI77289.	50

*Product information according to the manufacturers. (RDA= Relative Dentin Abrasivity). ¹Colgate-Palmolive Industrial LTDA (Sao Bernardo do Campo-SP, Brasil), ²Curaprox Brasil (São Caetano do Sul-SP, Brasil)

Table 3. Comparison between mean (\pm standard error) ΔE found for each composite after red wine staining (One-Way ANOVA followed by Tukey's test)

	Z350 XT 1 (n=40)	One Bulk Fill (n=40)	p
ΔE	24.48(\pm 0.72)	12.43(\pm 1,15)	<0,01

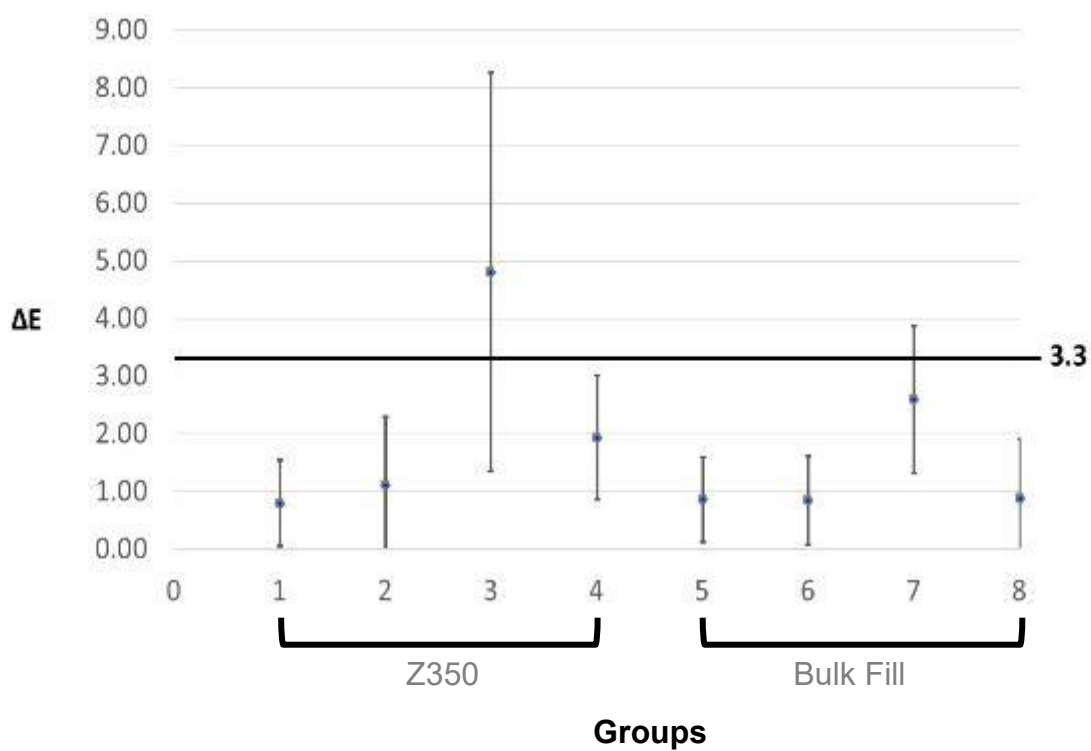


Figure 1. Mean (\pm standard deviation) ΔE (after staining x after brushing) found in each experimental group.

Table 4. Mean ΔE (\pm standard error) comparison among each experimental group. Capital letters indicate comparisons made within each column and lowercase letters compare rows. Different letters indicate the presence of statistically significant differences ($p < 0.05$).

ΔE			
Composite	Group	Baseline x After staining	Baseline x After brushing
Filtek™ Z350 XT	1	25.02(\pm 4.01)Aa	25.00(\pm 3.55)Aa
	2	23.92(\pm 5.31)Aa	23.01(\pm 4.03)Aa
	3	25.18(\pm 5.08)Aa	21.06(\pm 3.75)Aa
	4	23.79(\pm 5.99)Aa	22.20(\pm 5.39)Aa
Filtek™ One Bulk-Fill	5	13.51(\pm 2.72)Ba	13.81(\pm 2.63)Ba
	6	11.87(\pm 2.03)Ba	10.97(\pm 7.71)Ba
	7	13.26(\pm 4.73)Ba	10.80(\pm 5.99)Ba
	8	11.08(\pm 2.15)Ba	10.34(\pm 20.7)Ba

Table 5. Mean surface roughness (\pm standard error) of all experimental groups. Capital letters indicate comparisons made within each column and lowercase letters compare rows. Different letters indicate the presence of statistically significant differences ($p < 0.05$).

Composite	Groups	Baseline (μm)	After Staining (μm)	After Brushing (μm)
Filtek™ Z350 XT	1	1.929 (\pm 0.14)a	1.947 (\pm 0.34)a	2.472 (\pm 0.40)b
	2	1.949 (\pm 0.15)a	1.968 (\pm 0.08)a	2.357 (\pm 0.33)b
	3	1.964(\pm 0.15)a	1.988(\pm 0.16)a	2.489(\pm 0.16)Ab
	4	1.938(\pm 0.25)a	1.960(\pm 0.24)a	2.645(\pm 0.30)Ab
Filtek™ One Bulk-Fill	5	1.974(\pm 0.12)a	2.004(\pm 0.09)a	2.344(\pm 0.11)b
	6	1.958(\pm 0.15)a	1.986(\pm 0.16)a	2.129(\pm 0.12)Bb
	7	1.961(\pm 0.09)a	1.958(\pm 0.08)a	2.312(\pm 0.23)b
	8	1.972(\pm 0.15)a	1.981(\pm 0.16)a	2.4287(\pm 0.21)b

11. STATEMENTS

12.1 Acknowledgements

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12.2 Disclosure Statement

We certify that there is no actual or potential conflict of interest in relation to the article “Effect of whitening toothpastes on color recovery and surface roughness of red wine stained nanofilled composite resins.

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14. CONCLUSÃO

13. CONCLUSÃO

Considerando os resultados obtidos na presente pesquisa, observou-se que, devido à sua matriz orgânica, a resina composta Filtek Z350 foi mais suscetível a manchas que a resina composta Filtek Bulk-Fill. Além disso, os dentifrícios clareadores testados aqui não recuperaram a cor dos compósitos manchados de vinho tinto. A escovação com os dentifrícios clareadores utilizados aumentou a rugosidade da superfície dos compósitos nanoparticulados. No entanto, o desafio abrasivo não causou alterações diferentes daquelas observadas nos grupos controle.

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APÊNDICE

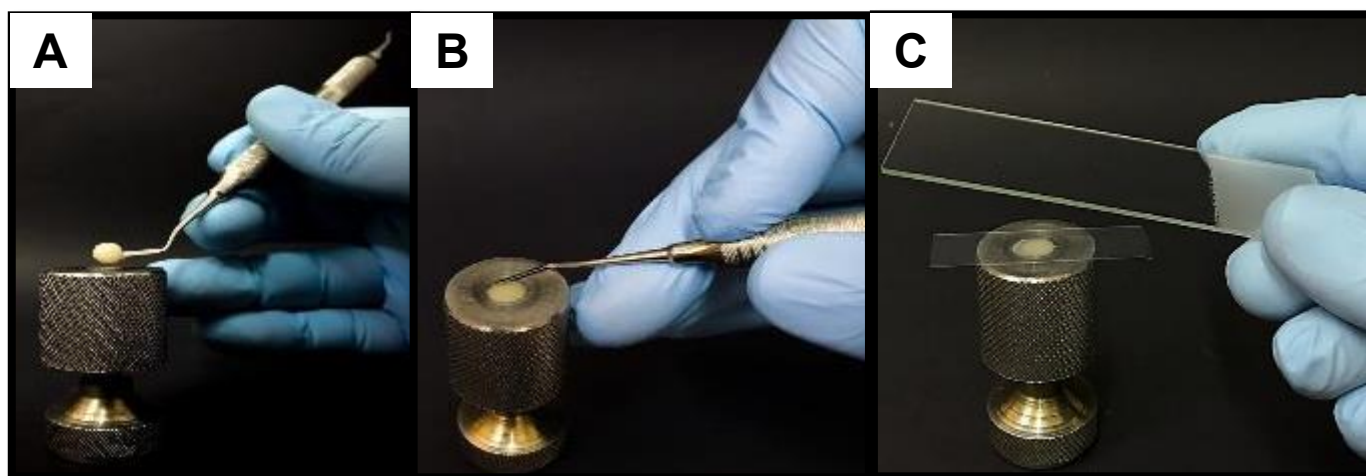


Figura 1. Preparo dos espécimes. A- Inserção do incremento de resina. B- Resina acomodada na matriz de aço (6mmx2mm) com espátula de resina. C- Colocação de tira de poliéster e lâmina de vidro, para compactar a resina.

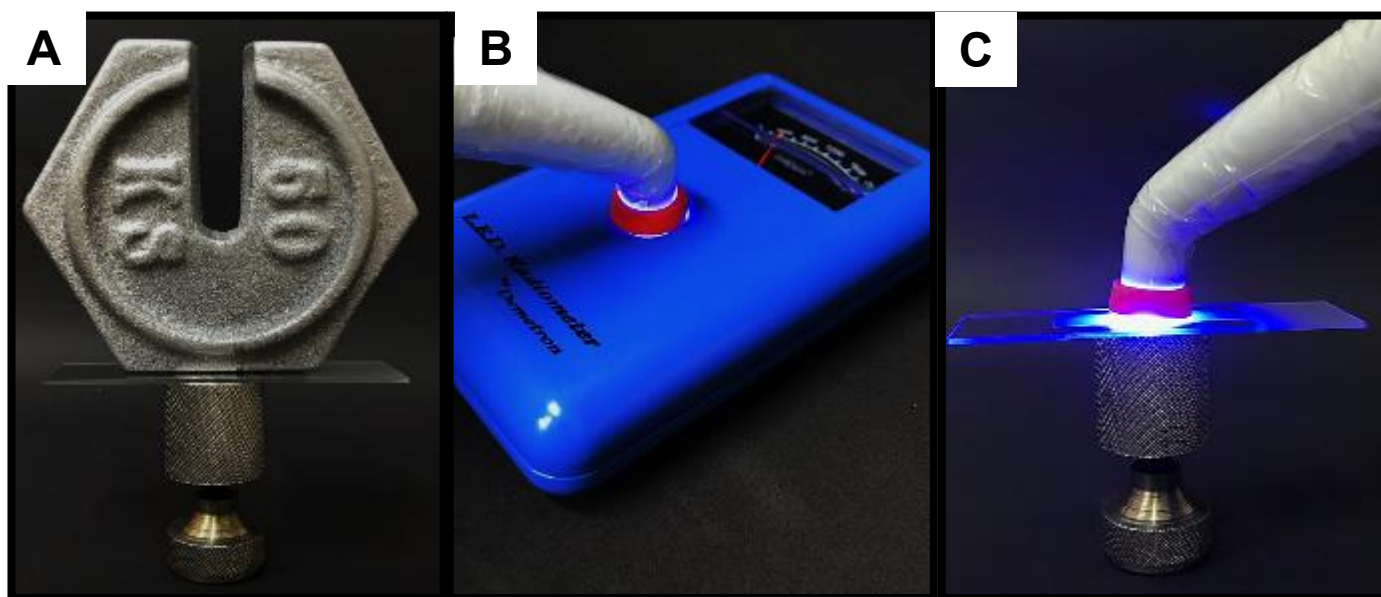


Figura 2. Preparo dos espécimes. A- Aplicação de carga axial de 500g durante um minuto. B- Medição da intensidade de luz do aparelho fotopolimerizador. C- Fotoativação durante 20 segundos.

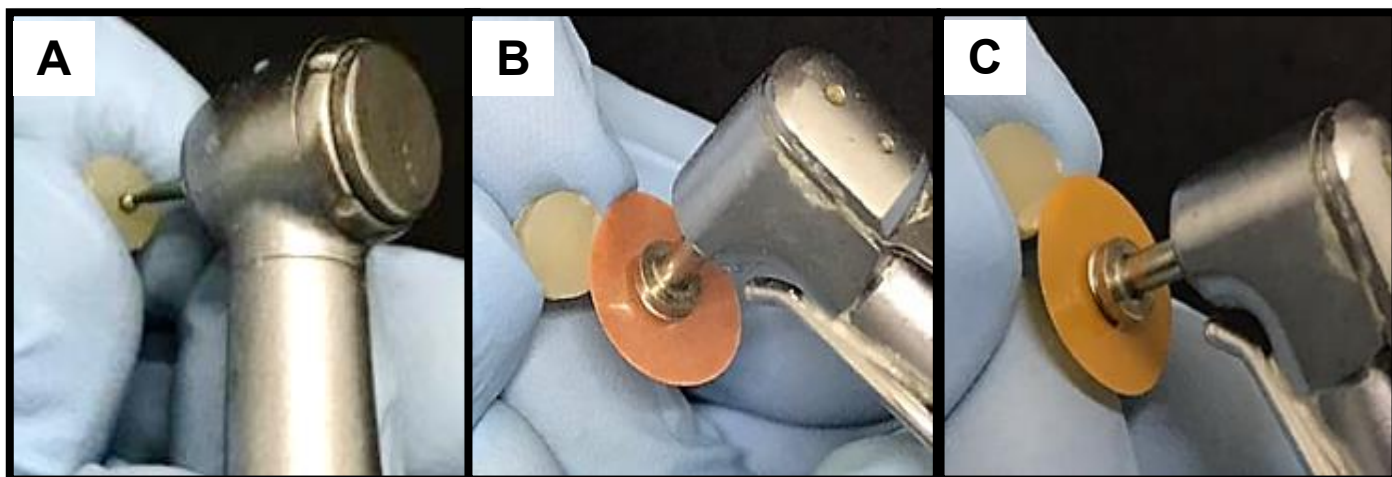


Figura 3. Polimento dos espécimes. A- Marcação com ponta diamantada 1012. B. Polimento com disco de granulação média. C. Polimento com disco de granulação fina.

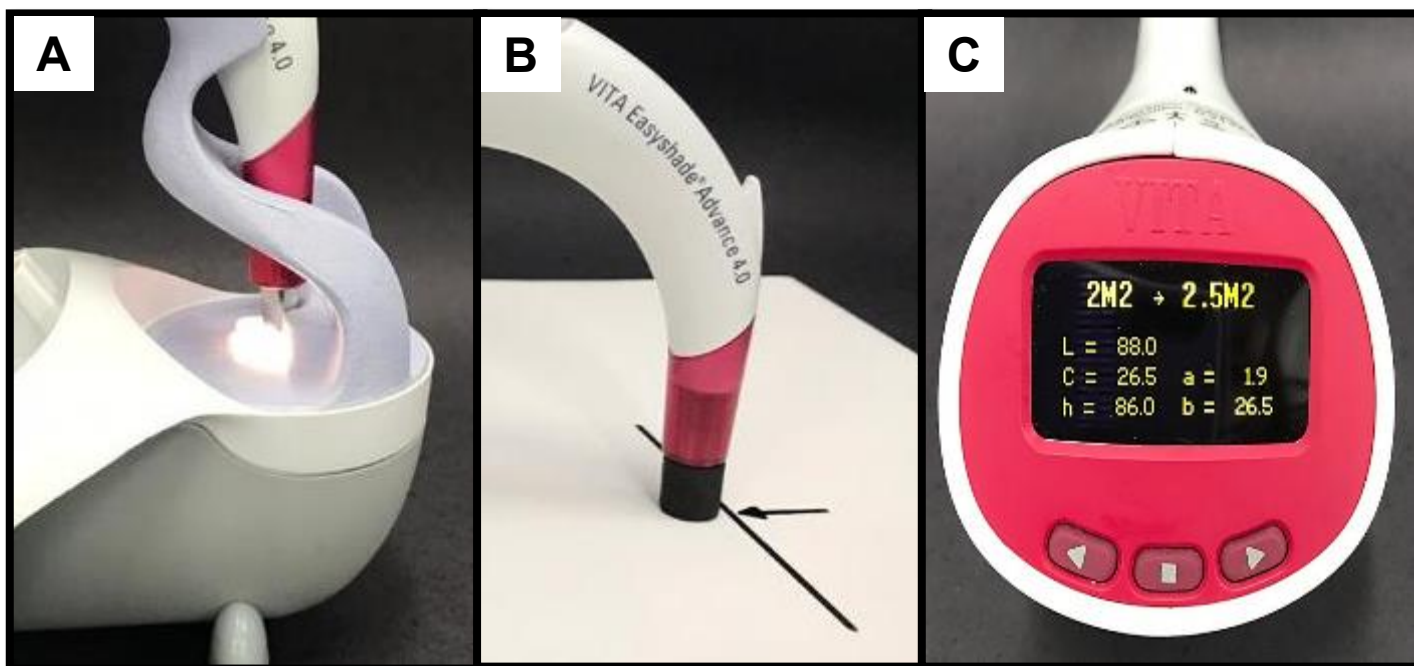


Figura 4. Leitura de cor dos espécimes. A- Calibração do Espectrofotômetro. B- Leitura da amostra de resina utilizando fundo branco. C- Valores da leitura de cor da amostra.

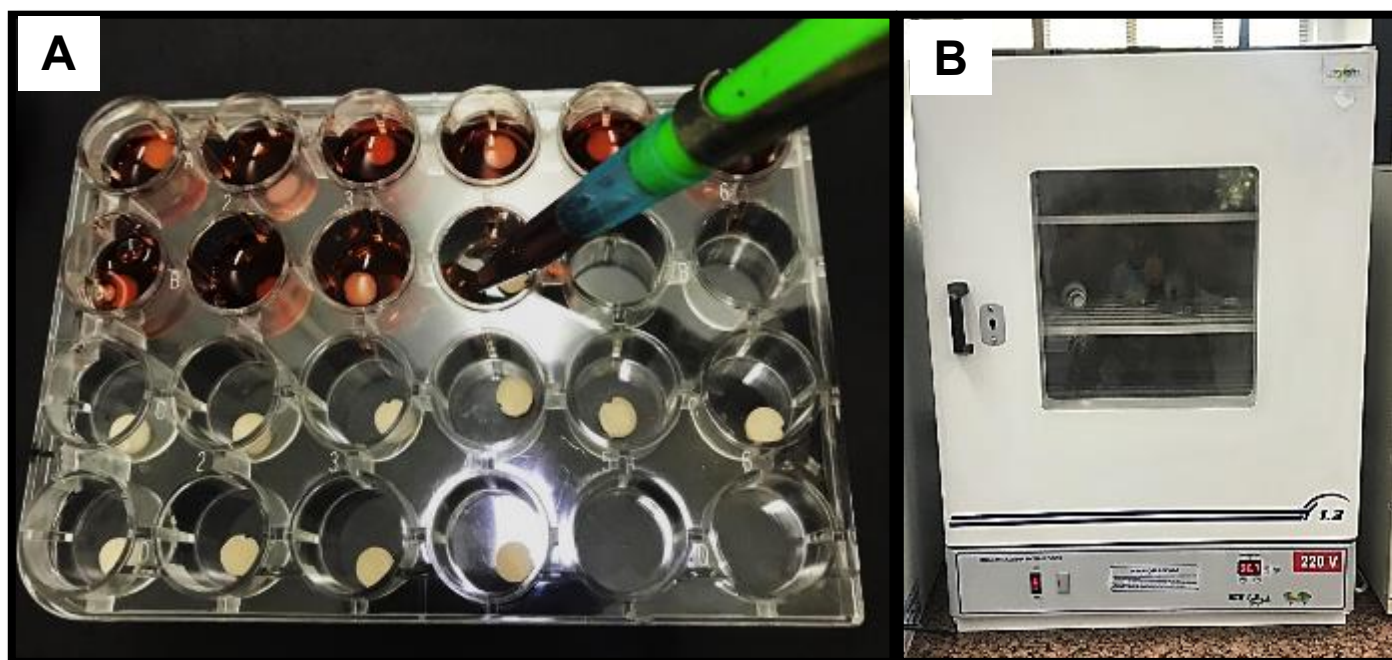


Figura 5. Manchamento dos espécimes. A- Imersão dos espécimes em vinho tinto. B- Espécimes mantidos em estufa.

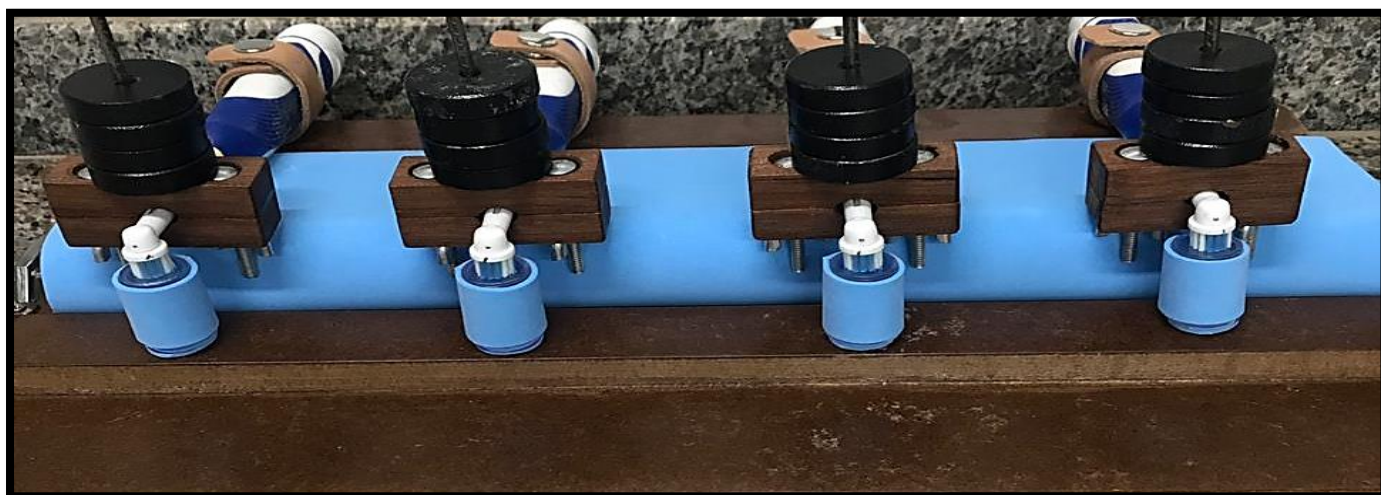


Figura 6. Desafio abrasivo. Escovas elétricas Oral B Pro Saúde Power acopladas em um suporte, com carga axial de 500g.

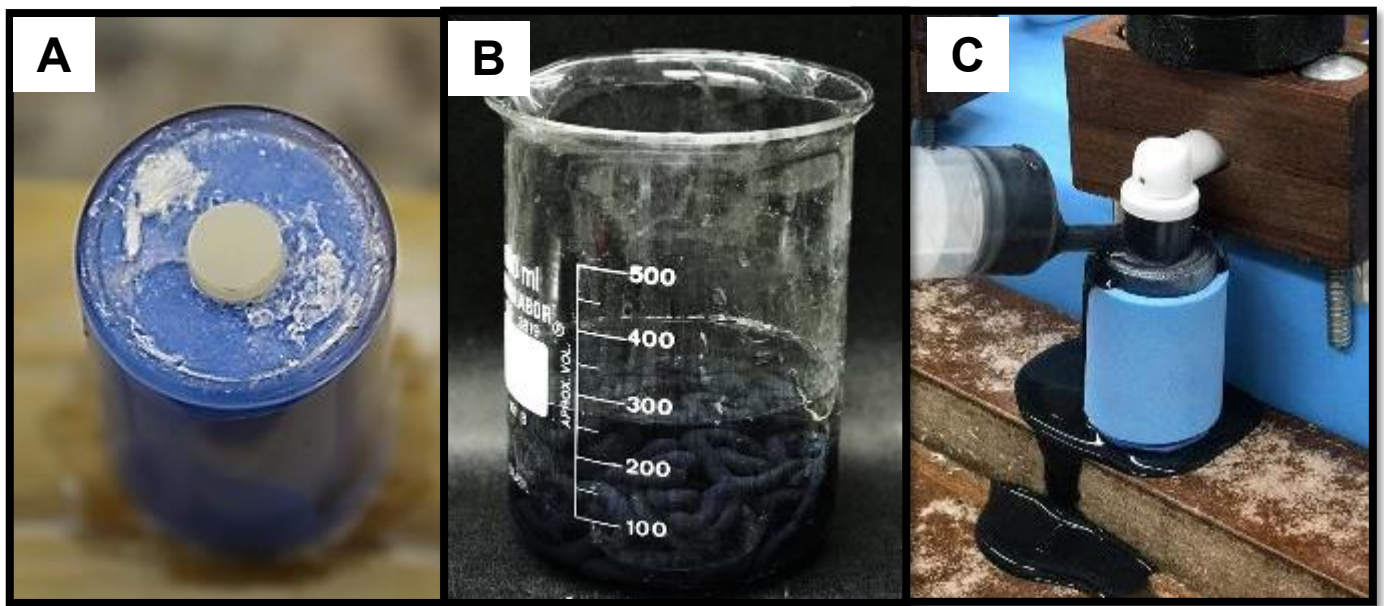
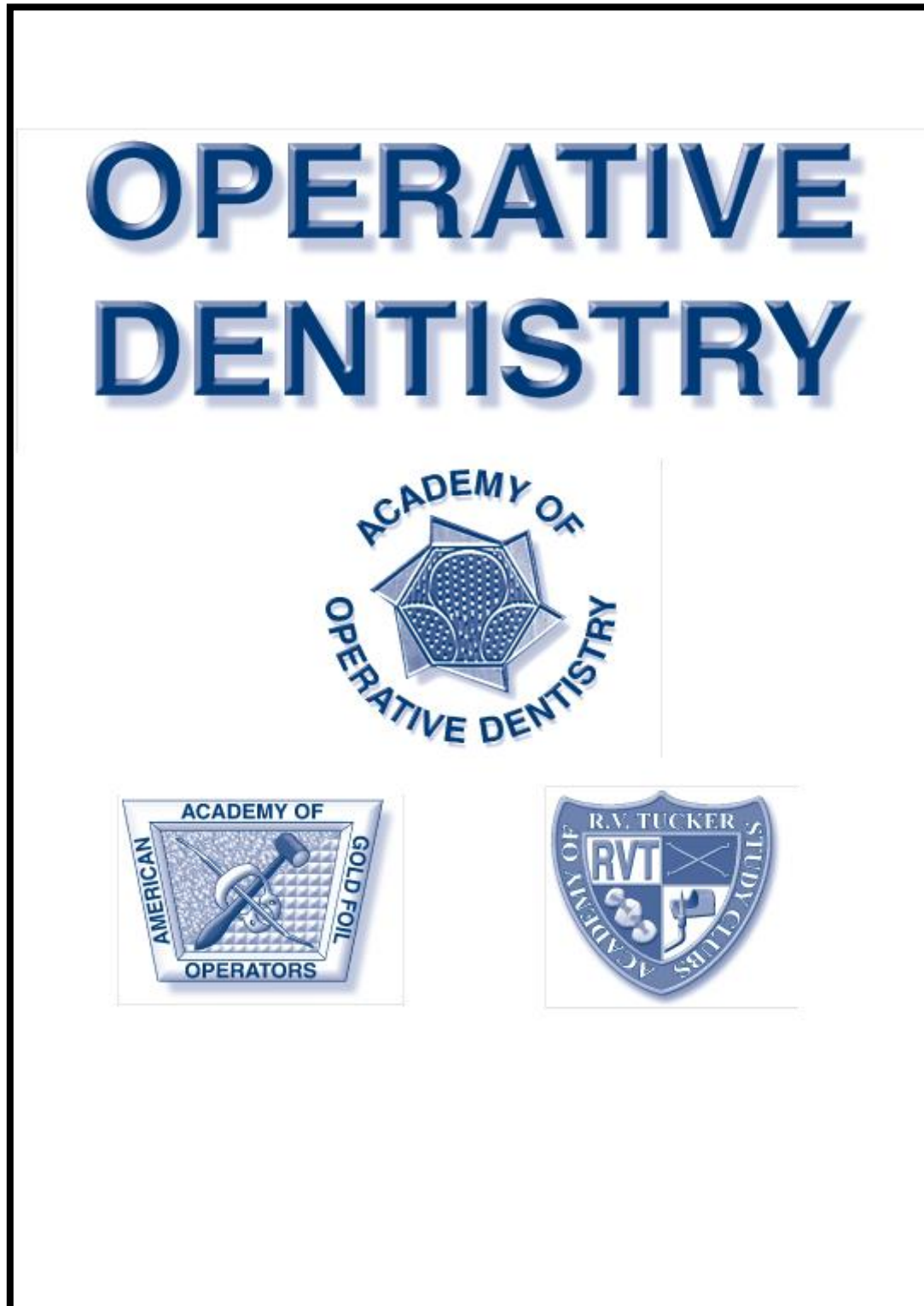


Figura 7. Desafio abrasivo. A- Fixação do espécime. B- *Slurry*. C- *Slurry* sendo injetado entre o corpo de prova e as cerdas da escova.

ANEXO

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- as fotografias coloridas devem ter tamanho aproximado de 3,5 "x 5" e resolução de 300 dpi.

• **OUTROS TIPOS DE MANUSCRITOS**

1. **Os MANUSCRITOS CLÍNICOS DE TÉCNICA / ESTUDO DE CASO** devem incluir:

- um título em execução (curto)
- objetivo
- descrição da técnica
- lista de materiais utilizados
- problemas potenciais
- resumo de vantagens e desvantagens
- referências (veja abaixo)

2. **OS MANUSCRITOS DE LITERATURA E DE REVISÃO DE LIVROS** devem incluir:

- um título em execução (curto)
- uma declaração de relevância clínica com base nas conclusões da revisão

- conclusões baseadas na revisão da literatura ... sem isso, a revisão é apenas um exercício
- referências (veja abaixo)

- **PARA REFERÊNCIAS**

As referências devem ser numeradas (números sobrescritos) consecutivamente, conforme aparecem no texto e, quando aplicável, devem aparecer após a pontuação.

A lista de referências deve ser organizada em sequência numérica no final do manuscrito e deve incluir:

1. Sobrenome (s) do autor e inicial (TODOS OS AUTORES devem ser listados), seguido da data da publicação entre parênteses.
2. Título do artigo completo.
3. Nome completo do diário em itálico (sem abreviações), números de volume e número de edição e primeira e última página completos (por exemplo, 163-168 NÃO atenuado 163-68).
4. Os resumos devem ser evitados quando possível, mas, se usados, devem incluir o acima, mais o número do resumo e o número da página.
5. Os capítulos dos livros devem incluir o título do capítulo, o título do livro em itálico, os nomes dos editores (se apropriado), o nome do editor e o endereço de publicação.
6. Os sites podem ser usados como referências, mas devem incluir a data (dia, mês e ano) acessada para as informações.
7. Os artigos no decorrer da publicação só devem ser inseridos nas referências se forem aceitos para publicação em uma revista e, em seguida, entregues de maneira padrão com “No prelo” após o nome da revista.
8. **NÃO** inclua dados **não** publicados ou comunicações pessoais na lista de referências. Cite essas referências entre parênteses no texto e inclua uma data.