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PRISCILA AGUSTINHA NEVES DE SOUZA

Influência do uso de dentifrícios a base de carvão ativado na estabilidade de cor e rugosidade de resina composta monocromática.

Dissertação apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia, como requisito parcial para obtenção do Título de Mestre em Odontologia naÁrea de Concentração de Clínica Odontológica Integrada.

Uberlândia, Julho 2022

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Uberlândia, Julho 2022

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ATA DE DEFESA - PÓS-GRADUAÇÃO

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Iniciando os trabalhos o(a) presidente da mesa, Dr(a). Gisele Rodrigues da Silva, apresentou a Comissão Examinadora e o candidato(a), agradeceu a presença do público, e concedeu ao Discente a palavra para a exposição do seu trabalho. A duração da apresentação do Discente e o tempo de arguição e resposta foram conforme as normas do Programa.

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SUMÁRIO

RESUMO

Influência do uso de dentifrícios a base de carvão ativado na estabilidade de cor e rugosidade de resina composta monocromática.

– Dissertação Mestrado – Programa de Pós-Graduação em Odontologia – Faculdade de Odontologia – UniversidadeFederal de Uberlândia.

RESUMO

Este estudo teve como objetivo investigar a influência de dentifrícios à base de carvão ativado na mudança de cor (ΔE00) e rugosidade superficial (Ra) de um compósito de cor única submetido a 12 meses de escovação simulada. Cento e trinta e duas coroas bovinas foram alocadas aleatoriamente e restauradas com um dos seguintes compósitos: SS- cor única (Vittra Unique) ou multi-cor convencional, NH- nanohíbrido (Opallis), NF- nanoparticulado (Vittra APS) e BF- bulk-fill (Opus bulk-fill). Foram avaliados três dentifrícios: Controle – dentifrícios convencional (Colgate total 12), DC – dentifrícios à base de carvão (Colgate Natural Extracts) e PO – pó de carvão ativado (Whitemax). Os dentes foram submetidos a 1.217; 2.434; 7.300; 14.600 ciclos de escovação, correspondendo a T1 – 1 mês, T2 – 2 meses, T6 – 6 meses e T12 – 12 meses de escovação simulada, respoctivamente. A cor (n=11, ΔE00) e a rugosidade da superfície (n=11, Ra) foram avaliadas no início e após cada período de avaliação.O dentifrício e a superfície da restauração em resina composta foram analisadas por microscopia eletrônica de varredura (MEV). Os dados de mudança de cor e Ra foram analisados por ANOVA de medidas repetidas de três vias seguido do teste de Tukey (α=0,05). A interação entre produto de escovação*tempo foi significativa para ΔE00 (P<0,001) e tempo*produto de escovação*compósito para Ra (P<0,001). A pasta de dente à base de carvão altera a morfologia, rugosidade e cor dos compósitos de uma ou várias tonalidades de maneira semelhante à pasta de dente comum. No entanto, o pó de carvão cria irregularidades na superfície, aumentando a cor e rugosidade, com os maiores impactos na superfície do compósito nanohíbrido a partir da escovação simulada de 2 meses.

Palavras chaves: Creme dental com carvão ativado, rugosidade, mudança de cor, abrasão dentária.

ABSTRACT

This study aimed to investigate the influence of charcoal-based dentifrices on color change (ΔE00) and surface roughness (Ra) of a single-shade composite submitted to 12-month simulated brushing. One hundred and thirty-two bovine crowns were randomly allocated and restored with one of the following composites: SS- single-shade (Vittra Unique) or conventional multi-shade, NH-nanohybrid (Opallis), NF- nano-filled (Vittra APS), and BF- bulk-fill (Opus bulk-fill). Three dentifrices were evaluated: Control- conventional toothpaste (Colgate total 12), DC- a charcoal-based toothpaste (Natural Extracts), and PO- a charcoal-based toothpowder (Whitemax). The teeth were subjected to 1,217; 2,434; 7,300, and 14,600 brushing cycles, corresponding to T1- 1-month, T2- 2-month, T6- 6-month, and T12- 12-month simulated tooth brushing, respectively. The color (n=11, ΔE00) and surface roughness (n=11, Ra) were assessed at baseline and after each period of brushing time. The dentifrice and composite surface were analyzed by scanning electron microscopy (SEM). Color change and Ra data were analyzed by Three-way repeated measure ANOVA followed by the Tukey test (α=0.05). The interaction between brushing product*time was significant for ΔE00 (P<0.001) and time*brushing product*composite for Ra (P<0.001). Charcoal-based toothpaste alters the morphology, roughness, and color of single or multi-shade composites similarly to regular toothpaste. However, the toothpowder creates surface irregularities increasing color and roughness, with the greatest impacts on the nanohybrid composite surface as of 2-month simulated tooth brushing.

Key words: Activated charcoal toothpaste, roughness, color change, tooth abrasion.

INTRODUÇÃO E REFERENCIAL TEÓRICO

1- Introdução/ referencial teórico

A procura pela odontologia estética tem aumentado cada vez mais e o clareamento dental, nesta área, constitui um dos procedimentos odontológicos mais procurados (1). A aparência dos dentes está diretamente relacionada com a autoestima e as interações sociais, sendo a cor dos dentes um dos fatores mais importantes na satisfação estética desejada pelos pacientes (2).

Associado a grande procura dos pacientes por clareadores dentais, tem-se aumentado a comercialização de produtos de venda livre, ou seja, que não necessitam da supervisão do dentista para serem comprados e utilizados [3]. Os produtos de venda livre têm ganhado bastante aceitação dos pacientes por possuírem um custo menor quando comparados aos custos dos produtos tradicionais que necessitam da supervisão do cirurgião dentista (3).

Há registros muito antigos do uso de carvão mineral para saúde bucal (4), e na atualidade sua aplicação, para essa finalidade, vem ganhando cada vez mais espaço na sociedade e são encontrados com facilidade em farmácias, supermercados e sites comerciais (3) nas formas de pós ou pastas para higiene dental. As promessas dos fabricantes desses pós a base de carvão ativado vão além das propriedades de clareamento e manutenção do clareamento, eles são também indicados como remineralizadores, antimicrobianos e antifúngicos (4). Porém foi observado aumento no índice de cárie entre os indivíduos que faziam uso de pós de carvão ativado comparado aos indivíduos que faziam uso de dentifrício convencional com flúor (4,5,6), fato talvez justificado pela redução na concentração de fluoreto (6)

Além disso, o pó tem poder abrasivo que causa danos na estrutura dental e tecidos periodontais, bem como o aumento da susceptibilidade em alterar a cor dos dentes, tornando-os mais amarelados (7). Esse alteração na cor foi associada à perda de estrutura de esmalte dental e maior evidenciação da dentina (4,6). Há também relatos de que a rugosidade do esmalte, após o uso de pós de carvão mineral, é diferente daquela apresentada no esmalte que recebe escovação com dentifrício tradicional (5).

Ademais, é bastante comum a presença de restaurações estéticas em resina compostas em dentes anteriores e posteriores (8,9) e a interação de agentes clareadores associados ou não ao consumo de bebidas de baixo ph, podem causar um aumento da rugosidade desses materiais, além da abrasão causada pela escovação (10). Produtos à base de carvão mineral podem alterar a cor e a rugosidade das resinas compostas convencionais (11). Porém, recentemente foram introduzidos no mercado odontológicos resinas cor única, que facilitam o procedimento restaurador e agilizam o atendimento, pois é um material que elimina a etapa de escolha de cores, além de eliminar a necessidade de utilização de vários compósitos em um único procedimento (8). No entanto, pouco se tem descrito sobre o comportamento desses materiais frente ao uso clínico e sobre os efeitos dos produtos a base de carvão ativado na alteração de cor e na morfologia superficial dessas resinas monocromáticas, em especial, se usado a longo prazo.

Dado o crescente número de pessoas que utilizam produtos sem supervisão profissional (12) e considerando a capacidade dos dentifrícios à base de carvão ativado de produzir uma espuma cinza escura durante a escovação e a capacidade de absorção de pigmentos das resinas compostas (4,11,13,14) o objetivo deste estudo foi investigar os efeitos de dentifrícios à base de carvão na estabilidade de cor e rugosidade da superfície de um compósito de cor única submetido a 12 meses de escovação simulada. As hipóteses nulas testadas foram as seguintes: 1- os dentifrícios à base de carvão não causam mais alteração de cor ou rugosidade do que o creme dental convencional com flúor, após 12 meses de escovação simulada, e 2- não há diferença na estabilidade de cor e rugosidade da superfície de resinas convencionais se comparado com a resina monocromática.

OBJETIVOS

1- OBJETIVO

Este estudo teve como objetivo analisar, *in vitro*, o uso de dentifrício e do pó à base de carvão ativado na estabilidade de cor e rugosidade de superfície de resina composta monocromática, comparando-a com as convencionais, em simulação de uso prolongado por até 12 meses.

CAPÍTULO ÚNICO

2- CAPÍTULO ÚNICO

Artigo será submetido ao periódico Brazilian Dental Journal

Can the color stability and roughness of single-shade composite be changed by using charcoal-based dentifrices?

Abstract

This study aimed to investigate the influence of charcoal-based dentifrices on color stability (CC) and surface roughness (Ra) of a single-shade composite submitted to 12-month simulated brushing. 132 bovine crowns were randomly allocated and restored with one of the following composites: SS- single-shade (Vittra Unique) or conventional multi-shade, NH- nanohybrid (Opallis), NF- nano-filled (Vittra APS), and BF- bulk-fill (Opus bulk-fill). Three dentifrices, 1- Control- conventional toothpaste (Colgate total 12), DC- a charcoal-based toothpaste (Natural Extracts), and PO- a charcoal-based toothpowder (Whitemax) were evaluated. The teeth were subjected to 1,217; 2,434; 7,300, and 14,600 brushing cycles, corresponding to T1- 1-month, T2- 2-month, T6- 6-month, and T12- 12-month simulated tooth brushing, respectively. The color change (n=11, ΔE00) and Ra (n=11) were assessed at baseline and after each period of brushing time. The dentifrice and composite surface were analyzed by scanning electron microscopy (SEM). Color and Ra data were analyzed by Three-way repeated measure ANOVA followed by the Tukey test (α=0.05). The interaction between brushing product*time was significant for ΔE00 ($P <0.001$) and time*brushing product*composite for Ra ($P<0.001$). Charcoal-based toothpaste alters the morphology, roughness, and color of single or multi-shade composites similarly to regular toothpaste. However, the toothpowder creates surface irregularities increasing color and Ra, with the greatest impacts on the nanohybrid composite surface as of 2-month simulated tooth brushing.

CLINICAL RELEVANCE: Charcoal-based toothpaste does not influence color change and surface roughness of single-shade as well multi-shade restorations after 12-month toothbrushing, however, the charcoal toothpowder alters these parameters compromising the longevity of restorations.

KEY WORDS: Activated charcoal toothpaste, roughness, color change, tooth abrasion

Introduction

Composite resins have grown in prominence as restorative materials since their advent and are commonly employed to restore anterior(1) and posterior(2) teeth because of their excellent esthetics, reduced sound tissue removal, and acceptable longevity(1,2). The physical and mechanical properties of this restorative material have been improved, as well as its cosmetic appearance through the developments in matrix and filler technology(3). To attain sufficient esthetics while providing speedy treatment for patients, newly launched single-shade restorative materials focus on color matching between materials and dental tissues, especially in anterior restorations(3–6). The capacity of these materials to blend colors is based on color shifting as well as increased translucency. When opposed to traditional composite systems based on several shades, single-shade composites were created to make restorative treatments easier by removing the process of color selection and eliminating the need for many composites of various translucencies(3). But the color stability is still an issue, despite all of the breakthroughs in the qualities of cosmetic restorative materials. After exposure to the oral environment, composite resins are prone to discoloration (7).

Associated with the development of new restorative materials, the demand for aesthetic dentistry has increased. The appearance of teeth is directly related to self-esteem and social interactions and whiter teeth are desired by patients for aesthetic satisfaction(8). Recently, a low-cost charcoal-based whitening toothpaste and toothpowder have been popular for oral hygiene and promise to be helpful to treat discolored teeth without the need for a dentist's supervision. These products can be easy to purchase through e-commerce sites, supermarkets, and pharmacies but are scarcely any evidence of their clinical benefits(9).

The composition, mode of production, and particle size distribution of the charcoal used in the formulation are thought to affect the abrasive of charcoal-containing dentifrices(10). The charcoal in "charcoal toothpaste" is a fine powder form of activated charcoal that has been oxidized through controlled reheating or chemical processes(10,11). The more abrasive the formulation, the more efficient it will be at removing extrinsic stain and other tooth surface deposits; nevertheless, if the formulation is abrasive, it may result in a negative increasing risk of superficial damage to dental tissues(10,12,13) or increasing color change and surface wear of tooth-colored restorations(7,10,14). However, time, brushing method, and brush features all affect the mode of action of activated-charcoal-based whitening dentifrices, just as they do with any other toothpaste(14), but it was found that these dentifrices promote more optical and morphological changes on ceramic(15) and conventional multi-shade composite surface(14)

Given the increasing number of people who use these products(9) for a long period without supervision, the ability of activated-charcoal-based dentifrices to produce a dark gray foam during brushing, and the pigment absorption capacity of resin composites(10,11,14), the goal of this study was to investigate the effects of charcoal-based dentifrices on color stability and surface roughness of a single-shade composite submitted to 12-months simulated brushing. The tested null hypotheses of this study were as follows: (1) charcoal-based dentifrices do not cause more composite color change or roughness than conventional fluoride toothpaste after 12-month simulated brushing, and (2) there is no difference in color stability and surface roughness of a single-shade than multi-shade conventional composite.

Materials and methods

Experimental design

Three independent variables were evaluated: the type of dentifrice (charcoal-based toothpaste-DC or toothpowder-PO and conventional fluoride toothpaste-Control), the type of resin composite: 1-Multi-shade (conventional nanohybrid - NH, conventional nano-filled-NF, and bulk-fill nanohybrid-BF) and 2- single-shade (nano-filled-SS), and simulated brushing time (T1: 1 month; T2: 2 months; T6: 6 months; T12: 12 months). Detailed information about the materials used is shown in Table 1.

The dependent variables were color change and surface roughness. Figure 1 shows a schematic illustration of the experimental design. The main outcome considered for determining the sample size was ΔE00 considering the perceptibility color difference thresholds of 0.80(16). The required sample size was calculated to be n= 11 per group, with a power test of 0.8, and $\alpha = 0.05$. The statistical software SigmaStat v.3.5 (Systat Software Inc., Chicago, IL, USA) was used to sample size calculation. Additionally, the particles of the dentifrices were characterized by scanning electron microscopy (SEM; (Zeiss EVO MA10, Jena, Thuringia, Germany).

Specimen preparation

One hundred and thirty-two freshly extracted bovine incisors were obtained, cleaned, and stored in deionized water in a refrigerator at 4 ºC for no more than one month until the experimental stage. Double-faced diamond discs were used to remove the roots from the coronary section (KG Sorensen, Barueri, Brazil). The crowns were sectioned, the pulp chamber was filled with nanohybrid composite resin (FGM, Dentscare LTDA, Joinville, SC, Brasil), and then embedded in polyether resin (Advanced Vacuum Trade-in Composite Materials LTDA, Santo André, SP, Brasil). The buccal surface was polished and standardized by wet grinding with #600-grit #1200-, #1500-, and #2000-grit silicon carbide papers (Wetordry 3M, Nova Veneza Sumaré, SP, Brasil), and alumina suspension at 3 µm (Buehler, Lake Bluff, IL, USA). The specimens were then numbered and randomly allocated and submitted to different restorative materials (n=33) (Figure 1).

In the cervical portion of the crown, a circular preparation of 5 mm in diameter and 2 mm in depth was performed, using a wheel-shaped diamond drill (3053 – FG AllPrime, Imported by Dental Cremer, Blumenau, SC, Brazil) coupled in a cavity machine preparation. Then, enamel and dentin were prepared with 37% phosphoric acid (Ângelus Indústria de Produtos Odontológicas S/A, Londrina – PR, Brazil) for 30s and 15s, respectively, then washed with distilled water per 30s and dried. Then, a single-bottle adhesive system (Ambar APS, FGM, Joinville – SC, Brazil) was applied and photoactivated for 20 s with a multiple peak light curing device (Valo Cordless, Ultradent, South Jordan, Utah, USA), with an irradiance of 1000 mW/cm^2 in standard mode. After inserting one of the tested composites in the prepared cavity (NH, NP, BF, or SS), a Myler strip was pressed over resin with a glass slab to obtain a flat surface. The composite was photoactivated using the same photoactivation protocol described above, perpendicular and at the shortest possible standardized position between the tip and the tooth. The Myler strip and glass slab were removed followed by additional light activation for 20 s. Then, after 24h, the specimens were polished with alumina suspension at 6 µm, 3 µm, 1 µm, and, 1/4 µm (Buehler, Lake Bluff, IL, USA).

Restored teeth were divided into three groups (n = 11), according to the dentifrice: 1- conventional fluoride dentifrice with no charcoal- Control (Colgate Total 12, Colgate-Palmolive, New York City, NY, USA.), 2- Charcoal-based toothpaste- DC (Colgate Natural Extracts) and 3- Charcoal-based toothpowder (Whitemax, Dermavita, Brusque, SC, Brazil).

The randomization process (blocked random scheme) was carried out using the website *www.sealedenvelope.com*. The identification of the treatment to be applied on samples followed the sequentially-numbered previously. Randomization and allocation were made by the same researcher (GRS), who was not involved in the implementation (MGC) and evaluation process (PANS). Because they didn't know the coding method, those who assessed color and roughness measures (LD) and statically evaluated the data (GRS) were blinded.

Brushing protocol

The specimens were submitted to mechanical brushing cycles in a toothbrushing machine (Odeme Dental Research, Luzerna, SC, Brazil) which was performed with vertical loading of 300g over the soft-bristle toothbrush heads (Colgate-Palmolive, São Paulo, SP, Brasil), temperature-controlled (25 ± 1°C) executing a linear motion at 2Hz (120 cycles/min). All specimens were immersed in a slurry prepared with toothpaste or charcoal powder and purified water and artificial saliva, in a 1:1:1 (w/v) proportion. Given that 73,000 mechanical brushing cycles correspond to 5 years of brushing exposure for a healthy person(17), the following brushing times were simulated: T1- 1-month, T2- 2-months, T6- 6-months, and T12- 12-months, with the specimens subjected to 1,217; 2,434; 7,300, and 14,600 brushing cycles, respectively. Every 300 cycles, the slurry created for each type of dentifrice was replaced. Before the brushing simulation, baseline values (T0) for color measurement and roughness were calculated, and after each simulated brushing period the specimens were ultrasonically washed for 10 minutes and re-evaluated for roughness and color. The samples were stored in artificial saliva (1.5 mM calcium [$CaCl_2$], 0.9 mM phosphate [NaH_2PO_4], 0.15 mM KCl, pH 7.0- Pharmus, Uberlândia – MG, Brasil) at 4ºC between the assessment time.

Surface roughness

At baseline (T0) and after each simulated period of toothbrushing (T1; T2; T6 and T12) the surface roughness was measured using a roughness meter (Mitutoyo, Aurora, IL, USA). With a static load of 5 N and a speed of 0.05 mm/s, the mean roughness (Ra) was measured. In sequential mode, the cutoff value was 0.25 m, and the measurement distance was 1 mm. For each specimen, three readings were taken from the surface's center, and the arithmetical mean was determined.

Color Analysis

The specimens` color was assessed with a visible/ultraviolet reflection spectrophotometer (Ci64UV, X-Rite, Grand Rapids, MI, USA). The device has an aperture diameter of 4 mm, and the readings were carried out with a 2° observer angle and illuminant D65. The coordinates of the LAB system from the Commission Internationale de L'Eclairage (CIE) were recorded. This system is based on the lightness (coordinate L*) and the chromaticity coordinates a* (red-green axis) and b* (yellow-blue axis). The color was measured at baseline (T0) and after T1; T2; T6 and T12 by placing the surfaces at the same position for all measurements.

The color measurements were performed in triplicate over a white background (ColorChecker grayscale, X-Rite, Grand Rapids, MI, USA, L*white = 95.2, a*white = 21.2, b*white = 50.3), and the mean values were used for data analysis. Overall color changes from baseline compared with other assessment times (T1; T2; T6 and T12) were calculated using the CIEDE2000 color difference (ΔE00) calculated as follows: ΔE00= [(ΔL/KLSL)2 + (ΔC/KCSC)2 + (ΔH/KHSH)2 + RT (ΔC/KCSC) (ΔH/KHSH)] 1/2 where ΔL, ΔC, and ΔH are considered lightness, chroma, and hue differences between color measurements. KL, KC, and KH are the parametric factors for viewing conditions and illuminating conditions influence. RT is the function for hue and chroma differences interaction in the blue region. SL, SC, and SH are the weighting functions for color difference adjustment considering the location variation of L*, a*, and b* coordinates(18).

Surface Morphology

The morphology of the composite resin surface was examined before and after 12-months of simulated charcoal-based toothpowder brushing. The specimens were subjected to vacuum in a sputter coater (SCD 050 Sputter Coater, Capovani Brothers Inc, New York, USA) to deposit a thin layer of gold before submitting to a scanning electron microscope (SEM) (Zeiss EVO MA10, Jena, Thuringia, Germany) at 2000X magnification. Moreover, approximately 1g of each toothpaste and toothpowder was dried at 37^{o}C in an incubator for 7 days. The dried materials were placed on stubs and sputter-coated with gold (SCD 050) followed by SEM analysis at 2000X.

Statistical analysis

Repeated-measures ANOVA and Tukey's tests were used to compare the roughness and the color parameter (ΔE00) to composites. The 'brushing time' was considered as a repetition factor. Statistical analysis was performed using Jamovi 2.0 statistical software package (dev.jamovi.org). The significance level was set at α = 0.05 for all data analyses.

Results

The mean and standard deviations of ΔE00 to composite resins are shown in table 2. A significant difference was verified on interaction between brushing product and brushing time (P <0.001) for color change (ΔE00) regardless of the composite resin (P=0.156). The color change of composites was similar when using charcoal-based and conventional toothpaste. After toothpowder brushing (PO) to T2, the color shift for all composites was considerable, but they reached values above the acceptable level (E00>1.77)(16) at T6.

Figure 2 shows the CIELAB parameters of composite resins. L* is the parameter that usually represents the major concern from an aesthetic standpoint (darkness to lightness). All composite resins tended of decreasing L values using PO over brushing time.

The composite roughness average mean and standard deviations are present in figure 3. Significant difference were verified on interaction between time*product (P<0.001), composite*product (P<0.001), time*brushing product*composite (P<0.001) for roughness (Ra). PO caused higher Ra than toothpaste (control or DC) on the resin surface. The roughness increases at T2 for nanohybrid and at T6 for the other composites. Single-shade composites showed similar Ra compared to conventional nano-filled and bulk-fill. After 12-months, NH NF and SS composites brushed with charcoal toothpowder were above the Ra threshold of 0.2 μm(19).

The photomicrographs of particles in the dentifrices are shown in Figure 4. The toothpaste (control and DC) showed irregularly shaped particles and spherical-shaped nanoparticle clusters. Abrasive particles with sharp angles, irregular appearance, and porosities were present in PO and they were larger particles than other dentifrices.

SEM images revealed surface alterations in all composites after 12-months of simulated brushing with toothpowder, however, the NH had more irregularity than the other restorative materials (Figure 5).

Discussion

According to the results, charcoal-based toothpowder causes more composite color change and surface roughness than conventional or charcoal-based toothpastes after simulated brushing, which led to the rejection of the first null hypothesis of the study. However, there were no differences observed between the two toothpastes. This suggested that the charcoal-based toothpaste tested did not induce further color change of the composite surface than that produced by the mechanical and chemical action of the regular fluoride toothpaste. Thus, despite the fact that the dark color of the material is similar to that of other pigmenting agents such as wine, black tea or coffee(20), these toothpastes may not be linked to an increased risk of making the composite resin darker in color after 12-months brushing. Controversially, a previous study (14) reported that charcoal-based toothpaste resulted in greater color change than conventional dentifrices. These contradictory results could be explained by differences in the tested formulations and methodology. Our samples had less baseline roughness as well as the cleaning ultrasonically vibration after brushing may have reduced the amount of charcoal-stain remained on the composite surface. As a result, the grey component of charcoal-based toothpaste had less impact on color change.

As well as color, no significant difference was found for composite surface roughness using toothpastes. The activated charcoal used in toothpastes is a fine powder that varies in abrasiveness according on its source, production method, and particle size, shape, and hardness. (21). When the SEM images were evaluated, a similar pattern of abrasive particles was observed between the toothpastes, which justifies similar behavior regarding abrasiveness, resulting in similar morphological pattern and surface roughness of composites. In this context, also has been shown in a previous study that charcoal-based toothpastes do not necessarily cause more abrasion on enamel than conventional toothpaste (12,14,22).

Visual thresholds are important qualitative indicators in dentistry for evaluating and interpreting clinical outcomes and comparing different treatments. After two-month period of charcoal-powder brushing was able to causes higher color changes than toothpastes, reaching values above the clinical acceptability threshold ($\Delta E00>1.77$)(16) at six months. Composite resins are restorative materials that are prone to color change as the organic matrix absorbs water, making them more sensitive to staining agents penetration. All composites tended of decreasing L values over brushing time which indicate that the using toothpowder more than to 1 month may lead to the need to replace the anterior restorations due to complaints about the esthetics in the smile zone. Similarly, surface roughness increased for almost all composites (NH, NP and SS), being considered above threshold roughness value of 0.2 μm after six months(19). Rough surfaces results in the accumulation of plaque and stains which may lead to discoloration and secondary caries(7). These data corroborate with other studies that reported greater composite wear and tear by toothpowder(7,14).

SEM analyses showed that the toothpowder had larger and porous particles with angular shape which visually differs to spherical nanoparticles and nanoclusters presented in both toothpastes. As a result, the increasing activated charcoal content, particle shape, and size of toothpowder may have been able to increase roughness on composite surfaces. Moreover, the lower abrasive behavior from the toothpastes may also be resultant from their formulations including water, xanthan and cellulose-based gums, carboxymethylcellulose which may present anti-erosive properties(23).

The current study confirmed that morphological composite surface changes are material(19,24) and aging-time dependent(14,17). Thus, the second null hypothesis of this study also was rejected. The composite matrix composition, matrix/particle interface, particle shape and size, degree of polymerization, and hardness of resin composites could all affect abrasion resistance(25). The conventional nanohybrid, with larger filler size, presented an increasing in mean surface roughness after 2-months simulated brushing with toothpowder. The Ra values was two time of clinical threshold(19) at 12-months. At analyzing the SEM images, holes and scratches are evident on the surface of the nanohybrid resin. The larger and more protruded the filler particles are, the more the energy generated by abrasion processes is transmitted directly to the surrounding matrix, causing microcracks to propagate and cause particle detachment, thereby increasing the roughness and potentiating the restoration wear process even more(25). Surface roughness is quite important from a clinical standpoint. Surface roughness can enhance biofilm formation/ buildup, causing irreversible damage to tooth hard tissues and restoration surfaces, because it has a large impact on bacterial adherence. Surface roughness can also cause gingival recession, periodontal inflammation, dentin hypersensitivity, and the accumulation of oral pigments, all of which can affect the appearance of enamel and restoration margins(14,24).

Remarkable, despite of differences among corresponding translucency, fillers and matrix characteristics and compositions, similar outcomes were obtained to both single-shade and conventional multi-shade nano-filled and bulk-fill composites. Regarding the reached outcomes, the incorporation of nanofillers, improves the abrasive resistance, promoting a higher filler loading with consequent protection to softer matrix, which reduce the interparticle spacing and, at same time, enhancing the potential the material to abrasive effect. After 12-month simulated toothpowder brushing, only bulk-fill composite presented Ra values below threshold of 0.2 μm(19) but they had not absolute value difference compared with the other composites.

There were some limitations to this investigation. One of these was a lack of accurate information regarding the compositions, physical, and mechanical qualities of dentifrices and composites; it was impossible to tell the percentage of each component or even if any component was not listed. This information was only available in scientific journals, and only in a limited way. Also, laboratory tests not simulating all oral environment conditions.

Toothpowder associated with acid and temperature changes, brushing force and occlusal wear could be more damage to resin composite and these aspects should be addressed in future investigations.

However, the clinical relevance of the study is demonstrated by the interaction of charcoal-based toothpowder, and the color and surface roughness changes that occurred similarly in multi-shade nanohybrid and nano-filler and single-shade composite resins. Moreover, this brushing product used for a long time may lead to more destruction, especially in nanohybrid restorations than was shown for the conventional toothpaste used such as negative control. Therefore, its use in oral hygiene is discouraged.

Conclusion

Brushing using charcoal-based toothpowder negatively increases the color change and roughness of single-shade as well as multi-shade composites as of 2-month tooth brushing. In this perspective, conventional nanohybrid composite shows the worst roughness scenario. However, after 12-month, the charcoal-based toothpaste had no greater effect on the composite than regular toothpaste.

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Figure Caption

Figure 1. Schematic illustration of the experimental design.

Figure 2. Graphs show the trends in the L*, a* and b*parameters of the composite resins over brushing time (L*: white/black; a*:red/green; b*: yellow/blue). Control: mechanical brushing with conventional fluoride dentifrice; PO: mechanical brushing with charcoal-based toothpowder; DC: mechanical brushing with charcoal-based toothpaste.

Figure 3. Means (±standard deviation) of composite roughness average (μm) after brushing products and times (n=11): T1- 1-month, T2- 2-months, T6- 6-months, and T12- 12-months. *Different letters (lowercase for comparing the brushing times for composite*brushing product; uppercase for comparing brushing products for composite*time; symbol for comparing composites for brushing product*time) indicate significant difference at Tukey`s test (P<0.05). *Threshold roughness* [19]

Figure 4. Representative Scanning microscopy images of brushing products. A- Conventional fluoride dentifrice (Control - Colgate total 12, Colgate-Palmolive). B- Charcoal-based dentifrice (DC- Colgate Natural Extracts, Colgate-Palmolive). C-Charcoal-based toothpowder (PO- WhiteMax).

Figure 5. Scanning electron microscope (SEM) of composites surface before (T0) and after 12-month simulated brushing (T12) at 2000X. NH. Conventional nanohybrid, A- (T0) and E- (T12); NF. Conventional nano-filled, B- (T0) and F- T12); SS. Single-shade, C- (T0) and G (T12); BF. Bulk-fill, D-(T0) and H- (T12).

Table 1 - Composition of materials used in this study (manufacturer data)

Material	*Composition*	*Manufacturer*
Colgate total 12 (Control)	Glycerin, aqua, hydrated Silica, Sodium Lauryl Sulfate, arginine, aroma, cellulose Gum, zinc oxide, poloxamer 407, zinc citrate, tetrasodium pyrophosphate, xanthan gum, benzyl alcohol, cocamidopropyl betaine, Sodium Fluoride (1500 ppm F−), sodium saccharin, and sucralose.	Colgate-Palmolive, São Paulo, SP, Brasil
Colgate Natural Extracts (Charcoal-based dentifrice - DC)	Aqua, glycerin, hydrated silica, sodium lauryl sulfate, aroma (peppermint oil), cellulose gum, xanthan gum, sodium fluoride (1450 ppm F−), sodium saccharin, charcoal powder, benzyl alcohol, eugenol.	
WhiteMax (Charcoal-based powder - PO)	Activated charcoal powder, Kaolin, Citrus aurantium dulcis peel oil,Aroma.	Dermavita, Brusque, SC, Brasil
Nanohybrid composite (Opallis) (NH)	Active Ingredients: Bis-GMA (Bis-Phenol A di-Glycidyl Methacrylate), BisEMA (Bis-Phenol A di-Glycidyl Methacrylate) Monomers, TEGDMA (Triethylene Glycol Dimethacrylate), UDMA (Urethane Dimethacrylate), Camphorquinone, Co-initiator and silane. Inactive ingredients: silanized barium-aluminum silicate glass, pigments and silica. Shade A2.	
Nano-filled (Vittra APS) (NF)	Active ingredients: monomeric matrix containing UDMA (Urethane Dimethacrylate) and TEGDMA (Triethylene Glycol Dimethacrylate) type monomers, photoinitiator composition (APS), co-initiators, stabilizer and silane. Inactive ingredients: zirconia filler, silica and pigments. Shade A2.	FGM, Dentscare LTDA, Joinville – SC, Brasil
Single-shade nano-filled composite (Vittra Unique - SS)	Active ingredients: mixture of methacrylate monomers, photoinitiator composition (APS), co-initiators, stabilizers and silane. Inactive Ingredients: barium-aluminium-silicate glass.	
Nanohybrid bulk fill (Opus Bulk fill) (BF)	Active Ingredients: Urethanedimethacrylic monomers, stabilizers, photoinitiators and co-initiators. Inactive Ingredients: Inorganic fillers of silanized silicon dioxide (silica), stabilizers and pigments. Shade A2.	

Table 2 - Means (±standard deviation) of composite resin ΔE00 comparing brushing times and dentifrices (n=11).

Composite resin	*Dentifrices*	*Brushing time*											
		T1			*T2*			*T6*			*T12*		
NH	Control	0.87 ±	0.4	Aa	0.90 ±	0.5	Aa	1.01 ±	0.5	Aa	0.65 ±	0.3	Aa
	PO	0.74 ±	0.4	Aa	1.47 ±	6.0	Bb	2.83* ±	1.0	Bb	2.30* ±	0.6	Bb
	DC	0.84 ±	0.3	Aa	1.19 ±	0.3	Aa	1.07 ±	0.6	Aa	1.08 ±	0.4	Aa
NF	Control	0.66 ±	0.5	Aa	0.53 ±	0.2	Aa	0.77 ±	0.5	Aa	0.75 ±	0.3	Aa
	PO	1.40 ±	0.9	Aa	1.73 ±	1.0	Bb	2.64* ±	1.1	Bb	3.05* ±	1.0	Bb
	DC	0.76 ±	0.7	Aa	0.87 ±	0.6	Aa	0.97 ±	0.6	Aa	0.93 ±	0.4	Aa
SS	Control	0.86 ±	0.5	Aa	0.77 ±	0.5	Aa	0.88 ±	0.5	Aa	0.89 ±	0.5	Aa
	PO	0.54 ±	0.3	Aa	1.27 ±	0.6	Bb	2.22* ±	1.1	Bb	2.67* ±	1.0	Bb
	DC	0.59 ±	0.4	Aa	0.60 ±	0.4	Aa	0.73 ±	0.4	Aa	0.83 ±	0.6	Aa
BF	Control	0.63 ±	0.5	Aa	0.85 ±	0.5	Aa	0.90 ±	0.6	Aa	1.06 ±	1.1	Aa
	PO	0.59 ±	0.4	Aa	0.87 ±	0.7	Bb	1.78* ±	0.8	Bb	2.30* ±	0.9	Bb
	DC	0.52 ±	0.4	Aa	0.53 ±	0.7	Aa	0.74 ±	1.2	Aa	0.90 ±	1.1	Aa

Simulated brushing times: T1: 1 month; T2: 2 months; T6: 6 months; T12: 12 months. Control: Colgate Total 12 (Colgate-Palmolive); PO- charcoal-based toothpowder - WhiteMax (Dermavita) and DC- charcoal-based toothpaste - Colgate Natural Extracts (Colgate-Palmolive). Different letters (lowercase for comparing the brushing times - in line; uppercase for comparing brushing products – in columns) indicate significant differences at Tukey`s test (P<0.05). * Color changes beyond the acceptability threshold (ΔE00=1.77)

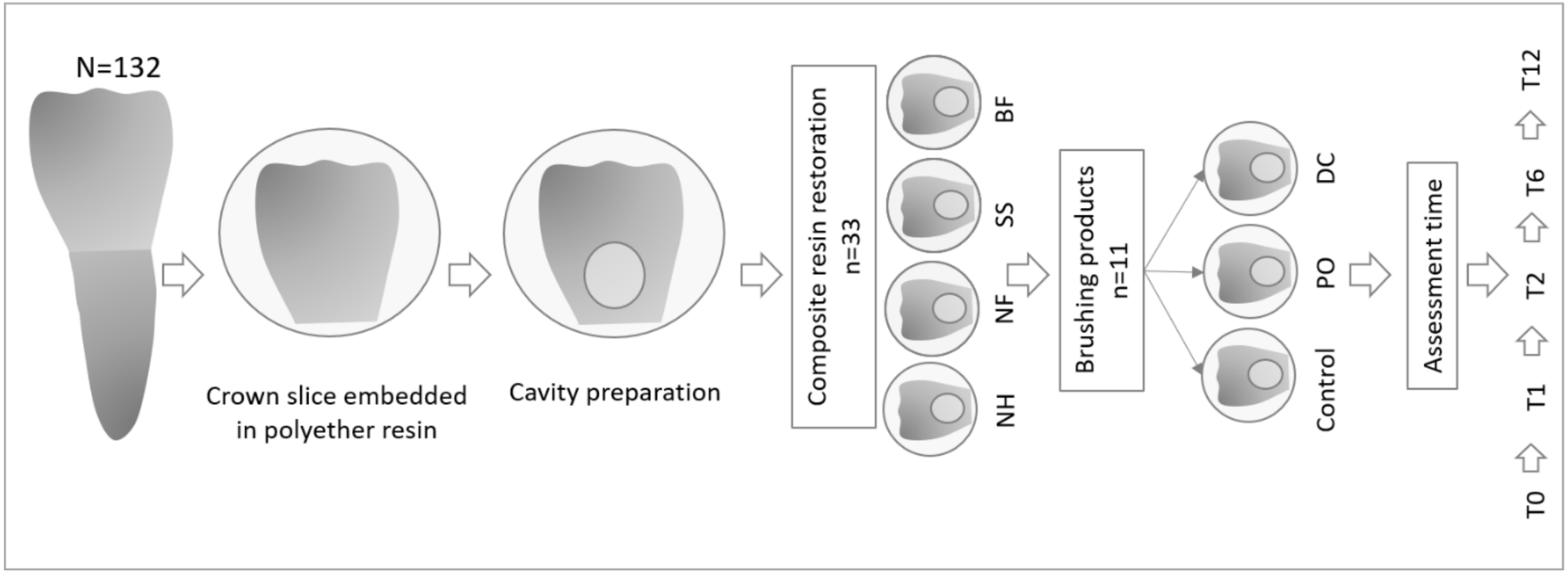
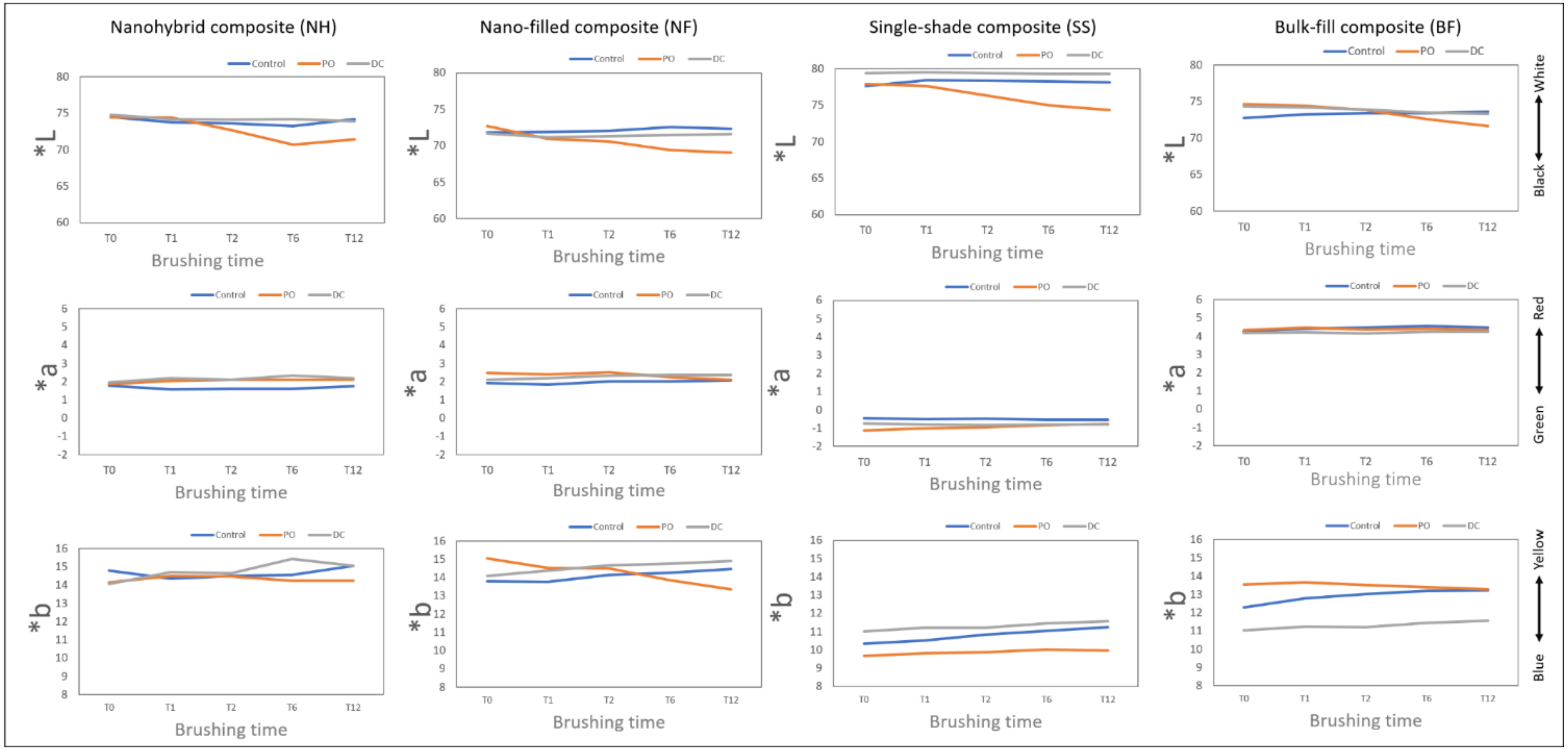


Figure 1. Schematic illustration of the experimental design.



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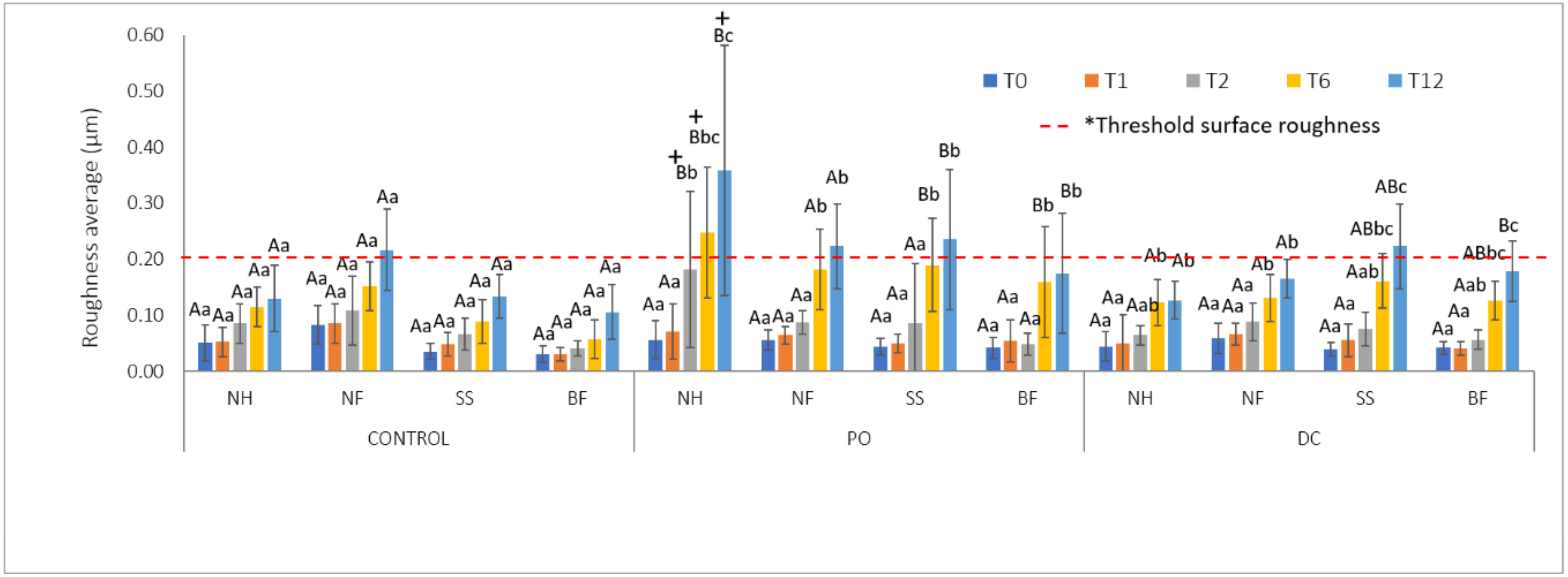

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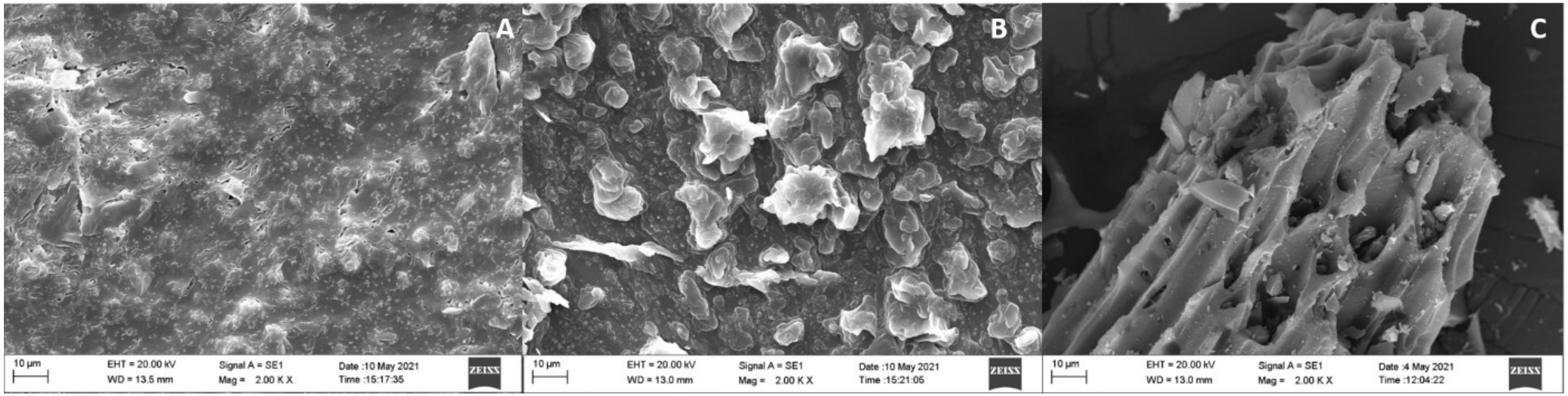

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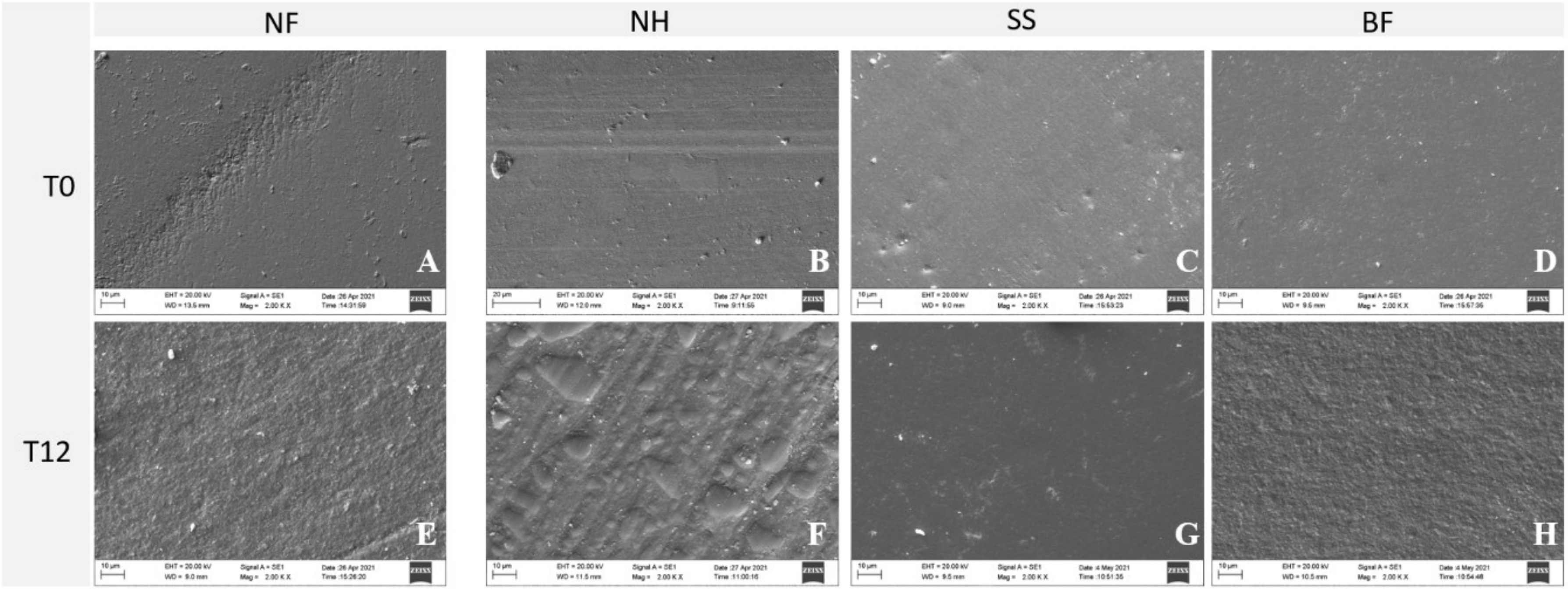

Figure 5. Scanning electron microscope (SEM) of composites surface before (T0) and after 12-month simulated brushing with activated carbon powder (T12) at 2000X. NH. Conventional nanohybrid, A- (T0) and E- (T12); NF. Conventional nano-filled, B- (T0) and F- T12); SS. Single-shade, C- (T0) and G (T12); BF. Bulk-fill, D-(T0) and H- (T12).

CONCLUSÃO

Conclusão

De acordo com o delineamento experimental proposto e a metodologia utilizada, pode-se concluir que:

- A partir de dois meses de escovação, o pó de carvão ativado altera negativamente a cor e rugosidade de resinas compostas monocromática e convencional.
- A resina nanohíbrida convencional apresentou o maior aumento na rugosidade superficial quando escovada com pó de carvão ativado.
- Após 12 meses de escovação simulada, o creme dental a base de carvão ativado apresentou efeito similiar àquele apresentado pelo dentifrício convencional fluoretado.

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