



Surface properties and susceptibility to staining of a resin composite after brushing with different whitening toothpastes

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ABSTRACT

Objectives: This study investigated the effects of different whitening toothpaste (WT) on the surface properties and staining susceptibility of a resin composite.

Methods: Cylindrical samples were prepared with a micro-hybrid resin composite and were randomized into groups according to the toothpaste ($n = 12$): distilled water (DW), regular toothpaste (RT), WT with silica + pyrophosphate (WT-S/P), WT with pentaphosphate and pyrophosphate (WT-P/P), WT with hydrogen peroxide and pyrophosphate (WT-HP/P) and WT with charcoal and pyrophosphate (WT-Ch/P). The samples were brushed for 825 cycles in an automatic brushing machine, simulating 30 days of brushing. After that, an immersion in coffee (10 mL/sample) was performed for 30 minutes for 30 days. The analyses of color, surface microhardness (SMH), and surface roughness (Ra) were performed at the initial time, after brushing with toothpaste and after immersion in coffee. The ΔL^* , Δa^* , Δb^* , ΔE_{ab} , Δ and E_{00} values were calculated comparing after toothpaste with initial time and after coffee with after toothpaste. Data were analyzed using a mixed linear model for repeated measures (SMH), Kruskal-Wallis, Dunn, Friedman, and Nemenyi tests, with $\alpha = 0.05$.

Results: For ΔL^* , the WT-Ch/P group had the lowest values and differed from the other groups comparing the after toothpaste with the initial time interval ($p < 0.001$). The WT-Ch/P group had the lowest SMH values in after-toothpaste time ($p < 0.001$). In after-toothpaste time and after coffee time, the WT-S/P group had the highest Ra values and differed from the groups except the WT-Ch/P group ($p < 0.001$).

Conclusions: The toothpaste composition affects the surface characteristics and susceptibility to staining of the resin composite. The charcoal-based toothpaste had the worst performance for the color analyses and SMH.

Keywords: Composite resins; Staining; Surface properties; Toothpastes

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INTRODUCTION

The desire for white teeth has led to a trend towards the increasing use of teeth whitening products [1–4]. Manufacturers of oral care products are constantly developing improvements and new approaches to tooth whitening [4]. Thus, at present, there are several types of products and technologies available, such as whitening toothpastes (WT), and these are used in an attempt to solve the problem of tooth discoloration [5,6].

WT is part of a category known as “over-the-counter” products (OTC). OTC products for teeth whitening are found on the shelves of pharmacies, supermarkets, or on the internet. These products promise tooth whitening and do not require a prescription or supervision of a dentist [7]. The majority of WT contains the same basic functional ingredients as conventional toothpaste [5,8]. The difference is the addition of a whitening agent, which can be of the abrasive, chemical, or optical type [5,8].

The abrasives added to WT act by means of mechanical removal of extrinsic pigmentations [5], and their effectiveness is related to the capacity for wear promoted by the abrasives [7]. Charcoal is considered an abrasive and may be found in WT. It has shown the ability to remove stains and deposits from teeth [9]. The most common chemical agents found in toothpastes include surfactants, enzymes, citrate, pyrophosphates, and hexametaphosphate. These components help to degrade stained biofilms, aiding their mechanical removal [5]. Furthermore, some WT contain hydrogen peroxide as the chemical agent; however, the presence of peroxides in toothpastes is less common and challenging, mainly due to aspects related to formulation [5]. Optical modifiers, such as blue covarine, mica, and titanium dioxide, act by modifying the interaction of light incident on the teeth, providing the sensation of whiter and brighter teeth [5,10,11].

WT is known to be capable of changing the surface properties of resin composite [12], such as reducing the surface microhardness (SMH) and increasing the surface roughness [13–15]. A resin composite with a rough surface is more susceptible to staining [16,17]. Moreover, the reduction in microhardness changes the properties of the resin composite making it more susceptible

to degradation of its surface and resin matrix, leading to more extensive sorption and solubility, and resulting in surface staining [18,19]. Long-term color alterations in resin composite restorations occur due to superficial and marginal staining, microleakage, surface alterations, and internal deterioration of the material [20,21]. Thus, resin composite pigmentation may compromise the visual acceptability of these restorations and result in the need for their replacement [20,21].

Resin composites are highly esthetic materials, as they aim to imitate the natural characteristics of the tooth, such as color, translucency, and texture [22–24]. Thus, color stability has become one of the main requirements of restorative materials [19]. The objective of this study was not to claim that a specific type of toothpaste can prevent coffee stains, but rather to evaluate the surface properties and staining of a resin composite that had its surface altered by the use of different WT. The use of WT has increased every day and their effects on resin composite are well established in the literature. However, there is no investigation of how this altered surface behaves when exposed to coffee. Furthermore, although there are manuscripts that evaluate the impacts of brushing with toothpastes on resin materials, there is a lack of studies that evaluate the toothpaste of the same manufacturer with different active ingredients. The toothpastes evaluated in this study are available to patients as part of a single product line from a specific manufacturer. It is important to understand how these different types of products offered directly to patients may affect the resin composites.

Thus, the aim of this *in vitro* study was to investigate the effects of WT containing different whitening agents on susceptibility to staining and surface characteristics of a resin composite by means of color analysis, SMH, and roughness. The null hypotheses tested in this study were: (1) the use of WT with different whitening agents would not interfere with SMH, (2) surface roughness, and (3) color variables/susceptibility to staining of a micro-hybrid resin composite.

METHODS

Experimental design

This *in vitro* study had as experimental units resin com-

posite specimens that were subjected to treatment with different toothpaste. Toothpaste varied their whitening agent (six levels): distilled water (DW), regular toothpaste (RT) (Colgate Total 12 Clean Mint; Colgate-Palmolive Company, São Bernardo do Campo, Brazil), WT with silica + pyrophosphate (WT-S/P) (Colgate Luminous White Brilliant; Colgate-Palmolive Company), WT with pentaphosphate and pyrophosphate (WT-P/P) (Colgate Luminous White Instant; Colgate-Palmolive Company), WT with hydrogen peroxide and pyrophosphate (WT-HP/P) (Colgate Luminous White Expert; Colgate-Palmolive Company), and WT with charcoal and pyrophosphate (WT-Ch/P) (Colgate Luminous White Carvão Ativado, Colgate-Palmolive Company). The detailed composition of the toothpaste is available in Table 1. The components of toothpastes and their derivatives are categorized in Table 1 according to the studies of Joiner [5] and Freitas *et al.* [25]. Time was the other factor under study for the analysis of surface roughness and SMH (initial, after brushing with toothpaste, and after immersion in coffee). The analyses performed were SMH, surface roughness (Ra), and color (ΔL^* , Δa^* , Δb^* , ΔE_{ab} , and ΔE_{00}).

Sample preparation

Cylindrical samples ($n = 120$) of Filtek Z250 micro-hybrid resin composite [12] (3M ESPE, St. Paul, MN, USA), color B1, were made. Of these 120 samples, 60 had 7.00 mm in diameter and 2.00 mm in thickness and were used to carry out the color and roughness analyses, and another 60 samples of 3.00 mm in diameter and 2.00 mm in height were used exclusively for SMH analyses. Twenty extra samples were prepared (10 with 7.00-mm diameter and 10 with 3.00-mm diameter), one of each diameter for each group as a precaution in case of accidental loss of a sample. At the end of the experiment, no samples were lost in any of the stages performed, so the data from the extra samples were disregarded. Samples were made by inserting an increment of resin composite into the polytetrafluoroethylene matrix. After inserting the resin composite, the increment was covered with a polyester strip and a glass slide under a weight of 500 g for 30 seconds. The samples were photoactivated for 20 seconds, as recommended by the manufacturer, with a diode emitting light (Valo Ultradent Products Inc.,

South Jordan, UT, USA) at standard power (1,000 mW/cm²). After 24 hours the polishing process was carried out using a polishing machine (model APL 4; Arotec, Cotia, Brazil) for 1 minute with silicon carbide sanding discs (600, 1,200, and P4000 - Buehler Ltd., Lake Bluff, IL, USA). Felt disks (TCT, TWI; Arotec) associated with diamond pastes (3 1/2 μ m and 1/4 μ m) were used to finalize the polishing process. The surfaces of the samples were protected with colorless acid-resistant varnish so that only the surface that was polished was exposed for the treatments. In this way, the treatments were limited to only one surface of the resin composite, getting closer to the condition found in the oral environment. The samples were randomized into groups of 12 ($n = 12$). Sample size calculation, based on a previous study [12], indicated that six samples per group would yield an 80% power ($\beta = 0.8$), at $\alpha = 0.01$. To avoid possible losses and to follow previous studies in the field, the sample was doubled ($n = 12$ per group). After that, each sample group was randomized to the experimental groups. All randomizations performed in this study were performed by a person without any participation in the experiment.

Brushing with toothpaste

The samples were submitted to simulated brushing using toothbrush heads (Oral-B Indicator 40 Soft, Gillette do Brasil Ltda., Manaus, Brazil) coupled with an automatic brushing machine (Equilabor, Piracicaba, Brazil). The brush head was attached parallel to the sample surface. The samples were brushed with toothpaste slurry (proportion of 1 g:3 mL) according to the group, using a static axial load of 200 g and a speed of 5 movements/sec, at 37°C [26]. One month of brushing was simulated with 825 cycles [27]. After brushing, the samples were washed with DW for 10 seconds to remove toothpaste residues on the surface and stored in relative humidity.

Immersion in coffee

Coffee was prepared in the proportion of 50 mL of hot water and 7 g of soluble coffee (Nescafé Original; Nestlé, Araras, Brazil). The samples were immersed in 10 mL of coffee per sample for 30 minutes daily, simulating the contact of coffee with the tooth structure for one day of consumption [28,29]. After immersion, the samples

Table 1. Composition of toothpaste used in this study according to the manufacturers' information

Product	Manufacturer	Composition	Category
Colgate Total 12 Clean Mint	Colgate - Palmolive Industrial LTDA, São Bernardo do Campo, SP, Brazil	Active ingredients: sodium fluoride 0.32%, zinc citrate, zinc oxide Ingredients: glycerin, water, hydrated silica, sodium lauryl sulfate, arginine, aroma, cellulose gum, zinc oxide, poloxamer 407, tetrasodium pyrophosphate, zinc citrate, benzyl alcohol, xanthan gum, cocamidopropyl betaine, sodium fluoride, sodium saccharin, phosphoric acid, sucralose, CI 77891 Contains sodium fluoride (1,450 ppm fluoride) Whitening agents: - Abrasives: hydrated silica - Chemical: tetrasodium pyrophosphate	Regular toothpaste (RT)
Colgate Luminous White Brilliant	Colgate - Palmolive Industrial LTDA, São Bernardo do Campo, SP, Brazil	Active ingredient: 0.32% sodium fluoride Ingredients: water, sorbitol, hydrated silica, PEG-12, sodium lauryl sulfate, aroma, cellulose gum, potassium hydroxide, tetrasodium pyrophosphate, phosphoric acid, cocamidopropyl betaine, sodium fluoride, benzyl alcohol, sodium saccharin, CI 77891, limonene Contains sodium fluoride (1,450-ppm fluoride) Whitening agents: - Abrasives: hydrated silica - Chemical: tetrasodium pyrophosphate	Whitening toothpaste (WT-SP)
Colgate Luminous White Instant	Colgate - Palmolive Industrial LTDA, São Bernardo do Campo, SP, Brazil	Active ingredient: 0.243% sodium fluoride Ingredients: water, sorbitol, hydrated silica, sorbitol, glycerin, PEG-12, pentasodium triphosphate, tetrapotassium pyrophosphate, sodium lauryl sulfate, aroma, cellulose gum, cocamidopropyl betaine, sodium saccharin, xanthan gum, sodium fluoride, sodium hydroxide, hydroxypropyl methylcellulose, propylene glycol, polysorbate 80, mica, CI 74160, CI 77891, CI 73360, CI 17200, CI 42051, eugenol Contains sodium fluoride (1,100 ppm fluoride). Whitening agents: - Abrasives: hydrated silica - Chemical: pentasodium triphosphate, tetrapotassium pyrophosphate - Others: mica	Whitening toothpaste (WT-PP)
Colgate Luminous White Expert	Colgate - Palmolive Industrial LTDA, São Bernardo do Campo, SP, Brazil	Active ingredients: hydrogen peroxide 2%, sodium monofluorophosphate 0.76% Ingredients: propylene glycol, calcium pyrophosphate, PVP-hydrogen peroxide, PEG/ PPG-116/ copolymer 66, PEG-12, glycerin, aroma, sodium lauryl sulfate, silica, PVP, tetrasodium pyrophosphate, sodium saccharin, sodium monofluorophosphate, disodium pyrophosphate, sucralose, BHT, eugenol Contains sodium monofluorophosphate (1,000-ppm fluoride). Whitening agents: - Abrasives: silica, calcium pyrophosphate - Bleaching: hydrogen peroxide - Chemical: tetrasodium pyrophosphate, disodium pyrophosphate	Whitening toothpaste (WT-HP/P)
Colgate Luminous White Carvão ativado	Colgate - Palmolive Industrial LTDA, São Bernardo do Campo, SP, Brazil	1,000-ppm sodium monofluorophosphate, water, sorbitol, hydrated silica, PEG-12, sodium lauryl sulfate, aroma, cellulose gum, potassium hydroxide, tetrasodium pyrophosphate, phosphoric acid, cocamidopropyl betaine, sodium fluoride, sodium saccharin, benzyl alcohol, charcoal powder, limonene Contains sodium fluoride (1,450-ppm fluoride). Whitening agents: - Abrasives: hydrated silica, calcium pyrophosphate - Chemical: pentasodium triphosphate, tetrapotassium pyrophosphate - Others: CI 77266 (powdered charcoal)	Whitening toothpaste (WT-Ch/P)

These toothpastes were chosen based on the different whitening agents of the same commercial brand. The classification of whitening agents was made following the work of Joiner [5] and Feitas *et al.* [25]. An important observation is that mica and titanium dioxide can be considered whitening agents depending on the quantity. Thus, mica would be an abrasive agent and titanium dioxide would be an abrasive or optical agent. Despite this, mica and titanium dioxide are often just dyes and the quantity of these components in the toothpastes used in this study is unknown, thus making it difficult to classify these components (Vilhena *et al.* [11]).

BHT, butylated hydroxy toluene; PEG, polyethylene glycol; PPG, polypropylene glycol; PVP, polyvinylpyrrolidone.

were washed in DW for 10 seconds. This protocol was repeated for 30 days.

Surface microhardness

SMH analysis was performed at the initial time, after toothpaste, and after coffee. A Knoop indenter was used with a load of 25 g and time of 5 seconds in a micro-durometer (HMV-2000; Shimadzu, Tokyo, Japan). Five indentations were made in each sample, with a distance of 100 μm , and the average was calculated to determine the Knoop hardness number.

Surface roughness (Ra)

Ra analyses was performed at the initial time, after toothpaste, and after coffee. Roughness was measured using a profilometer (Surf-Corder 1700; Kosaka, Tokyo, Japan). Three different equidistant directions were measured on the surface of each sample, with a cutoff point of 0.25, a reading length of 1.25 mm, and a velocity of 0.1 mm/sec. The average of the values obtained was calculated and one Ra value per sample was considered.

Color analyses (ΔL^* , Δa^* , Δb^* , ΔE_{ab}^* , and ΔE_{00})

Color analyses was performed at the initial time, after toothpaste, and after coffee. The samples were placed on a white background (sample holder) inside a light booth (GTI mini matcher MM1e; GTI Graphic Technology, Newburgh, NY, USA) to standardize the ambient light during the measurement process. The samples were subjected to color reading using a spectrophotometer (Konica Minolta CM-700d spectrophotometer; Konica Minolta Investment, Shanghai, China) calibrated according to the manufacturer's instructions. The values obtained were quantified in L^* , a^* , and b^* according to the CIE $L^*a^*b^*$ system. The calculation of ΔL^* , Δa^* , and Δb^* was performed. The overall color change was measured by calculating the ΔE_{ab} using the following formula: $\Delta E_{ab} = ([L_1 - L_0]^2 + [a_1 - a_0]^2 + [b_1 - b_0]^2)^{1/2}$. ΔE_{00} was calculated using the following formula:

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L} \right)^2 + \left(\frac{\Delta C'}{K_C S_C} \right)^2 + \left(\frac{\Delta H'}{K_H S_H} \right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C} \right) \left(\frac{\Delta H'}{K_H S_H} \right) \right]^{1/2}$$

All color analysis parameters were calculated by comparing after toothpaste with the initial time (impact of

toothpaste) and after coffee after toothpaste (impact of staining on the brushed sample).

Statistical analyses

Descriptive and exploratory data analyses were performed. A mixed linear model for repeated measurements over time was then applied for SMH. The Ra and color data did not meet the assumptions of parametric analysis and so were analyzed by nonparametric tests. For comparisons between groups, the nonparametric tests of Kruskal-Wallis and Dunn were used, and for the comparisons of Ra between times, the nonparametric tests of Friedman and Nemenyi were used. Analyses were performed using the R program [30] with a significance level of 5%.

RESULTS

Surface microhardness

The results of the SMH analysis are shown in Table 2. When the time intervals for the same group were compared, the WT-S/P, WT-P/P, and WT-HP/P groups showed lower values after toothpaste use, compared with the initial time. For the other groups, no statistically significant differences were found when the time after toothpaste use was compared with the initial time. When the time after coffee use was compared with the time after toothpaste use, it was possible to observe that all groups showed statistically significant differences, with lower values in the time after coffee use compared with the time after toothpaste use.

When the groups were compared at the same time of analysis, no statistically significant differences were found between the groups at the initial time and after the coffee time. After the toothpaste time, the WT-Ch/P group had the lowest values and differed statistically from all other groups.

Surface roughness (Ra)

The results of the surface roughness tests are available in Table 3. When the times for the same group were compared, all groups showed statistically significant differences between these times. Lower values were observed in the time after toothpaste use compared with the initial time, irrespective of the toothpaste and lower

Table 2. Surface microhardness as a function of group and time

Group	Time		
	Initial	After toothpaste	After coffee
DW	90.37 ± 4.41 ^{Aa}	86.99 ± 5.21 ^{Aa}	75.41 ± 3.49 ^{Ba}
RT	91.13 ± 3.91 ^{Aa}	86.96 ± 5.04 ^{Aa}	72.95 ± 3.59 ^{Ba}
WT-S/P	91.34 ± 4.17 ^{Aa}	81.87 ± 3.26 ^{Ba}	75.27 ± 5.01 ^{Ca}
WT-P/P	90.53 ± 3.86 ^{Aa}	84.81 ± 4.80 ^{Aa}	76.58 ± 2.26 ^{Ba}
WT-HP/P	92.40 ± 4.30 ^{Aa}	81.83 ± 7.09 ^{Ba}	74.97 ± 2.81 ^{Ca}
WT-Ch/P	90.40 ± 2.94 ^{Aa}	70.92 ± 5.40 ^{Bb}	75.06 ± 2.12 ^{Ba}

Values are presented as mean ± standard deviation.

DW, distilled water; RT, regular toothpaste; WT-Ch/P, whitening toothpaste with charcoal and pyrophosphate; WT-HP/P, whitening toothpaste with hydrogen peroxide and pyrophosphate; WT-P/P, whitening toothpaste with pentaphosphate and pyrophosphate; WT-S/P, whitening toothpaste with silica and pyrophosphate.

Distinct letters (uppercase horizontally and lowercase vertically) indicate statistically significant differences ($p \leq 0.05$).

p (group) < 0.0001; p (time) < 0.0001; p (interaction) < 0.0001.

Table 3. Surface roughness (Ra) as a function of group and time

Group	Time			p -value
	Initial	After toothpaste	After coffee	
DW	0.23 ± 0.01 ^{Ca}	0.26 ± 0.00 ^{Bd}	0.30 ± 0.01 ^{Ad}	<0.0001
RT	0.23 ± 0.01 ^{Ca}	0.33 ± 0.01 ^{Bdc}	0.39 ± 0.01 ^{Abc}	<0.0001
WT-S/P	0.23 ± 0.01 ^{Ca}	0.50 ± 0.00 ^{Ba}	0.53 ± 0.01 ^{Aa}	<0.0001
WT-P/P	0.23 ± 0.01 ^{Ca}	0.29 ± 0.01 ^{Bcd}	0.34 ± 0.01 ^{Accl}	<0.0001
WT-HP/P	0.23 ± 0.01 ^{Ca}	0.35 ± 0.01 ^{Bbc}	0.40 ± 0.01 ^{Abc}	<0.0001
WT-Ch/P	0.23 ± 0.01 ^{Ca}	0.48 ± 0.01 ^{Bab}	0.53 ± 0.01 ^{Aab}	<0.0001

Values are presented as mean ± standard deviation.

DW, distilled water; RT, regular toothpaste; WT-Ch/P, whitening toothpaste with charcoal and pyrophosphate; WT-HP/P, whitening toothpaste with hydrogen peroxide and pyrophosphate; WT-P/P, whitening toothpaste with pentaphosphate and pyrophosphate; WT-S/P, whitening toothpaste with silica and pyrophosphate.

Distinct letters (uppercase horizontally and lowercase vertically) indicate statistically significant differences ($p \leq 0.05$).

values in the time after coffee compared with the after toothpaste time for all groups.

When the groups were compared at the same time, no statistically significant differences in the initial time were found. At the time after toothpaste use and after coffee time, the same results were observed between groups. The DW group had the lowest values and differed from all other groups brushed with toothpastes. The WT-S/P group had the highest roughness values and did not differ statistically from the WT-Ch/P group. The WT-HP/P group and the RT group showed intermediate roughness values and did not differ from each other and did not differ from the WT-Ch/P. The WT-P/P group had the lowest values among the toothpastes evaluated and did not differ from the RT group and from the WT-HP/P.

Color analyses (ΔL^* , Δa^* , Δb^* , ΔE^*_{ab} , and ΔE_{00})

The Δa and Δb analyses did not provide relevant results.

The ΔL^* results are shown in Table 4. The WT-Ch/P group had the lowest values and differed from all other groups in the time interval *after toothpaste–initial time*. In the time interval *after coffee–after toothpaste*, all groups that were brushed with toothpaste did not differ from the DW group, irrespective of the active principle.

For ΔE^*_{ab} (Table 5) in the time interval *after toothpaste–initial*, the WT-S/P group had the lowest values and differed from the DW group and the RT group but did not differ from the other groups brushed with WT. In the time interval *after coffee–after toothpaste*, the WT-Ch/P group had the highest values and differed from all other evaluated groups.

For ΔE_{00} (Table 6), the WT-S/P group presented the lowest values and differed from the DW group but did

Table 4. of variation in color (ΔL^*) according to time interval

Group	Time interval	
	After toothpaste–initial time	After coffee–after toothpaste
DW	0.24 (0.03 to 0.42) ^{ab}	–1.69 (–2.05 to –1.25) ^{ab}
RT	0.42 (–0.60 to 0.85) ^a	–1.48 (–2.07 to –0.19) ^a
WT-S/P	0.17 (–0.15 to 0.30) ^{abc}	–1.52 (–1.79 to –1.29) ^a
WT-P/P	0.10 (–0.41 to 0.35) ^{bc}	–1.51 (–3.77 to 0.62) ^a
WT-HP/P	0.01 (–0.63 to 0.26) ^{bc}	–1.71 (–2.33 to –1.26) ^{ab}
WT-Ch/P	0.02 (–0.38 to 0.15) ^c	–2.12 (–2.88 to –1.90) ^b
<i>p</i> -value	<0.0001	

Values are presented as median (range).

DW, distilled water; RT, regular toothpaste; WT-Ch/P, whitening toothpaste with charcoal and pyrophosphate; WT-HP/P, whitening toothpaste with hydrogen peroxide and pyrophosphate; WT-P/P, whitening toothpaste with pentaphosphate and pyrophosphate; WT-S/P, whitening toothpaste with silica and pyrophosphate.

Different vertical letters indicate statistically significant differences ($p \leq 0.05$).

Table 5. Variation in color (ΔE_{ab}^*) according to time interval

Group	Time interval	
	After toothpaste–initial time	After coffee–after toothpaste
DW	0.48 (0.38–0.76) ^a	2.03 (1.64–2.37) ^b
RT	0.67 (0.31–1.91) ^a	1.91 (1.36–2.46) ^b
WT-S/P	0.28 (0.17–0.60) ^b	1.95 (1.69–2.19) ^b
WT-P/P	0.41 (0.21–1.16) ^{ab}	1.83 (1.26–4.08) ^b
WT-HP/P	0.36 (0.22–1.14) ^{ab}	2.12 (1.59–3.07) ^b
WT-Ch/P	0.45 (0.29–0.60) ^{ab}	2.52 (2.27–3.28) ^a
<i>p</i> -value	0.0003	0.0003

Values are presented as median (range).

DW, distilled water; RT, regular toothpaste; WT-Ch/P, whitening toothpaste with charcoal and pyrophosphate; WT-HP/P, whitening toothpaste with hydrogen peroxide and pyrophosphate; WT-P/P, whitening toothpaste with pentaphosphate and pyrophosphate; WT-S/P, whitening toothpaste with silica and pyrophosphate.

Different vertical letters indicate statistically significant differences ($p \leq 0.05$).

not differ from the other groups brushed with WT in the time interval *after toothpaste–initial*. In addition, the WT-P/P group and the WT-HP/P group differed from the DW group but did not differ from the RT group or from the other groups of WT. In the time interval *after coffee–after toothpaste*, the WT-Ch/P group had the highest values and statistically differed from all other evaluated groups.

DISCUSSION

This study evaluated the action of WT containing dif-

Table 6. Variation in color (ΔE_{00}) according to time interval

Group	Time interval	
	After toothpaste–initial time	After coffee–after toothpaste
DW	0.57 (0.46–0.83) ^a	1.84 (1.48–2.04) ^b
RT	0.51 (0.27–1.34) ^{ab}	1.80 (1.53–2.26) ^b
WT-S/P	0.25 (0.17–0.42) ^c	1.84 (1.61–2.03) ^b
WT-P/P	0.30 (0.15–0.94) ^{bc}	1.79 (1.49–3.02) ^b
WT-HP/P	0.37 (0.23–1.11) ^{bc}	1.90 (1.44–2.51) ^b
WT-Ch/P	0.37 (0.32–0.65) ^{abc}	2.23 (2.07–2.80) ^a
<i>p</i> -value	<0.0001	

Values are presented as median (range).

DW, distilled water; RT, regular toothpaste; WT-Ch/P, whitening toothpaste with charcoal and pyrophosphate; WT-HP/P, whitening toothpaste with hydrogen peroxide and pyrophosphate; WT-P/P, whitening toothpaste with pentaphosphate and pyrophosphate; WT-S/P, whitening toothpaste with silica and pyrophosphate.

Different vertical letters indicate statistically significant differences ($p \leq 0.05$).

ferent active agents on surface characteristics and susceptibility to staining of a micro-hybrid resin composite. The first and second null hypotheses tested in this study were rejected because the WT interfered with both the SMH and surface roughness of the micro-hybrid resin composite, promoting alterations in different intensities, which varied according to the active agent of the toothpaste used. The third null hypothesis tested in this study was also rejected because, depending on the toothpaste used and the time interval, the WT was able to interfere with the parameters evaluated in the color analysis. The use of WT was capable of promoting changes in surface characteristics and favored the staining of a micro-hybrid resin composite.

In the SMH analysis, the WT-Ch/P group not only showed statistically significant differences when the initial time and after toothpaste time were compared, but was also the only group in after toothpaste time that differed from the DW group and from all other groups evaluated, and showed the lowest values. This result may be associated with the action of charcoal. Charcoal is obtained as a residue when carbonaceous material is partially burned, or heated and is characterized by its high capacity for adsorption due to its high porosity [31]. Charcoal is used as an adsorbent in several areas such as medicine to aid in detoxification. In the industrial environment, it is used for the adsorption of gases and liquids and has been increasingly used, particularly in

water treatment [31,32]. Charcoal is still present in some toothpaste and acts as an abrasive component, as it has a very high capacity for adsorbing pigments, chromophores, and extrinsic stains by means of dental abrasion [33]. Studies have shown that the action of charcoal on the resin composite surface is associated with the characteristics of its particles [34]. The shape, structure, and size of charcoal particles define its abrasiveness, ie, the larger the particle size, the more abrasive this component will be [34]. The study showed that toothpastes containing charcoal have irregular abrasive particles, which enhances their abrasive effect [34]. In this study, in addition to changing the SMH, the toothpaste containing charcoal also resulted in a statistically significant increase in surface roughness at the after toothpaste time, in agreement with several studies in the literature [15,35]. The literature reports that insufficient clinical and laboratory data to substantiate the safety and efficacy claims of charcoal and charcoal-based toothpastes [36].

In addition to the action of charcoal, the action of the brush bristles together with the brushing force is associated with increased roughness and decreased microhardness of the composite [37–39]. Since the resin composite is more conducive to mechanical fatigue, charcoal enhances the wear and weakening of the material, leading to cracks and ruptures [40]. This fact would also explain the increase in surface roughness after toothpaste time. This was observed in all groups evaluated irrespective of the toothpaste used, including the DW group. The abrasion caused by the toothbrush bristles is capable of wearing down the organic portion of the resin composite, forming spaces and consequently increasing the surface roughness [41]. This result is in agreement with other studies that also demonstrated an increase in surface roughness of the resin composite due to the action of the brush bristles [42,43]. When the roughness values between the groups at the after toothpaste time were compared, it was possible to observe that despite having increased roughness, the DW group was the one that showed the lowest values when compared with the groups that used toothpaste. Moreover, in these groups the increase in roughness in comparison with the initial values and those after brushing varied according to the active agent of the toothpaste used.

The brushing with WT with silica + pyrophosphate

and WT with charcoal and pyrophosphate resulted in the highest increase in roughness values among the toothpaste evaluated. Studies have shown that hydrated silica was a highly effective abrasive and that it showed a higher increase in roughness values when associated with other abrasives [44–46], which was titanium dioxide, in the case of this group.

The WT-P/P group, despite showing an increase in surface roughness when the initial time and the after toothpaste time were compared, was the only type that did not differ from the DW group in the after toothpaste time. The mica and titanium dioxide can be considered whitening agents depending on the quantity [11]. Thus, mica would be an abrasive agent and titanium dioxide would be an abrasive or optical agent. Despite this, mica and titanium dioxide are often just dyes and the quantity of these components in the toothpastes used in this study is unknown, thus making it difficult to classify these components [11]. Based on the results found, it is assumed that the quantity of these components present in the toothpaste may have been sufficient to increase roughness but not for this increase to be of the same magnitude as that found in the other WT evaluated, thus justifying the results found.

The WT-HP/P group contains calcium pyrophosphate, silica, tetrasodium pyrophosphate, disodium pyrophosphate, and 2% hydrogen peroxide in their composition. According to a study, 2% hydrogen peroxide can cause an increase in the roughness of the resin composite, as it has the ability to release free radicals due to its high oxidation and reduction capacity [47]. This phenomenon acts by degrading the resin matrix and breaking the polymer chains of the resin composite [47], resulting in changes in their properties such as increased roughness and decreased microhardness [47–50]. Nevertheless, for the SMH analysis in this group, no statistically significant differences were found with the DW group. Furthermore, for the surface roughness analysis, this group did not show the highest values despite having differed from the DW and conventional toothpaste groups. This result may be associated with the fact that the toothpaste remained in contact with the resin composite surface for a short time, which reduced the adverse effects of the bleaching agent. Studies reported that the main factor related to the degradation of the resin composite

surface is the time of exposure to the bleaching agent and not the concentration of this agent [51–53].

The RT group showed the least change in its properties. This is a common toothpaste without any addition of abrasive agents other than the abrasive common to non-WT [54]. In 2003, a study observed that non-WT showed a smaller increase in roughness compared with WT [52]. This can be explained by the size and shape of their abrasives, since the smaller the abrasive particles and the more regular their shape, the less wear of the resin composite structure [55].

After immersion in coffee, all groups were observed to have an increase in roughness and a decrease in SMH, irrespective of the values shown after brushing. This result may be associated with the pH of the coffee. Coffee has an acidic pH (pH 5.0) [38]. Acidity can have an effect on the resin matrix, thus promoting the displacement of filler particles, and generating alterations on the resin composite surface [38]. Furthermore, coffee is composed of water and coffee powder and the effect of water absorption can lead to degradation of polymeric materials [56]. The particles detach from the outer surface of the material, resulting in an increase in surface roughness [38,57]. These results were in agreement with other studies [56,58], which showed that coffee was able to alter the microhardness and surface roughness of the resin composite.

Relative to the resin composite susceptibility to staining, after the color analysis, statistically significant differences were found for the WT-Ch/P group in some of the parameters evaluated. When the initial time interval and that after toothpaste use were compared for ΔL^* , it was the only group that differed from the control group, showing a decrease in values, that is, its color was closer to black. This result may have been associated with the dark color of charcoal. Recent studies have concluded that charcoal-based toothpaste resulted in a more significant color change than that produced by conventional toothpaste. The organic matrix of resin composite restorative materials absorbs water, consequently, they are more prone to the penetration of coloring agents [59]. This result was in agreement with the studies [60] that proved that all charcoal-based toothpastes caused stains on resin composite restorations. Furthermore, in the composition of charcoal, there is pure, highly porous

carbon, plus abrasives, detergents, therapeutic agents, and charcoal microparticles [34]. These components promote an increase in the porosity of the resin which becomes rough so that it can easily retain the charcoal pigments [9].

When the time interval after toothpaste use and time interval after coffee use were compared in the color analyses, the WT-Ch/P group showed higher ΔE_{00} and ΔE_{ab}^* values than the other groups; nevertheless, the values were statistically similar or lower than those for the control group and lower than that 4.2 units [61] or 3.3 units [62] that are the suggested default values for clinical acceptability of color differences. All other groups did not differ from the control group and also presented values lower than 4.2 and 3.3 units. Comparing the initial time and after toothpaste time interval, only the WT-S/P group differed from the DW group; however, it presented the lowest values than the other groups and lower than the clinical acceptability values.

This study was limited by the use of only one type of resin composite in the preparation of the samples. Furthermore, although the relationship between relative dentin abrasivity and the abrasive potential of toothpastes on restorative materials is controversial [63–65], it would be interesting to evaluate the abrasiveness of the tested toothpastes and see if it correlates with the results found in this study. In order to conduct new studies, it would be interesting to use different brands and types of resin composite, associating the carrying out of the staining protocol at other times, in addition to the use of other methodologies such as performing three-dimensional profilometry.

CONCLUSIONS

This study made it possible to conclude that the use of WT interferes with the SMH, surface roughness, and susceptibility to staining of the resin composite according to their composition. Furthermore, charcoal toothpaste was the WT that most reduced microhardness, influenced the increase in surface roughness, and interfered with the susceptibility to staining of a micro-hybrid resin composite. Clinically, the use of toothpaste impacts the properties of the resin composite and can even enhance or increase staining. These effects are

greater in activated charcoal.

CONFLICT OF INTEREST

No potential conflict of interest relevant to this article was reported.

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AUTHOR CONTRIBUTIONS

Conceptualization, Data curation, Project administration: Barros AS, Barbosa CM, Ferraz LN. Formal analysis, Investigation, Methodology, Software, Validation, Visualization: all authors. Funding acquisition: Barros AS. Resources, Supervision: Barros AS, Barbosa CM. Writing - original draft: Barros AS, Barbosa CM, Ferraz LN. Writing - review & editing: all authors. All authors read and approved the final manuscript.

DATA SHARING STATEMENT

The datasets are not publicly available but are available from the corresponding author upon reasonable request.

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