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# Effect of charcoal-containing toothpastes on roughness and color stability of bulk-fill resin composites

Efeito de dentifrícios contendo carvão na cor e rugosidade de resinas compostas bulk-fill

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# ABSTRACT

Objective: To evaluate the effects of different commercially available charcoal-based toothpastes (CBTs) on the roughness and color of bulk-fill resin composites (RCs). Materials and Methods: Disc-shaped samples (6 × 2 mm) were made with nanofilled (NF) bulk-fill (Filtek One, 3M Oral Care) or nanohybrid (NH) bulk-fill (Aura, SDI) RCs. The analyses were performed initially (baseline) and after 10,000 brushing cycles in a toothbrushing machine using (n=10): regular toothpaste (Colgate Total 12, Colgate-Palmolive) or three types of CBTs (Colgate Luminous White Activated Charcoal - Colgate-Palmolive; Black is White - Curaprox; 3D White Mineral Clean - Oral-B). The specimens were analyzed for roughness (Ra,  $\mu$ m) and quantified by coordinates of the CIEL\*a\*b\* color space, Vita Classical scale (shade guide unit, SGU), and general color alteration ( $\Delta E_{ab}$ ;  $\Delta E_{00}$ ). The data were evaluated using generalized linear models (Ra, L\*, b\*,  $\Delta E_{ab}$ ;  $\Delta E_{00}$ ), Mann-Whitney, Kruskal-Wallis, and Dunn tests (a<sup>\*</sup>;  $\Delta$ SGU), with  $\alpha$ =0.05. **Results:** Regardless of the toothpaste, Ra increased after brushing, but was significantly higher in NH than NF (p=0.0001). L\* significantly decreased after brushing with Black is White toothpaste (p=0.0027). NF showed higher  $\Delta E_{00}$  values after brushing with the CBTs, compared with regular toothpaste. Moreover, NH exposed to Black is White exhibited higher  $\Delta E_{ab}$  and  $\Delta E_{00}$  values than the other toothpastes (p < 0.0001). Conclusion: The roughness alteration was not mediated by the type of toothpaste. However, the CBTs were able to change the optical properties of bulk-fill RCs, with more pronounced effects, as observed with Black is White.

# **KEYWORDS**

Activated charcoal; Color; Composite resins; Toothbrushing; Toothpastes.

# **RESUMO**

**Objetivo:** Avaliar os efeitos de diferentes dentifrícios contendo carvão (DCCs) na rugosidade e cor de resinas compostas (RCs) bulk-fill. **Material e Métodos:** Amostras cilíndricas (6 × 2 mm) foram confeccionadas com as RCs nanoparticulada (NP) bulk-fill (Filtek One, 3M Oral Care) e nano-híbrida (NH) bulk-fill (Aura, SDI). As análises foram realizadas nos tempos: inicial (baseline) e após 10000 ciclos de escovação em máquina simuladora utilizando (n = 10): dentifrício regular (Colgate total 12, Colgate-Palmolive) e DCCs (Colgate Luminous White Carvão Ativado - Colgate-Palmolive; Black is White - Curaprox; 3D White Mineral Clean - Oral-B). As amostras foram analisadas quanto à rugosidade (Ra,  $\mu$ m) e quantificadas nas coordenadas do sistema CIEL\*a\*b\*, na escala Vita Classical (SGU), e em valores de alteração geral da cor ( $\Delta$ Eab;  $\Delta$ E00). Os dados foram avaliados por modelos lineares generalizados (Ra, L\*, b\*,  $\Delta$ Eab;  $\Delta$ E00), testes de Mann Whitney, Kruskal-Wallis e Dunn (a\*;

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 $\Delta$ SGU), com  $\alpha$ =0,05. **Resultados:** Independentemente do dentifrício, a Ra aumentou após a escovação, mas com valores significativamente maiores para a NH do que para a NP (p = 0,0001). Os valores de L\* diminuíram significativamente (p = 0,0027) após escovação com Black is White. NP mostrou maiores valores de  $\Delta$ E00 após escovação com os DCCs comparada ao dentifrício regular. Adicionalmente, NH exibiu maiores valores de  $\Delta$ Eab e  $\Delta$ E00 quando exposta ao dentifrício Black is White comparada aos outros dentifrícios (p < 0,0001). **Conclusão:** A alteração de rugosidade não foi mediada pela tipo de dentifrício. Entretanto, os DCCs foram capazes de alterar as propriedades ópticas das RC bulk-fill, com efeitos mais potencializados com o dentifrício Black is White.

# PALAVRAS-CHAVE

Carvão ativado; Cor; Cremes dentais; Escovação; Resinas compostas.

# INTRODUCTION

Charcoal is a carbon compound produced by burning materials, such as coconut shells, nutshells, bamboo, animal bones, and other sources [1]. It has been used for medical purposes since Ancient Greece [2,3]. Currently, charcoal or activated charcoal has been used in dentistry, marketed as toothpaste or powder for toothbrushing, primarily due to its purported whitening properties [3] enabled by its potential capacity to adsorb pigments [4]. Nevertheless, this whitening effect has not been reported in previous studies [5-8]. Many digital influencers advertise these products, and encourage their followers to purchase them. Such products are often available on e-commerce platforms at lower prices, and can easily be obtained. In recent years, well-established oral care manufacturers have also introduced their versions of products containing whitening agents [1,9,10], although there are few investigations regarding their safety or effectiveness.

As a result, some adverse effects caused by these products have already been reported, such as abrasive tooth wear, increased dentin hypersensitivity, and degradation of restorative materials [3,8,11]. Thus, just like common abrasives, charcoal can have abrasive properties that vary among manufacturers [1], or that can even involve non-controlled manufacturing of its particles. Among the toothpastes commercially available to consumers, whitening toothpastes containing conventional abrasives can alter roughness [12], and cause color changes in conventional resin composites, depending on the type of toothpaste and resin composite [13-15]. However, studies investigating the effects of charcoal-based toothpastes on the properties of bulk-fill resin composites are very scarce.

Bulk-fill resin composites have emerged on the market to reduce the effects of polymerization

shrinkage associated with incremental techniques, thereby also reducing clinical time by increasing the depth of cure, and enabling the insertion of larger increments [16,17]. Categorically, bulk-fill composites can be: I) applied as a restoration base that must be overlaid with a conventional resin composite; or II) applied as a single body (fullbody), used in a single increment (4-5 mm) [16]. High-viscosity bulk-fill composites represent resin composites with the highest inorganic content, microhardness, and wear resistance [16,17], thus providing them with adequate properties for exposure to the oral environment, and for use in direct restorations in posterior teeth. The good clinical performance of specific resin-based materials requires enhancing the parameters that mediate clinical success over time, such as the surface characteristics, and the color stability of the material and restoration [18]. However, there is a lack of investigations into the resistance of these bulk-fill resin composites to roughness and color alterations resulting from exposure to conventional or charcoal-containing toothpastes.

Therefore, studies are needed to investigate the possible effects of toothpastes containing charcoal on hygiene and oral health, and on the properties of dental hard tissues and dental materials. The foremost concern warranting further discussion is the safety of these products, especially because consumers are using these charcoal-based products without knowing the risks to the oral environment [19]. Thus, the null hypotheses were that toothpastes containing charcoal do not differ from conventional/regular toothpaste regarding 1) surface roughness and 2) color change, and that (3) the nanohybrid and nanofilled bulk-fill resin composites studied do not differ when brushed with the same type of toothpaste.

# **MATERIAL & METHODS**

# Study design

The present study was designed using a factorial scheme, considering the following factors: I) resin composite (experimental unit,  $\emptyset$  6 mm; n = 10): nanofilled bulk-fill resin composite (Filtek One Bulk-Fill, 3M Oral Care), and nanohybrid bulk-fill resin composite (Aura Bulk-Fill, SDI); II) toothpaste: regular toothpaste containing either silica (Total 12 [RT], Colgate-Palmolive – pH: 8.29); or charcoal (Black is White [BWC], Curaprox – pH: 6.64; Luminous White Activated Charcoal [LWC], Colgate-Palmolive - pH: 7.88; and 3D White Mineral Clean [WMC], Oral-B – pH: 7.60); and III) time points: initial (baseline) and after cycling in the brushing machine (10,000 cycles). The dependent variables were surface roughness (Ra) and color (Vita Classical, L\*, a\*, and b\* coordinates,  $\Delta E_{ab}$ and  $\Delta E_{00}$ ).

Sample size was calculated using G\*Power 3.1.5 software (Heine, Universität Dusseldorf, Germany), considering the following parameters:  $\alpha = 0.05$ , 1- $\beta = 0.9$ , and size effect and standard deviation calculated according to a pilot study (n = 3). The results indicated that 7 samples would be needed to determine the roughness variable ( $\Delta$ Ra), and 3 samples for the color analyses ( $\Delta$ E<sub>00</sub>). Therefore, the study was conducted considering n = 10.

# Sample preparation

Filtek One Bulk-Fill (3M Oral Care) and Aura Bulk-Fill (SDI) resin composites were inserted in a single increment in a rubber elastic matrix ( $6 \times 2$  mm), over a microscope glass slide. After insertion, the specimen was covered with a mylar strip and another microscope glass slide. Next, a 500 g weight was applied over it for 10 seconds to remove any bubbles that might have been created during the increment insertion. The specimen was then light-cured using an LED device (Valo, Ultradent, South Jordan, UT, USA) for 20 seconds (standard mode, 1000 mW/cm<sup>2</sup>). After the specimen was cured, it was removed from the matrix, and the excess was removed with a scalpel blade. The composition and classification of the evaluated resin composites are presented in Table I.

# Experimental groups and simulated brushing

The specimens were submitted to initial color and roughness analyses, and then to simulated brushing in an automatic machine (MEV 3T – 10 XY, Odeme Dental Research, Luzerna, SC, Brazil) with the toothpastes presented in Table II. The specimens were fixed individually in the brushing machine with a hot-melt adhesive, so that their surface remained parallel to the toothbrushes (Oral-B Indicator 40 soft, Gillette do Brasil, Manaus, AM, Brazil). Each specimen was randomly assigned to a group, and exposed individually to a certain toothpaste.

Brushing was performed with 10 mL of the slurry created by the mixture of the toothpaste and distilled water, in a 1:3 ratio by weight, with a 200 g axial load, in a zig-zag pattern (150 oscillations/min). The specimens were submitted to 10,000 cycles, equivalent to one year of use of the toothpaste [20]. After the specimens were subjected to the brushing cycling, they were rinsed in distilled water for 30 seconds, stored, and once again submitted to color and roughness analysis.

# Surface roughness analysis

Surface roughness analysis of the specimens was performed using a roughness tester (Surftest SJ-210, Mitutoyo Corporation, Kanagawa, Japan) at the following time points: initial (baseline) and after automatic brushing. Each reading

Table I - Composition, classification, and manufacturers of resin composites<sup>1</sup>

Resin composite	Classification	Manufacturer	Composition
Filtek One Bulk- Fill Shade: A1	Nanofilled Bulk-fill	3M, St. Paul, Minnesota, USA	Aromatic urethane dimethacrylate, UDMA, DDDMA, water, silane treated ceramic (60-70% in weight); silane treated silica (1-10% in weight); silane treated zirconia (<5% in weight), ytterbium fluoride.
Aura Bulk-Fill Shade: BKF (universal)	Nanohybrid Bulk-fill	SDI, Bayswater, Victoria, Australia	UDMA, Bis-EMA, Bis-GMA, TEGDMA, amorphous SiO <sub>2</sub> , barium aluminosilicate glass, prepolymerized filler particles.
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<sup>1</sup> The composition is presented in the MSDS (Material Safety Data Sheet) provided by the manufacturers. Abbreviations: UDMA, urethane dimethacrylate; DDDMA, 1,12-dodecanediol dimethacrylate; Bis-EMA, bisphenol-A hexaethoxylated dimethacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate.

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Toothpaste	Code	Classification	Manufacturer	Composition
Colgate Total 12 pH: 8.29	RT	Regular toothpaste	Colgate-Palmolive (São Paulo, Brazil)	Sodium fluoride (1450 ppm), cellulose gum, zinc oxide, poloxamer 407, tetrasodium pyrophosphate, zinc citrate, benzyl alcohol, cocamidopropyl betaine, xanthan gum, sodium saccharin, phosphoric acid, sucralose, titanium dioxide (CI 77891)
Curaprox® Black is White pH: 6.64	BWC	Charcoal- containing toothpaste	Curaprox (Kriens, Switzerland)	Sodium monofluorophosphate (950 ppm), water, sorbitol, glycerin, hydrated silica, charcoal powder, flavor (aroma), decyl glucoside, cocamidopropyl betaine, tocopherol, mica, xanthan gum, hydroxyapatite, 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 lemon peel oil, citric acid, lactoperoxidase, glucose oxidase, amyloglucosidase, tin oxide, sodium bisulfite, hydrogenated lecithin, limonene, CI 75810, CI 77289
Colgate® Luminous White Activated Charcoal pH: 7.88	LWC	Charcoal- containing toothpaste	Colgate-Palmolive (São Paulo, Brazil)	Sodium monofluorophosphate (1000 ppm), CI 77266 (charcoal powder), CI 16035, CI 42090, CI 19140, water, hydrated silica, sorbitol, calcium pyrophosphate, glycerin, peg-12, pentasodium triphosphate, tetrapotassium pyrophosphate, flavor (aroma), sodium lauryl sulfate, cellulose gum, sodium saccharin, xanthan gum, cocamidopropyl betaine, limonene
Oral-B® 3D White Mineral Clean pH: 7.60	WMC	Charcoal- containing toothpaste	Procter & Gamble (São Paulo, Brazil)	Sodium fluoride (1100 ppm), water, sorbitol, hydrated silica, disodium pyrophosphate, sodium lauryl sulfate, cellulose gum, sodium hydroxide aroma, sodium saccharin, carbomer, charcoal powder, mica, limonene, sucralose, dioxide titanium 80, polysorbate

Table II - Information on commercial toothpastes used in the present study according to the manufacturer<sup>1</sup>

<sup>1</sup>The slurry pH was evaluated in triplicate using a pH meter (MPA 210, MS Tecnopon Instrumentação, Piracicaba, Brazil).

presented an average roughness (Ra,  $\mu$ m) with a 0.25 mm cut-off, at a 0.25 mm/s speed, and a distance length of 1.25 mm. Three readings were performed on each surface; the needle always passed through the geometric center of the specimen in three different positions, obtained after rotating the base at 120°. Thus, the average roughness of each sample was obtained by the mean of the three readings.

#### Color analyses

Color analyses for each sample were performed initially (baseline) and after brushing with the toothpastes in a brushing machine, using a digital spectrophotometer (VITA Easyshade, VITA Zahnfabrik, Bad Säckingen, Germany), a white background, and an ambient light patterning box [21]. The spectrophotometer was calibrated prior to use according to the manufacturer's instructions. The results were quantified on the Vita Classical scale and the CIEL\*a\*b\* system coordinates. Vita Classical data were categorized into scores (shade guide units, SGU) based on the degree of luminosity [22], and the variation among these scores was then determined ( $\Delta$ SGU). The CIEL\*a\*b\* system was employed to determine the values for the L\*, a\*, and b\* coordinates. The  $\Delta E_{ab}$  and  $\Delta E_{00}$  values indicated the overall color difference, calculated as follows:

$$\Delta E_{ab} = \sqrt{\left(\Delta L^*\right)^2 + \left(\Delta a^*\right)^2 + \left(\Delta b^*\right)^2} \tag{I}$$

$$\Delta E_{00} = \sqrt{\left(\Delta L'/k_L S_L\right)^2 + \left(\Delta C'/k_C S_C\right)^2 + }$$
(II)

$$\left(\Delta H'/k_H S_H\right)^2 + RT\left(\Delta C'/k_C S_C\right) \left(\Delta H'/\left(k_H S_H\right)\right)$$

The  $\Delta E_{00}$  values were calculated sequentially [23], and then compared with the acceptability/perceptibility threshold values [24].

#### Statistical analysis

The roughness (Ra) data and the L\* and b\* values were analyzed using generalized linear models for repeated measures over time. These analyses considered the effects of resin composite, toothpaste, and time, along with their double and triple interactions. The general color change values ( $\Delta E_{ab}$  and  $\Delta E_{00}$ ) were analyzed

using generalized linear models, considering the effects of resin composite and toothpaste, as well as their interaction. The data for the a\* coordinate and Vita scale scores ( $\Delta$ SGU) were analyzed using the Mann-Whitney test for comparisons between the resin composites, the Kruskal-Wallis and the Dunn tests for comparisons among the toothpastes, and the paired Wilcoxon tests for comparisons over time. All the analyses were performed using the R program (R Core Team, Vienna, Austria), with a significance level set at 5%.

# RESULTS

The roughness results are presented in Table III. The roughness values increased significantly in all the groups after brushing (p = 0.0001); however, there was no significant difference among the toothpastes (p = 0.4491). At the initial time point (baseline), the roughness was significantly higher (p = 0.0001) in the nanofilled (Filtek One) than the nanohybrid resin composite (Aura). After brushing, the roughness values were significantly higher (p = 0.0001) in the nanohybrid (Aura) than the nanofilled (Filtek One) resin composite, regardless of the toothpaste.

The L\* coordinate results are presented in Table IV. At baseline, the nanofilled bulk-fill resin composite (Filtek One) presented significantly higher L\* values than the nanohybrid bulk-fill resin composite (p = 0.0027). Both resin composites indicated a significant increase in the L\* values (p < 0.0001) after brushing with the

RT and charcoal-containing toothpastes (LWC and WMC). When brushed with BWC charcoal toothpaste, the resin composites presented a significant decrease in L\* values (p < 0.0001). After brushing, the nanofilled bulk-fill resin composite (Filtek One) presented higher L\* values in the group brushed with LWC, and lower L\* values in the BWC than the RT and WMC groups (p < 0.0001). As for the nanohybrid bulk-fill resin composite (Aura), the L\* values after brushing were significantly higher for the RT and LWC groups than the BWC group (p < 0.0001).

The results of the a\* coordinate are presented in Table IV. The a\* values were significantly more negative (p < 0.05) in the nanohybrid than the nanofilled resin composite (p < 0.05). After brushing, there was a significant increase in the a\* values in all the groups (Filtek One, p = 0.03; Aura, p < 0.01). The nanofilled bulkfill resin composite presented more negative a\* values for BWC than WMC after brushing (p = 0.005). In contrast, the nanohybrid bulkfill presented more negative a\* values in the RT, LWC, and WMC groups than the BWC group after brushing (p < 0.0001). Considering the b\* results (Table IV), the nanofilled bulk-fill resin composite presented significantly higher b\* values at the initial time period than the nanohybrid resin composite (p < 0.0001), and significantly lower b\* values for WMC than the other toothpastes after brushing (p < 0.0001). As for the nanohybrid resin composite, the b\* values were significantly lower for WMC (p < 0.05) after brushing, compared with the other groups. There was a significant decrease

Table III - Mean (standard deviation) surface roughness value	es (Ra, $\mu\text{m})$ according to the toothpaste, resin composite, and time
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Desin composite	Taathaasta	Time		
Resin composite	Toothpaste	Baseline	After brushing	
	RT	0.11 (0.04) Ba	0.25 (0.14) Aa	
Nanafillad hull: fill	BWC <sup>#</sup>	0.10 (0.03) Ba	0.24 (0.05) Aa	
	LWC <sup>#</sup>	0.11 (0.04) Ba	0.18 (0.08) Aa	
	WMC <sup>#</sup>	0.10 (0.03) Ba	0.15 (0.06) Aa	
	RT	0.08 (0.04) Ba*	0.32 (0.18) Aa*	
Nanahuhrid hullt fill	BWC <sup>#</sup>	0.07 (0.03) Ba*	0.26 (0.12) Aa*	
	LWC <sup>#</sup>	0.08 (0.04) Ba*	0.23 (0.09) Aa*	
	WMC <sup>#</sup>	0.08 (0.04) Ba*	0.26 (0.15) Aa*	

<sup>#</sup>represents a charcoal-containing toothpaste. \*indicates a statistical difference between the nanohybrid bulk-fill resin composite (Aura) and nanofilled bulk-fill resin composite (Filtek One), under the same conditions of toothpaste and time (p < 0.05). Distinct letters (uppercase letters in rows and lowercase letters in columns, comparing the toothpastes in each resin composite) indicate statistically significant differences (p < 0.05). p-values: toothpaste = 0.6390; resin composite = 0.0955; time < 0.0001; toothpaste vs. resin composite = 0.4491; toothpaste vs. time = 0.3047; resin composite vs. time = 0.0001; toothpaste vs. resin composite vs. time = 0.9956. Table IV - Mean (standard deviation) or median (minimum; maximum) of L\*, a\*, and b\* values, according to the toothpaste, resin composite, and time

Variable	Desin composite	Teethneete	Time		
variable	Resin composite	Tootnpaste	Baseline	After brushing	
		RT	84.97 (1.02) Ba	85.67 (1.10) Ab	
	Nanofilled bulk-fill	BWC <sup>#</sup>	84.96 (0.84) Aa	83.00 (0.84) Bc	
		LWC <sup>#</sup>	85.22 (0.80) Ba	87.07 (0.77) Aa	
1*		WMC <sup>#</sup>	85.38 (1.00) Ba	86.18 (1.04) Ab	
L		RT	82.18 (1.07) Ba*	84.42 (0.94) Aa*	
		BWC <sup>#</sup>	82.45 (0.91) Aa*	78.70 (1.77) Bc*	
	Nanohybrid bulk-fill	LWC <sup>#</sup>	82.51 (0.71) Ba*	84.88 (0.81) Aa*	
		WMC <sup>#</sup>	82.31 (1.06) Ba*	83.48 (0.80) Ab*	
	Nanofilled bulk-fill	RT	-0.1 (-0.6; 0.0) Ba	0 (-0.4; 0.01) Aab	
		BWC <sup>#</sup>	-0.25 (-0.6; 0.1) Ba	-0.1 (-0.8; 0) Ab	
		LWC <sup>#</sup>	-0.2 (-0.3; 0.1) Ba	-0.1 (-0.3; 0.1) Aab	
. *		WMC <sup>#</sup>	-0.15 (-0.4; 0.2) Ba	0.1 (-0.1; 0.2) Aa	
a	Nanohybrid bulk-fill	RT	-0.9 (-1; -0.7) Ba*	-0.7 (-0.8; -0.5) Ab*	
		BWC <sup>#</sup>	-0.85 (-1.1; -0.4) Ba*	-0.35 (-0.6; -0.1) Aa*	
		LWC <sup>#</sup>	-0.9 (-1; -0.6) Ba*	-0.65 (-0.7; -0.5) Ab*	
		WMC <sup>#</sup>	-0.9 (-1.1; -0.8) Ba*	-0.7 (-0.9; -0.5) Ab*	
		RT	20.37 (0.80) Aa	18.26 (0.62) Ba	
	Nanofilled bulk-fill	BWC <sup>#</sup>	20.23 (2.04) Aa	18.53 (0.44) Ba	
		LWC <sup>#</sup>	20.67 (0.63) Aa	18.51 (0.49) Ba	
L *		WMC <sup>#</sup>	20.57 (0.34) Aa	17.02 (0.65) Bb	
D		RT	16.71 (0.38) Aa*	15.78 (0.56) Ba*	
	Nanohybrid bulk-fill	BWC <sup>#</sup>	16.59 (0.57) Aa*	15.39 (0.63) Bab*	
		LWC#	16.83 (0.65) Aa*	15.33 (0.33) Bb*	
		WMC <sup>#</sup>	17.01 (0.54) Aa*	14.55 (0.67) Bc*	

<sup>#</sup>represents a charcoal-containing toothpaste. \* indicates a statistical difference between the nanohybrid bulk-fill resin composite (Aura) and the nanofilled bulk-fill resin composite (Filtek One), under the same conditions of toothpaste and time (p < 0.05). Distinct letters (uppercase letters in rows and lowercase letters in columns, comparing toothpaste in each resin composite) indicate statistically significant differences (p < 0.05). Considering the variables and p values, L\* coordinate: toothpaste < 0.0001, resin composite < 0.0001, time = 0.0023, toothpaste vs. resin composite = 0.0517, toothpaste vs. time < 0.0001, resin composite vs. time = 0.5601, toothpaste vs. resin composite vs. time = 0.0027. The a\* coordinate: Filtek One (baseline = 0.2822, after brushing = 0.0051, comparing the time points = 0.03, and other comparisons < 0.05) and Aura (baseline = 0.5180, after brushing < 0.0001, and comparing the time points < 0.01). The b\* coordinate: toothpaste = 0.0047, resin composite < 0.0001, time < 0.0001, toothpaste vs. resin composite = 0.4628, toothpaste vs. time < 0.0001, resin composite vs. time = 0.3708, and toothpaste vs. resin composite vs. time = 0.1270.

in the b\* values in all the groups after brushing (p < 0.0001).

Overall color change data are presented in Figures 1 ( $\Delta E_{ab}$ ) and Figure 2 ( $\Delta E_{00}$ ). The  $\Delta E_{ab}$  values for the nanofilled bulk-fill resin composite were significantly higher for BWC and WMC (charcoal toothpastes) than RT (p < 0.0001). As for the nanohybrid resin composite,  $\Delta E_{ab}$  was significantly higher for BWC than for the other toothpastes (p < 0.0001). Comparatively, the  $\Delta E_{00}$  values for the nanofilled bulk-fill resin composite were significantly lower for RT than for the charcoal toothpastes (p < 0.05), and the  $\Delta E_{00}$  values for the nanohybrid resin composite

were significantly higher for BWC than for the other toothpastes (p < 0.0001).

The variation in the shade guide unit ( $\Delta$ SGU) results is presented in Table V. When the brushing was performed with RT, LWC and WMC, the scores decreased more for the nanofilled bulk-fill than the nanohybrid resin composite (p = 0.0002). Regarding the nanofilled resin composite, the scores decreased more for LWC and WMC toothpastes than the BWC toothpaste (p < 0.0001). As for the nanohybrid resin composite, the scores decreased more for RT, LWC, and WMC than for BWC (p < 0.0001).



**Figure 1** - Box-plot of the  $\Delta E_{ab}$  values according to the toothpaste and the resin composite. # represents a charcoal-containing toothpaste. Distinct uppercase letters indicate statistically significant differences between the resin composites (p < 0.05). Distinct lowercase letters indicate statistically significant differences among the toothpastes. p-values: p(toothpaste) < 0.0001; p(resin composite) = 0.6705; p(toothpaste vs. resin composite) = 0.1307.



Figure 2 - Box-plot of the  $\Delta E_{00}$  values according to the toothpaste and the resin composite. # represents a charcoal-containing toothpaste. Distinct uppercase letters indicate statistically significant differences between the resin composites (p < 0.05). Distinct lowercase letters indicate statistically significant differences among the toothpastes. p-values: p(toothpaste) < 0.0001; p(resin composite) = 0.0229; p(toothpaste vs. resin composite) = 0.0794.

Table V -	Median (maximum; min	imum value) color (	change caused by	/ the toothpaste on t	he resin composite, as qua:	antified by the Vita scale ( $ riangle S$	GU)
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Teethneete	Resin co	Resin composite		
Tootnpaste	Nanofilled bulk-fill	Nanohybrid bulk-fill	p-value	
RT	-2 (-3; -2) Aab	-1 (-1; -1) Bb	0.0002	
BWC <sup>#</sup>	0 (0; 3) Aa	0 (-1; 1) Aa	0.9699	
LWC <sup>#</sup>	-3 (-3; -2) Ab	-1 (-1; 0) Bb	0.0002	
WMC <sup>#</sup>	-3 (-3; -3) Ab	-1 (-1; -1) Bb	0.0002	
p-value	<0.0001	<0.0001		

<sup>#</sup> represents a charcoal-containing toothpaste. Distinct letters (uppercase letters in rows and lowercase letters in columns) indicate statistically significant differences (p < 0.05).

# DISCUSSION

Charcoal-based products are sold using different marketing strategies, and consumers and patients are exposed to abrasive toothpastes and powders without knowing the health risks [19]. Bearing this in mind, the present study investigated the potential effects of these toothpastes on bulk-fill resin composites, and restorative materials commonly used in dental practice. Considering the results found, the color stability was mediated by the toothpastes, but the surface roughness was especially affected by the composition of the bulk-fill resin composites. Therefore, the null hypothesis was rejected.

The clinically acceptable performance of certain resin-based materials requires analyzing some parameters that mediate clinical success over time, such as the surface characteristics and color stability of the material and restoration [18]. The roughness results are corroborated by Shimokawa et al. [25], who reported elevated roughness values for the Aura bulk-fill resin composite after exposure to RT. The resin composite composition impacts wear resistance and surface characteristics, and conventional nanohybrid resin composites are more susceptible to roughness and surface morphology changes than nanofilled resin composites [26,27]. In addition to its nanohybrid characteristics, Aura Bulk-Fill presents irregular matrix-dispersed clusters [28], monomeric alteration, and incorporation of prepolymerized particles in its composition, all of which could intensify its surface changes [29].

The roughness values were not mediated by the type of toothpaste, and no differences were found between the RT and the charcoal toothpastes [30]. However, the higher number of abrasive particles in the whitening toothpastes than the RTs could explain the color changes [14,31]. The results showed higher roughness and color changes ( $\Delta E_{00}$  - RT and BWC) in the nanohybrid bulk-fill resin composite (Aura), compared with the nanofilled composite (Filtek One), bearing in mind that the latter is composed of a nanosized filler and nanoclusters, which present better physical properties and surface smoothness for better wear resistance to abrasion [14,32,33].

The composition of the tested materials also impacted the initial values of the color coordinates and the surface roughness, thus corroborating previous studies [25,29,34]. Aura resin composite is marketed in a non-VITA shade (BFK, universal), while Filtek One is offered in a VITA shade (A1). As a result, initial differences between the composites can be expected due to variations in pigment incorporation and the translucency modifiers specific to each brand. Filtek One, for instance, appears to be more saturated with white pigments, as indicated by the L\* values. Moreover, initial differences in roughness can be associated with the specimen preparation, since the specimens were prepared using a polyester strip [35], whereas the materials exhibited surface characteristics based on their organic matrix (Table I).

According to the color results, the charcoal toothpastes promoted a general color change, specifically in the CIEL\*a\*b\* coordinates at different intensities, thus indicating the rejection of the null hypothesis. In the CIEL\*a\*b\* system, the L\* coordinate indicates luminosity (blackwhite axis; 0 - 100), the a\* coordinate represents saturation at the green (-) and red (+) axis, and the b\* coordinate, at the blue (-) and yellow (+) axis. The toothpaste that presented the highest color changes was BWC, with a decrease in L\* values (towards black) and higher values of color change ( $\Delta E_{ab}$  and  $\Delta E_{00}$ ) than RT.

However, all the evaluated toothpastes containing charcoal promoted color changes compared with RT, for at least one of the studied optical variables, regardless of the resin composite. Considering the general color change thresholds [24], all the toothpastes promoted clinically perceptible color changes ( $\Delta E_{ab} > 1.2$ ;  $\Delta E_{00} > 0.8$ ), but only the toothpastes containing charcoal surpassed the suggested acceptability limits for  $\Delta E_{ab}$  (> 2.7) and  $\Delta E_{00}$  (> 1.8), except the nanofilled bulk-fill resin composite (Filtek One) exposed to LWC ( $\Delta E_{00} = 1.7$ ).

The color results are in line with what was suggested by a previous study [36], which presented color change for a conventional resin composite exposed to toothpastes and powders containing charcoal. Although the importance of color in posterior restorations is seldom discussed, marginal and bulk discoloration represents up to 18% of the reasons for substitution of restorations [37]. Considering that no differences were found between the toothpastes regarding the surface roughness pattern, it could be hypothesized that the color changes of the resin composites could have been caused by the penetration of external pigments from the toothpastes by charcoal solubilization, together with some degradation or pigment incorporation on the polymeric chain.

The pigmentation seems to be mediated by the toothpaste composition, manufacture of the abrasive particle, and concentration of these compounds in the toothpaste [30, 31], whereas charcoal-containing toothpastes had an impact on the color of the evaluated resin composites in different intensities. The most significant changes in the color of the materials were caused by BWC, a toothpaste free of sodium lauryl sulfate (Table II), a common surfactant [38]. New studies are recommended to help better understand their mode of action, especially over the potential of these toothpastes to remove pigments inherent to the resin composite itself, represented by the alterations found in the b\* coordinate.

The findings of the present study add important considerations to previous discussions [1,3,9] on the general use of products containing charcoal and their effects on restorative materials. Although this was a controlled in vitro study, the results should be interpreted cautiously, and cannot be directly extrapolated to a clinical context, since some effects cannot be fully replicated due to the overly rigorous nature of the protocols used. Further studies employing methodologies, such as an optical profilometer or in vivo assessments, are essential to fully investigate the effects of these toothpastes on bulk-fill resin composites.

# CONCLUSION

The greatest changes in roughness were found for the nanohybrid bulk-fill resin composite (Aura, SDI), regardless of the toothpaste used. Charcoalcontaining toothpastes did not intensify the change in roughness differently from regular toothpastes. However, they did change the optical properties of the bulk-fill resin composites, in that the effects were more enhanced when the resin composites were brushed with Black is White toothpaste.

# Author's Contributions

WFVJ: contributed with the conceptualization, data curation, writing – original draft preparation, supervision and project administration. RQC: contributed with the methodology, writing – original draft preparation, validation. and investigation. LSB: contributed with the writing – review & editing, formal analysis, and visualization. MMM: contributed with the methodology, data curation and investigation. CPT: contributed with the conceptualization, formal analysis, and validation. FMGF: contributed with the conceptualization, data curation and validation. NIPP: contributed with the resources, writing – review & editing, validation and visualization. RTB: contributed with the conceptualization, supervision and investigation.

# **Conflict of Interest**

The authors have no conflicts of interest to declare.

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# **Regulatory Statement**

This study was exempted from review by the local ethics committee, since it did not involve the participation of any volunteers, or the use of any human material.

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