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EFEITOS DO CARVÃO ATIVADO EM PÓ COMBINADO COM DENTIFRÍCIOS NA MODIFICAÇÃO DE COR E SUPERFÍCIE DE ESMALTE DENTAL

EFFECTS OF ACTIVATED CHARCOAL POWDER COMBINED WITH TOOTHPASTES ON ENAMEL COLOR CHANGE AND SURFACE PROPERTIES

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Trabalho de Conclusão de Curso apresentado à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Cirurgião Dentista.

Udergraduate final work presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Dental Surgeon.

Orientador: Prof. Dr. Vanessa Cavalli Gobbo

Coorientador: Msc. Matheus Kury Rodrigues

ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DO TRABALHO DE CONCLUSÃO DE CURSO DEFENDIDA PELO ALUNO SAMUEL DA SILVA PALANDI E ORIENTADO PELA PROF[A.] DR[A].VANESSA CAVALLI GOBBO.

PIRACICABA

2021

Ficha catalográfica
Universidade Estadual de Campinas
Biblioteca da Faculdade de Odontologia de Piracicaba
Marilene Girello - CRB 8/6159

P172e Palandi, Samuel da Silva, 1999-
Efeitos do carvão ativado em pó combinado com dentifrícios na modificação de cor e superfície de esmalte dental / Samuel da Silva Palandi. – Piracicaba, SP : [s.n.], 2021.

Orientador: Vanessa Cavalli Gobbo.
Coorientador: Matheus Kury Rodrigues.
Trabalho de Conclusão de Curso (graduação) – Universidade Estadual de Campinas, Faculdade de Odontologia de Piracicaba.

1. Carbono ativado. 2. Peróxido de carbamida. 3. Escovação dentária. 4. Dentifrícios. 5. Dentes - Clareamento. I. Cavalli, Vanessa,1977-. II. Kury-Rodrigues, Matheus,1991-. III. Universidade Estadual de Campinas. Faculdade de Odontologia de Piracicaba. IV. Título.

Informações adicionais, complementares

Título em outro idioma: Effects of activated charcoal powder combined with toothpastes on enamel color change and surface properties
Palavras-chave em inglês:
Carbon, activated
Carbamide peroxide
Toothbrushing
Dentifrices
Teeth - Bleaching
Titulação: Cirurgião-Dentista
Data de entrega do trabalho definitivo: 17-05-2021

DEDICATÓRIA

Dedico o presente trabalho principalmente aos meus pais que tudo me proporcionaram, aos quais devo todo o apoio na conquista de um sonho.

E também dedico a todo o suor e lagrima derrubado até o presente dia por mim.

AGRADECIMENTOS

A Faculdade de Odontologia de Piracicaba por me abrigar durante todo o período de aprendizado.

A Prof. Dr. Vanessa Cavalli por toda a confiança em meu potencial e por todas as oportunidades que me proporcionou.

Aos meus dois grandes coorientadores e companheiros Matheus Kury e Mayara Zaghi por todo o apoio e ensinamentos que me deram.

Aos meus queridos amigos Kiq, Kaio, Fepa e Vinão aos quais compartilharam comigo dos melhores anos, maiores alegrias e gargalhadas, e primordial apoio durante o curso.

A todos que de alguma forma contribuíram na conclusão desta etapa e deste sonho.

RESUMO

Este estudo avaliou a alteração de cor e rugosidade do esmalte submetido à escovação com carvão ativado em pó (COAL), associado ou não à escovação com dentifrício convencional (RT) ou clareador (WT), em comparação ao clareamento com peróxido de carbamida 16% (CP). Blocos de esmalte/dentina pigmentados com chá preto, foram distribuídos em grupos, de acordo com escovação simulada e/ou clareamento (n=10): COAL/RT, COAL/WT, COAL, CP/RT, CP/WT, CP, RT, WT e CONT (sem tratamento). A alteração de cor (ΔE_{00}) e as coordenadas L* a* b* foram avaliadas por espectrofotômetro digital. A rugosidade (Ra) e morfologia do esmalte foram analisadas utilizando um rugosímetro e microscópio eletrônico de varredura, respectivamente. Os dados foram submetidos à ANOVA dois fatores (ΔE_{00}) ou três fatores com medidas repetitivas (L*, a*, b* e Ra) e ao teste Tukey (α=5%). COAL apresentou ΔE_{00} superior a CONT ($p < .05$), mas similar a COAL/RT e COAL/WT ($p > .05$). A escovação prévia com COAL não aumentou a ΔE_{00} e Ra dos grupos RT e WT ($p > .05$). Os grupos CP, CP/RT e CP/WT promoveram os maiores valores de ΔE_{00} e L*, e menores de b* ($p > .05$). A escovação com COAL aumentou Ra ($p < .001$) e alterou a topografia do esmalte. CP/WT aumentou Ra, e alterou a topografia do esmalte em menor extensão do que COAL. Conclui-se que a escovação com carvão ativado aumentou a rugosidade e alterou a morfologia do esmalte, não promoveu alteração de cor quando associado aos dentifrícios e não atingiu a eficácia de CP.

Palavras-chave: Carbono ativado. Peróxido de carbamida. Escovação dentaria. Dentifrícios. Dentes - Clareamento.

ABSTRACT

This study evaluated color change and roughness of enamel submitted to brushing with activated carbon powder (COAL), combined or not with brushing with conventional (RT) or whitening toothpaste (WT), in comparison to whitening with 16% carbamide peroxide (CP). Enamel/dentin blocks stained with with black tea were allocated in groups, according to simulated brushing and / or whitening (n = 10): COAL / RT, COAL / WT, COAL, CP / RT, CP / WT, CP, RT, WT and CONT (without treatment). Color change (ΔE_{00}) and L* a* b* coordinates were evaluated by a digital spectrophotometer. Roughness (Ra) and enamel morphology were analyzed using a rugosimeter and scanning electron microscope, respectively. Data were submitted to ANOVA two factors (ΔE00) or three factors with repetitive measures (L *, a *, b * and Ra) and to Tukey test (α = 5%). COAL showed higher ΔE_{00} than CONT (p <.05), but similar to COAL / RT and COAL / WT (p> .05). Previous brushing with COAL did not increase the ΔE_{00} and Ra of the RT and WT groups (p> .05). CP, CP/RT and CP / WT groups displayed the highest ΔE_{00} and L* values , and the lowest b* values (p> .05). Brushing with COAL increased Ra (p <.001) and altered the enamel topography. CP / WT significantly increased Ra, and changed topography to a lesser extent than COAL. It was concluded that brushing with activated carbon powder increased roughness and changed enamel morphology, did not cause color change when combined with toothpaste and did not reach the effectiveness of CP.

Keywords: Carbon, activated. Carbamide peroxide. Toothbrushing. Dentifrices. Teeth – Bleaching.

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1 INTRODUÇÃO

Nas últimas décadas, têm-se observado um crescente aumento da busca pela melhora da estética do sorriso, sendo que uma grande parcela da população brasileira (86%) relata o desejo de ser submetido ao clareamento dental como possível ferramenta para melhorar a qualidade de vida (Silva et al., 2018). Como consequência, o clareamento dental tornou-se um dos procedimentos estéticos mais realizados pelos cirurgiões-dentistas, sendo um tratamento conservador e minimamente invasivo, que busca reverter descolorações dentais de origem congênita ou adquirida (Rodríguez-Martínez et al., 2019).

O clareamento supervisionado pelo dentista pode ser realizado com agentes clareadores, como o peróxido de carbamida (PC) ou de hidrogênio (PH), em diferentes concentrações (Kwon e Wertz, 2015). A concentração do agente clareador é determinada pelo regime planejado pelo dentista, de acordo com as necessidades do paciente, variando de 3.5 a 20% no clareamento caseiro supervisionado, até 45% no clareamento de consultório (Kwon e Wertz, 2015; Rodríguez-Martínez et al., 2019). Hipotetiza-se que o baixo peso molecular do peróxido permite a difusão do mesmo pelo esmalte dental, até atingir a dentina, onde os radicais livres do PH seriam responsáveis por clivarem os cromóforos dentinários, conhecidos como os pigmentos intrínsecos e responsáveis pela coloração dentária (Joiner, 2006).

Além disso, métodos clareadores sem a necessidade de supervisão do cirurgião-dentista são relatados na literatura. Os produtos de prateleiras como dentifrícios, enxaguantes, tiras e pincéis, são também conhecidos como over-the-counter (OTC), constituindo uma categoria de clareamento com eficácia controversa e mecanismos de ação distintos (Devila et al., 2019; Naidu et al., 2020). Por exemplo, enquanto alguns dentifrícios com propostas clareadoras agem seguindo o princípio de remoção de pigmentações extrínsecas da superfície do esmalte por meio de abrasivos específicos, outros incorporam partículas, como dióxido de titânio e covarina azul, com o intuito de promover uma alteração óptica e, por conseguinte, os dentes apresentarem um aspecto mais claro (Vieira-Junior et al., 2016; Naidu et al., 2020).

Embora faltem evidências para indicar se existe um tipo de dentifrício mais eficaz para alterar a coloração dentária, uma recente revisão sistemática demonstrou redução da pigmentação extrínseca para todos os produtos de clareamento testados (Soeteman et al., 2018). Há também evidências de que os dentifrícios com propostas clareadoras resultam em alteração de cor significativamente superior em comparação a dentifrícios regulares (Devila et al., 2019).

Entretanto, o aumento da rugosidade superficial (Ra) do esmalte submetido à escovação com dentifrícios clareadores foi observado, independentemente da combinação destes dentifrícios com agentes à base de peróxido (Hilgenberg et al., 2011; Attia et al., 2015). Tais eventos poderiam ser explicados pelo fato dos dentifrícios com propostas clareadoras apresentarem maior abrasividade (Leandro et al., 2008; Paula et al., 2010; Soeteman et al., 2018;), bem como os géis clareadores apresentam um mecanismo de ação oxidativo com possíveis variações de pH (Cavalli et al., 2019). Dessa maneira, é necessário cautela e boa orientação ao paciente durante o tratamento clareador. É importante ressaltar que dentifrícios branqueadores também podem ocasionar efeitos adversos a tecidos adjacentes como os do periodonto (Havwood et al., 1994; Joiner et al., 2008).

Nos últimos anos, produtos naturais têm sido propostos em diversos produtos de cuidado oral ou facial (Kim e Seock, 2009), como o carvão ativado. Tal agente foi introduzido recentemente em produtos destinados à escovação dental, com a sugestão de promover efeito clareador por meio de um método natural (Brooks et al., 2017; Franco et al., 2020). O carvão ativado difere do carvão comum por passar por um processo de queima com temperaturas mais altas e por maior tempo, com adição de gases oxidantes, volatilizando assim mais substâncias presentes na fonte orgânica e obtendo uma estrutura interna muito mais porosa, o que permite maior absorção (Mohammad-khah e Ansari, 2009).

Atualmente, o carvão ativado pode ser encontrado em formato de pó ou incorporado diretamente nos dentifrícios com proposta clareadora (Palandi et al., 2020). O carvão ativado em formato de pó tem como fonte primaria a casca de cocos secos (https://blog.carvvo.com.br/carvao-ativado/), o que confere ao produto uma matéria prima renovável e ecológica, tendo grande apelo ao público que procura hábitos mais naturais (Kim e Seock, 2009). Entretanto, uma das principais desvantagens e preocupações em relação à escovação com pó de carvão ativado baseia-se na ausência de fluoreto na sua composição (Brooks et al., 2017). Uma vez que a compra do produto não necessita de prescrição odontológica, e nem recomendação dos dentistas, os pacientes erroneamente substituírem a escovação com o dentifrício com fluoreto pela escovação com o pó de carvão ativado, aumentando o risco à cárie (Franco et al., 2020; Palandi et al., 2020). Além da escassez de estudos avaliando a eficácia clareadora destes produtos, faltam estudos que avaliem a abrasividade e os efeitos do pó de carvão de ativado na superfície do esmalte dental.

Outro aspecto ainda não investigado consiste no efeito da escovação com carvão ativado associado a dentifrícios regulares ou dentifrícios com proposta clareadora. Em geral, os fabricantes instruem os consumidores a realizarem a escovação com carvão ativado previamente aos procedimentos de higiene oral de rotina por um período que pode variar de 14 dias a 6 meses (“How to use”, Carvvo). O procedimento combinado (escovação com pó de carvão e escovação com dentifrício) aumenta consideravelmente o tempo de exposição das estruturas dentais à outros tipos de abrasivos, fato que pode acarretar em impactos negativos na estrutura dental. Ainda, cabe ressaltar que a escolha do dentifrício a ser utilizado na escovação compete aos próprios pacientes na maioria dos casos (Adegbulugbe, 2007), o que poderia acarretar na combinação de dois agentes altamente abrasivos e com proposta clareadora.

Desta forma, este estudo teve como objetivo avaliar a alteração de cor, a rugosidade e morfologia de superfície do esmalte, em dentes submetidos à escovação simulada com pó de carvão ativado, associado ou não a dentifrícios convencionais ou com propostas clareadoras, comparando ao tratamento clareador com PC 16%.

2 ARTIGO: EFFECTS OF ACTIVATED CHARCOAL POWDER COMBINED WITH TOOTHPASTES ON ENAMEL COLOR CHANGE AND SURFACE PROPERTIES

Artigo publicado: Palandi SDS, Kury M, Picolo MZD, Coelho CSS, Cavalli V. Effects of activated charcoal powder combined with toothpastes on enamel color change and surface properties. J Esthet Restor Dent. 2020 Dec;32(8):783-90. doi: 10.1111/jerd.12646. (Anexos 2 e 3)

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ABSTRACT

Aim: To evaluate the effects of activated charcoal powder (COAL) combined with regular (RT) or whitening (WT) toothpastes on enamel color and surface in comparison to carbamide peroxide (CP).

Methods: Dental blocks (n=10/group) were randomly divided into COAL, COAL/RT, COAL/WT, CP, CP/RT, CP/WT, RT, WT, and CONT (without treatment). Simulated toothbrushing and whitening treatments were followed by colorimetric (ΔE_{00}, L*, a*, b*), surface roughness (Ra) and enamel topography assays. ΔE_{00} was submitted to two-way ANOVA and Tukey test. Color coordinates and Ra were tested with three-way repeated measures ANOVA (α=5%).

Results: COAL exhibited greater ΔE_{00} than CONT (*p* = .048), but it did not enhance ΔE_{00} promoted by RT or WT (*p* > .05). COAL alone increased Ra (*p* < .001) and altered enamel topography. COAL did not increase Ra caused by RT and WT (*p* > .05). CP exhibited the highest ΔE_{00} (*p* < .05), but it raised Ra and changed enamel topography to a less extent than COAL.

Conclusion: Even though charcoal powder did not increase enamel Ra when combined with toothpastes, the topography was negatively impacted by COAL. Also, COAL was unable to enhance the color change of RT and WT, or reach the effectiveness of CP.

Clinical Significance: The use of activated charcoal-based product, claimed as a natural whitener, before brushing with toothpastes is not only ineffective to change the color of teeth, but also it might result in alterations on the enamel surface. Whitening with CP, instead, was effective during the same period of treatment, which still represents a more appropriate technique to whiten teeth.

Keywords: Whitening, Toothbrushing, Charcoal, Toothpastes, Carbamide Peroxide

INTRODUCTION

At-home tooth whitening supervised by dentists is a technique widely described in the literature as effective and safe for patients.[1] Low-concentrated peroxide agents are credited to promote color change possibly due to the interaction of oxygen free radicals with pigments of dentin, the so-called chromophores, which defines the color of this dental tissue.[2] It is hypothesized that this phenomenon breaks down the intrinsic pigments into smaller molecules, thereby whitening tooth structure.[2,3] However, extrinsic tooth staining may also play a role in the tooth color.[3] Intake of staining such as tea, coffee, red wine, or smoking habits are involved in extrinsic discoloration.[4] Therefore, recent researches have investigated the impact of toothpastes on changing tooth color. [5,6]

Although several types of whitening toothpastes with different mechanism of actions are available, it is known that the abrasivity of a toothpaste itself could remove extrinsic staining.[7] Other than that, titanium dioxide or blue covarine can be added to the products, depositing a thin layer of these pigments. As a consequence, teeth could seem brighter because of changes in the interaction with incident light.[8] Even though evidence lacks to indicate if there is a most effective type of toothpaste to change the color of teeth, a systematic review demonstrated reduction of extrinsic pigmentation for all whitening products tested.[9] A more recent systematic review and meta-analysis pointed out that whitening toothpastes produces a more effective whitening effect compared to the regular ones.[5] Nevertheless, an increase in surface roughness has already been detected *in vitro* in enamel submitted to brushing with whitening toothpastes, regardless of the combination of the whitening toothpastes with peroxide-based gels.[10] Thus, caution should be taken when combining both methods clinically.

In recent days, activated charcoal-based powders and toothpastes have gained popularity among patients.[11,12] The majority of charcoal-containing products are claimed to promote color change, being advertised as over-the-counter whitening options.[13] Similar to abrasion provoked by toothpastes, activated charcoal is expected to act on extrinsic pigments and not on dentin chromophores.[7] However, literature is not consistent with the whitening effectiveness of charcoal powder as well as the safety of this product to the enamel surface.[11,14] Moreover, the use of activated charcoal raises concerns about the risk of caries due to the predominance of non-fluoridated charcoal-containing products marketed for patients.[12] Aiming to reverse such disadvantage, some manufacturers indicate toothbrushing with activated charcoal powders before use of toothpastes.[15] To date, there is no evidence of the effectiveness of charcoal powder used in combination with regular or whitening toothpastes, and studies lack to define the effect of charcoal-based products on surface properties of enamel, especially when this product is combined with toothpastes (regular or whitening).

Hence, this study evaluated the color change and surface properties of artificially stained enamel submitted to brushing with activated charcoal powder combined or not with regular or whitening toothpastes in comparison to low-concentrated carbamide peroxide (CP). The null hypotheses evaluated were that charcoal powder 1: would not enhance color change promoted by toothpastes, 2: would not be as effective as low-concentrated CP, and 3: would not influence the enamel surface roughness.

MATERIAL AND METHODS

Experimental Design

The bovine enamel blocks (n= 10/group) were submitted to the factors (a) “whitening agent” in three levels, considering the activated charcoal powder (COAL), 16% carbamide peroxide (CP) and the absence of a whitening agent, and (b) “toothpastes” also in three levels: regular (RT), whitening (WT) and without toothbrushing. Color change (ΔE_{00}) and surface roughness (Ra) were measured after the staining protocol (baseline - T_0), and 7 days elapsed from whitening protocols (T_B). The enamel surface morphology was observed under scanning electron microscopy (SEM) at T_B.

Specimens Preparation

Bovine incisors were extracted, cleaned and stored in 0.1% thymol solution at 4^0C for no longer than 60 days. Ninety teeth without cracks and defects in the enamel were selected. The roots were cut in a low speed saw (Isomet, Buehler;

Lake Bluff, IL, USA) under refrigeration 2 mm below the cementoenamel junction. Afterward, blocks (7mm x 4mm and 3 mm thick) were obtained from the central region of the crown. The area corresponding to dentin was flattened using a polishing machine (Arotec Ind. Com., São Paulo, Brazil) with silicon carbide papers #600 under water-cooling. The outer enamel surface of each block was finished with aluminum oxide grit #600, 1200, and 2000. The blocks were polished using diamond aqueous suspensions (6 μm, 3 μm, 1 μm and 0.25 μm, Metaldi Supreme, Buehler, Lake Bluff, IL, USA) and polishing cloths (Buehler, Lake Bluff, IL, USA), and ultrasonically cleaned in distilled water for 10 minutes. The specimens were artificially stained with a solution of black tea (2 g of tea - Leão, São Paulo, SP, Brazil - boiled in 100 ml of distilled water) for 24 h.[16,17] The dentin walls were isolated using a nail varnish before the blocks being completely immersed in the tea infusion and continuously stirred.

Group Division

The specimens were divided into groups (n=10) according to the association of the factors under study:

- COAL/RT: simulated toothbrushing with activated charcoal powder and regular toothpaste;
- COAL/WT: simulated toothbrushing with activated charcoal powder and whitening toothpaste;
- COAL: brushing only with activated charcoal powder;
- CP/RT: whitening with 16% CP and simulated toothbrushing with regular toothpaste;
- CP/WT: whitening with 16% CP and simulated toothbrushing with whitening toothpaste;
- CP: whitening with 16% CP;
- RT: simulated toothbrushing only with regular toothpaste;
- WT: simulated toothbrushing only with whitening toothpaste;
- CONT: without whitening or simulated toothbrushing (untreated).

Brushing protocol

The specimens of each treatment group were attached to a mechanical brushing machine (MSet, Nucci ME, São Carlos, SP, Brazil) with the enamel surface positioned upwards. Forty-hundred and twelve brushing cycles (824 strokes) were performed to simulate 14-day brushing with a frequency of 5Hz and under a load of 200g.[18] All specimens were brushed with a soft nylon toothbrush (Colgate Twister®; Colgate-Palmolive Company, São Paulo, SP, Brazil), and immersed in a slurry prepared with toothpastes or charcoal powder and purified water, in a 1:3 proportion. After the brushing cycles, the samples were washed in running water, dried with paper towels, and stored in artificial saliva [1.5 mM calcium ($CaCl_2$), 0.9 mM phosphate (NaH_2PO_4), 0.15 mM KCl, pH 7.0] at 37°C,[19] and the solution was renewed every two days.

CP Whitening Protocol

Whitening with 16% CP was performed for 14 days (4 hours/daily). An amount of 0.01 g of the gel was applied over the enamel surface of the specimens (CP groups). The gel was removed from the surface with purified water at the end of each session. The materials used are described in Table 1**.** Specimens were stored in the artificial saliva previously described among whitening intervals at 37°C, and the solution was also renewed every two days.[19]

Table 1. Materials used in the project.

Commercial name (Manufacturer, Address and Batch Number)	Composition	Manufacturer's Instruction
COAL: Carvvo (Carvvo®, Salvador, BA, Brazil - 302145869441218)	Activated charcoal, kaolin clay, orange essential oil.	Brush teeth gently with a soft toothbrush for 3 minutes and rinse water. Afterwards, brush teeth with toothpaste. It is recommended to repeat this procedure twice a day for 14 days. Continuous use, twice a week, is recommended after the first week.
RT: Colgate Luminous White (Colgate Palmolive Company, São Paulo, SP, Brazil - 8337BR123B)	Sodium carbonate, water, sorbitol, hydrated PEG-12, silica glycerin, sodium lauryl sulphate, cellulose gum, flavor, tetrasodium pyrophosphate, potassium hydroxide, phosphoric acid, cocamidopropyl betaine, 0.32% sodium fluoride (1450 µg F/g), benzyl alcohol, saccharin sodium, sodium hydroxide, titanium dioxide (CI 77891).	Use during toothbrushing three times a day or following the recommendations of a dentist.
WT: Colgate Máxima Proteção Anticáries (Colgate Palmolive Company, São Paulo, SP, Brazil - 8326BR121J)	Calcium carbonate, water, glycerin, sodium lauryl sulphate, sodium monofluorophosphate, flavor, cellulose gum, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, sodium hydroxide, sodium monofluorophosphate (1450 µg F/g).	Use during toothbrushing three times a day or following the recommendations of a dentist.
CP: Whiteness Perfect (FGM Dental Products, Joinville, SC, Brazil - 191118)	16% carbamide peroxide, neutralized carbopol, inert filler, glycol and deionized water, pH was informed by the manufacturer as 7.0.	Apply the gel on a customized acetate tray and wear it for 3-4 hours for no longer than 2 weeks. No recommendations are given about toothbrushing during the treatment.

Colorimetric evaluation

Three measurements were taken from the enamel surface using a digital spectrophotometer (EasyShade, Vita Zahnfabrik, Bad Säckingen, Germany) after staining (T_0), and 7 days elapsed from the end of bleaching (T_B). Mean values were obtained for each specimen. The spectrophotometer was fixed in a platform with the tip positioned downwards to read the dental blocks, which were placed in a standardized background (an opaque tile).

Data were recorded according to the CIE system: L* (black to white), a* (red to green), b* (yellow to blue). However, the color change (T_B and T_0) was calculated by means of the CIEDE2000 system, which uses h (hue) and C (chroma) values: $\Delta E_{00} = [(\Delta L'/K_LS_L)^2 + (\Delta C'/K_CS_C)^2 + (\Delta H'/K_HS_H)^2 + R_T*(\Delta C'/K_CS_C)*(\Delta H'/K_HS_H)]^{1/2}$. [1,20]

Surface Roughness (Ra)

Surface roughness was determined after the staining protocol (T_0), and 24 hours after whitening protocols using a rugosimeter (Surfcorder SE 1700, Kosalab), calibrated at a 0.25 mm cut-off and 0.2 mm/s speed. Three measurements were performed rotating the specimen 45^o, and the mean value for each sample was calculated. Following specimen preparation and prior to bleaching and toothbrushing, the specimens were randomly allocated into the groups. One-way ANOVA test detected no statistical differences in the Ra values between groups (p > .05) before treatments.

Morphology of enamel surface

Three samples of each experimental group were allowed to dry in an oven overnight and sputter-coated with gold (MED 010, Balzers, Balzer, Liechtenstein). Enamel surface morphology was qualitatively observed under SEM (JEOL-JSM, 6460LV, Tokyo, Japan) at 2000x magnification.

Statistical analyses

Data were submitted to exploratory analyses to verify the normal distribution (Shapiro-Wilk) and homoscedasticity (Brown-Forsythe). Values obtained from the colorimetric evaluation were submitted to two-way ANOVA (ΔE_{00}), or to three-way repeated measures ANOVA (L*, a*, b*, and Ra) and Tukey test. A 0.05 level of significance was adopted for all analyses.

RESULTS

Color Change

Two-way ANOVA test revealed that "whitening agents" significantly affected the ΔE_{00} results (p < .001). The "toothpastes" factor (p = .670) and the interaction "whitening agents*toothpastes" (p = .197) did not influence ΔE_{00}. Pairwise comparisons (Figure 1) showed that ΔE_{00} was statistically higher in CP groups than in the others (p < .05), regardless of the brushing type. COAL alone exhibited higher color change than CONT (p = .048), but COAL was similar to COAL/RT (p = .724) and COAL/WT (p = .230). COAL/RT and COAL/WT were statistically similar to RT (p = .289) and WT (p = .170), respectively. WT presented ΔE_{00} higher than CONT (p = .031), but similar to RT (p = .114). Three-way repeated measures detected that only "whitening agents" (p < .001) significantly affected the results of L*, a* and b* coordinates. Figure 2 shows a significant decrease in luminosity (L) and yellow*blue axis (b*) only in CP-groups, which exhibited higher L* and b* values than COAL/RT and COAL/WT at T_B (p < .05). Regarding the a* coordinate, differences over time were also observed for COAL-groups, regardless of the type of toothpaste combination (p < .05).

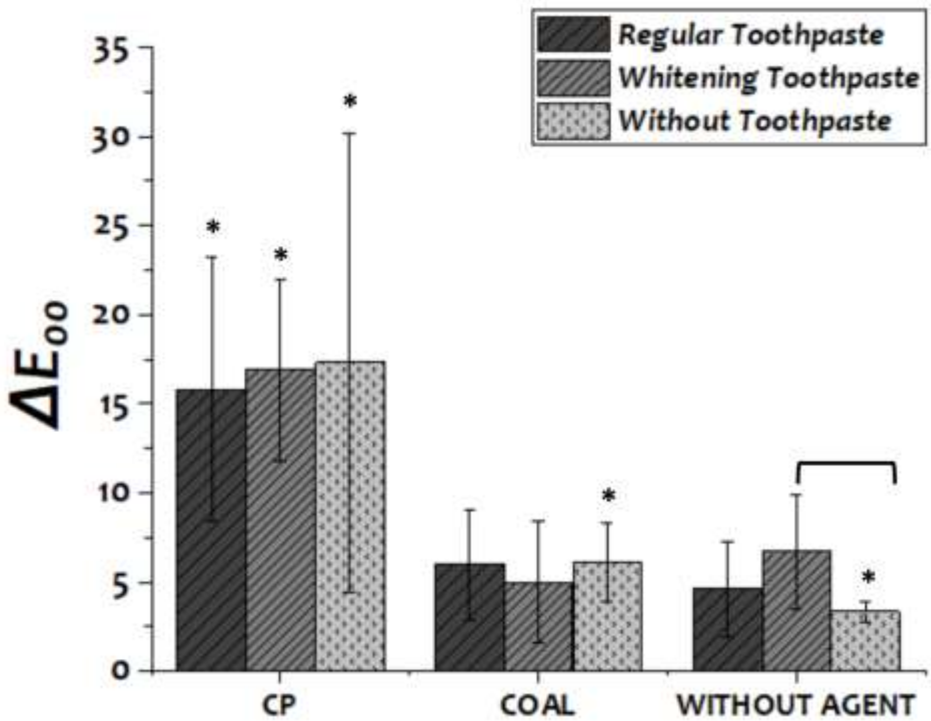

Figure 1. Mean values and standard deviations of ΔE_{00} obtained for whitening and toothpaste combinations over time. Means with symbols differ at 5% according to two-way ANOVA and Tukey test.

Bars connected by the bracket differ statistically within the same whitening agent (x axis) over time (T_B-T_0) Asterisks indicate statistical differences and compare toothpaste separately, according to the whitening treatment performed, at a specific time.

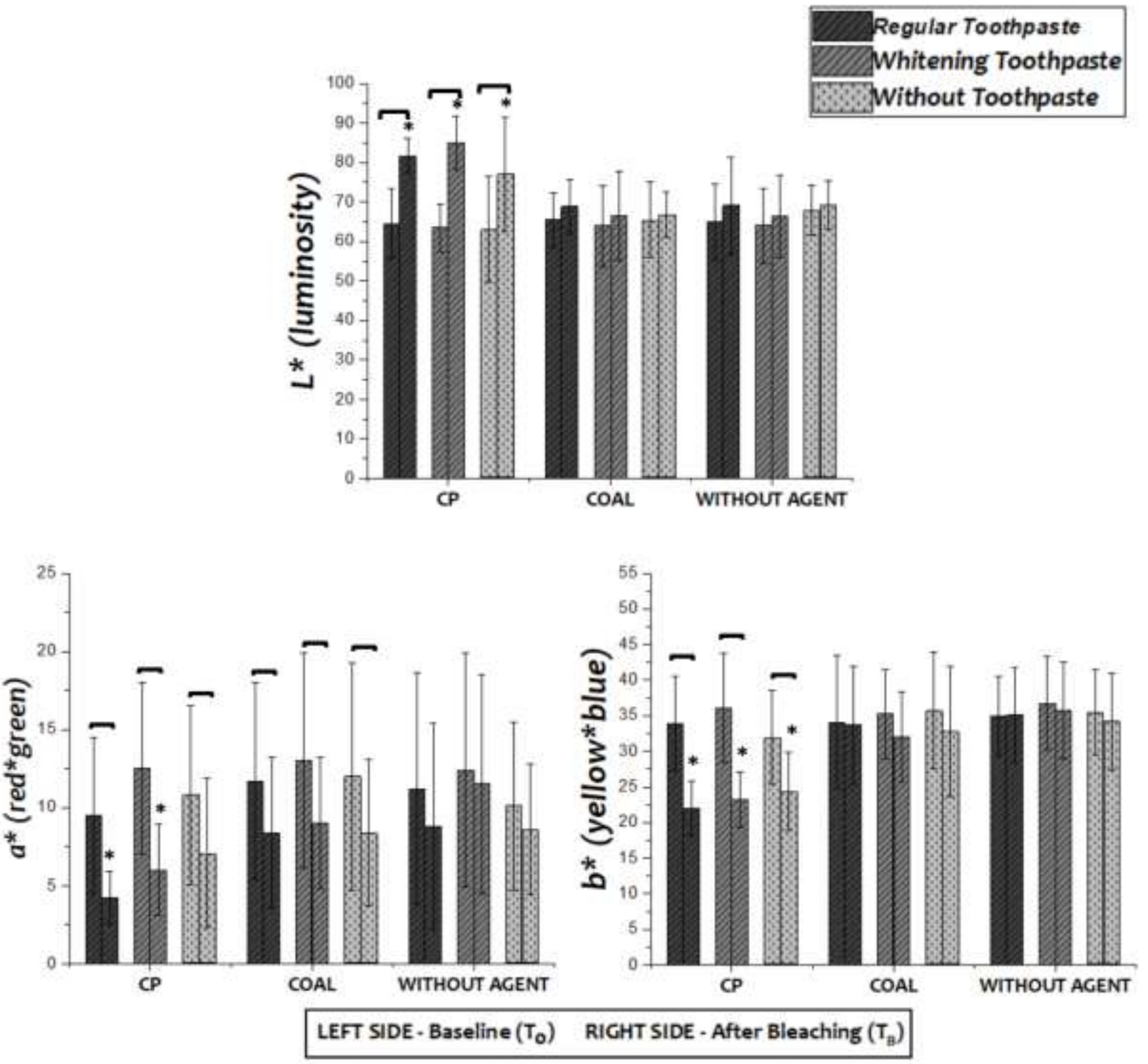

Figure 2. Mean values and standard deviation of L*, a* and b* obtained for whitening and toothpaste combinations over time (T_B and T_0). Mean values and standard deviations of ΔE_{00} obtained for whitening and toothpaste combinations over time. Means with symbols differ at 5% according to two-way ANOVA and Tukey test. Bars connected by the bracket differ statistically within the same whitening agent (x axis) over time (T_B-T_0) Asterisks indicate statistical differences and compare toothpaste separately, according to the whitening treatment performed, at a specific time.

Surface Roughness

Figure 3 displays the results of surface roughness analysis, in which no differences were detected at baseline (T_0) among groups (p > .05). Both CP and COAL-groups significantly increased enamel roughness after treatments (T_B) (p < .05), with the exception of COAL/WT (p > .05). CP/WT exhibited Ra significantly greater than CP and toothpaste-corresponding groups (p < .05). The combination of COAL with toothpastes did not increase the surface roughness of RT and WT groups, and RT and WT exhibited higher Ra values than CONT (p < .05) at T_B.

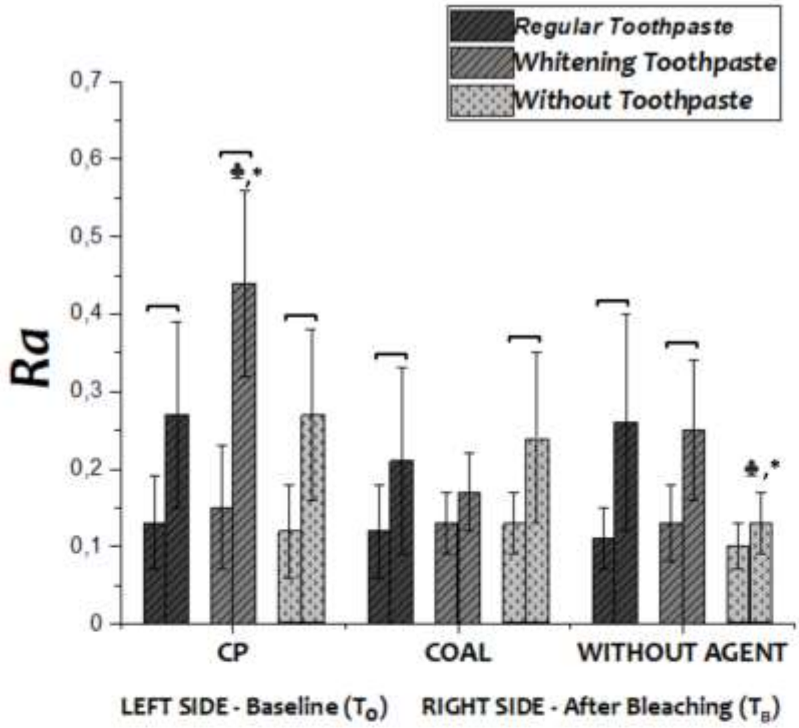

Figure 3. Mean values and standard deviation of Ra obtained for whitening and toothpaste combinations before (T_0) and after treatments (T_B).

Morphology of enamel surface

Representative images of SEM can be observed in Figure 4 COAL promoted smoothness loss in comparison to CONT groups (CONT, WT and RT). RT and WT alone showed alterations compatible with bristles marks. Areas with depressions (arrows) and discontinuity along the surface could be observed. The combination of COAL with toothpastes (RT or WT) did not promote the same surface morphology pattern. CP alone maintained the surface flatness similar to that observed for CONT, but the combination with toothpastes (RT and WT) caused enamel depressions (arrows) and increase surface porosity.

COAL/RT and COAL/WT groups exhibited slight alterations on the enamel surface, e.g. low level of porosity and loss of flatness, while COAL group presented pronounced and deeper porosities sites and visible depressions (arrows) throughout the surface. Although no surface alterations could be observed in the CP-treated group, the combination of CP with both RT and WD resulted in a pattern of continuous depressions or porosities (arrows), which was more observed in CP/WT. Brushing with RT and WT led to bristles marks on enamel surface, which was not seen in CONT group.

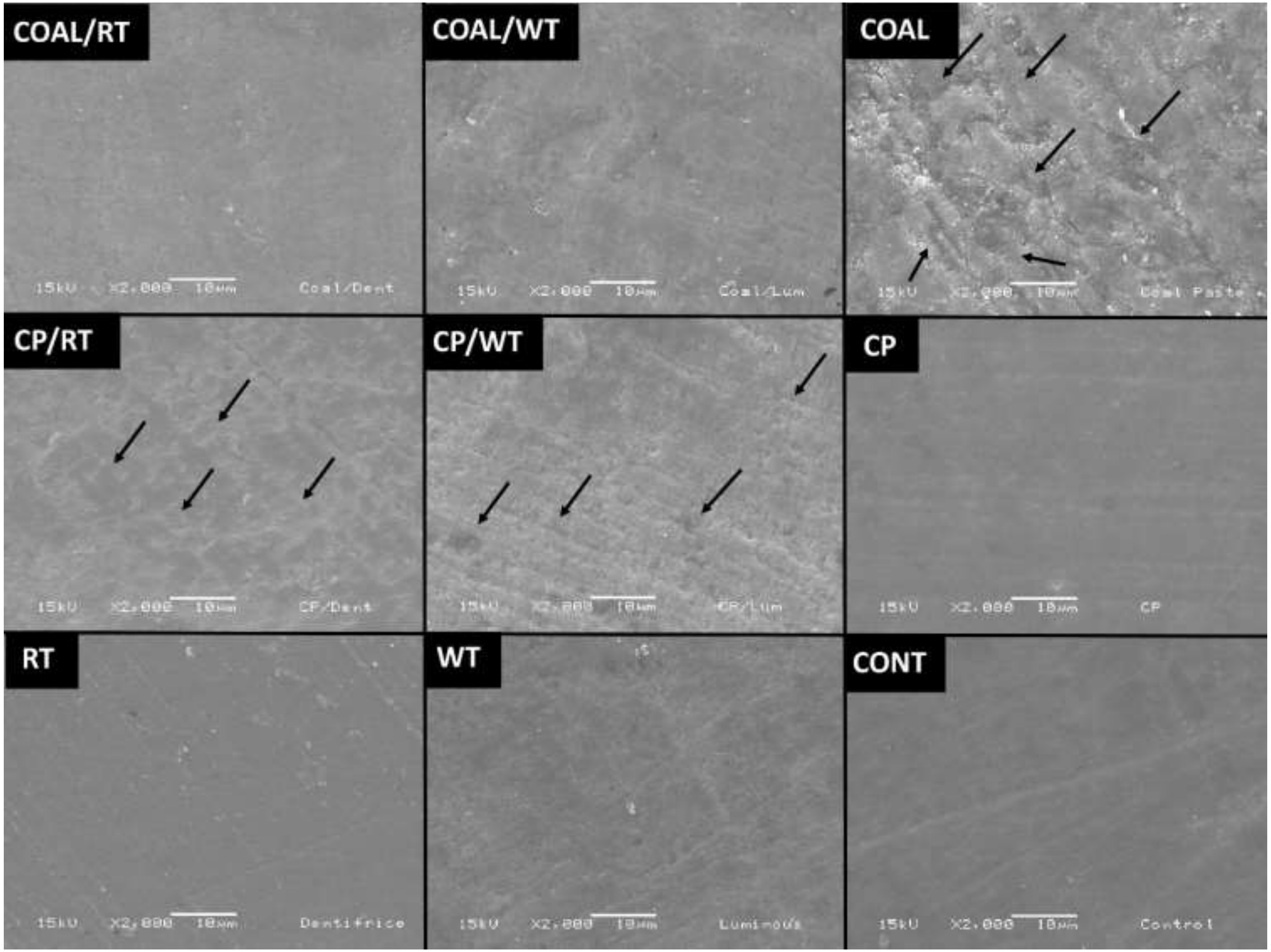

Figure 4. Representative images of enamel surface obtained by SEM after treatments.

DISCUSSION

The first null hypothesis was accepted because the combination of charcoal powder with toothpaste did not enhance the color change promoted by toothpaste, regardless of the brushing protocol. As the charcoal used in this study is free of fluoride, the manufacturer recommends its use before brushing with fluoridated toothpastes.[15] Therefore, two types of toothpaste (regular and whitening) with the same abrasive particle were selected. Also, the toothpastes contained at least 1000 ppm of fluoride, which represents an appropriate concentration to prevent risk of caries.[21] The results indicated that charcoal powder would add an additional step in brushing without benefits for the *in vitro* color change already promoted by regular or whitening toothpastes. This study combined COAL with more than one type of toothpaste considering the large number of products available for the patients, who normally holds the decision of which toothpaste will use.

Simulated toothbrushing with activated charcoal significantly decreased a* values, and this outcome occurred possibly due to the staining protocol adopted with black tea, which deposits extrinsic pigments on the enamel surface.[3,7,17] However, it should be pointed out that isolation of dentin previous to black tea immersion prevented the impregnation through dentin tubules. The reduction in tooth's yellow appearance (b*) could represent the action of bleaching gels by-products in the colored chromophores located in dentin, which was probably hampered by the abrasive agents due to their superficial action.[7] Therefore, it should be assumed that abrasiveness of charcoal powder could have been responsible for removing pigments adhered to outer enamel surface, but it did not affect intrinsic pigments.

To date, there is a lack of data in the literature about the effect of activated charcoal powder on whitening. Vaz et al.[22] were the first to assess an activated charcoal-containing toothpaste, and the authors showed *in vitro* superior whitening effectiveness for microbreads abrasives and blue covarine toothpastes. Meta-analysis evaluations pointed out that the majority of whitening toothpastes evaluated exhibited a beneficial effect on extrinsic discoloration removal in comparison to regular toothpaste.[5,9] In the current study, WT resulted in color change significantly higher than CONT. Nonetheless, the combination of charcoal powder eliminated this difference. As the WT herein used is based on the interaction of pyrophosphates with stains,[23] previous brushing with activated charcoal could have interfered in this process. However, differences between COAL/RT and COAL/WT were not clinically perceptible.[20] Also, it is important to highlight that WT changed color to the same extent as RT, which brings into question the real necessity to use whitening toothpastes. Although ΔE_{00} values of charcoal and toothpaste-treated groups were perceptible,[20] the presence of artificial staining (black tea) to standardize color parameters and to evaluate the capability of extrinsic staining removal could have exacerbated color change. Therefore, these data should be cautiously extrapolated to clinical conditions. Besides, the clinical perceptible ΔE_{00} difference exhibited by control group was probably a consequence of staining dissolution (red and yellow) promoted by storage in artificial saliva.

Other than brushing protocols, the comparison between activated charcoal and low-concentrated CP was conducted, because manufacturer states that activated charcoal powder is a natural whitener,[15] which could increase the expectation of the patients. As richly described,[1] peroxide led to significant changes in tooth's luminosity, red and yellow appearance, regardless of the combination with RT or WT. Most importantly, alteration towards blue in b* coordinate indicated that CP level was the only whitening agent to possibly cause intrinsic staining removal.[3] Therefore, the second null hypothesis that charcoal would not reach the effectiveness of CP was also accepted. Hence, it is necessary to aware patients that, apart from advertisements promoting successful treatments with charcoal-based products, peroxides will promote more effective and long-term results. Even though COAL and toothpastes were compared also to CP in this study, it is important to note that the statistical differences found among these groups might be explained by the different mechanisms of actions of such products. As previously cited, while toothpastes and powders would act on the extrinsic stained adhered to enamel, CP is believed to cleave organic chromophores present in dentin.[3]

Charcoal-treated groups increased surface roughness, rejecting the third and last null hypothesis. Nevertheless, Ra was statistically equal between charcoal and CP alone. While the charcoal effect on the surface is likely to be a result of abrasivity or brushing movements, CP mechanism of action may be responsible for roughness alterations.[2,10] The CP is combined with urea within the bleaching gel, which is decomposed into hydrogen peroxide and ammonium when getting in contact with teeth. The low-molecular weight of hydrogen peroxide promotes diffusion through interprismatic spaces, which may alter enamel surface morphology.[2,17] Even though this was not the main purpose of this research, it was observed that the combination

CP/WT increased Ra in comparison to CP and CP/RT, which is an important evidence corroborating another finding that combination of CP with whitening toothbrushing may compromise enamel surface roughness.[10] Although SEM analysis of CP/RT indicated continuous irregularity of the enamel surface, it was less pronounced than that of CP/WT group. Based on this observation and on the fact that CP/RT promoted high-perceptible color change[20] and lower surface roughness than CP/WT, regular toothpastes could the best option during whitening with CP. *In situ* and *in vivo* studies could help to clarify the impacts of toothbrushing during tooth whitening.

On the contrary, the combination of COAL with WT did not statistically increase the surface roughness. However, it is important to emphasize that Ra maintenance is not a warranty of enamel surface preservation, as this scenario could be a result of enamel surface loss. As COAL/WT was exposed to twofold brushing cycles because that charcoal powder is utilized before toothpaste-containing toothbrushing, the surface could have been polished to a greater extent than that observed in CP/WT and WT groups. A limitation of our study was that the loss of surface was not measured to indicate whether the 1,648 strokes performed in COAL/WT and COAL/RT groups were capable to promote a surface polishing greater than the respective control groups with 824 strokes. The number of strokes was based on one-month simulation of normal toothbrushing.[18] As both charcoal and CP's manufacturers stipulate two weeks of treatment, 412 cycles were performed for COAL, RT and WT protocols. Previous reports have indicated that longer brushing cycles diminished the volume of the enamel surface,[24] and further studies could enlighten if the same is true for charcoal-based products.

COAL group exhibited representative microscopy areas with irregular depressions, which were not observed in groups COAL/RT and COAL/WT. Even though studies lack to explain the effect of charcoal on dental morphology, an *in vitro* evaluation using another charcoal powder showed low abrasiveness on dentin.[25] However, the powder evaluated in that study also contained kaolin clay, which could not be suitable for brushing due to its shape and abrasitivity.[25]

In vivo evidence demonstrated that enamel properties are preserved after whitening with peroxide gels in the presence of human saliva and adhered pellicle.[26] Therefore, evidence-based research indicates that carbamide peroxide treatment is an effective and safe option for patients seeking dental whitening.[1] Because charcoal powder requires the act of brushing, this could mislead patients not to brush teeth with fluoride-containing toothpastes. Since the deleterious effect of charcoal powder was observed and further investigations are still necessary to determine the safety of techniques herein investigated, the practical implication of the present research is that the use and indication of charcoal-based products should be discouraged by the clinicians.

CONCLUSION

Within the limitations of this study, the following conclusions could be drawn:

1. The activated charcoal powder did not enhance color change when combined with regular and whitening toothpastes;
2. Low-concentrated carbamide peroxide resulted in greater color change than charcoal powder;
3. The charcoal powder alone increased the enamel surface roughness.

REGULATORY STATEMENT

This research was conducted respecting the rules of the local Ethical and Research Committee and the policies of the University.

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3 CONCLUSÃO

Pode-se concluir que:

a) A escovação com pó de carvão ativado não promoveu alteração de cor significativa previamente à escovação com dentifrícios convencionais ou com propostas clareadoras;
b) Gel de peróxido de carbamida 16% apresentou maior alteração de cor em comparação ao carvão ativado.
c) O pó de carvão ativado sozinho promoveu um aumento de rugosidade superficial do esmalte.

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ANEXOS

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ANEXO 2 - Primeira página do artigo publicado

Received: 8 June 2020 | Revised: 1 August 2020 | Accepted: 9 August 2020
DOI: 10.1111/jerd.12646

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RESEARCH ARTICLE

Effects of activated charcoal powder combined with toothpastes on enamel color change and surface properties

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Abstract

Aim: To evaluate the effects of activated charcoal powder (COAL) combined with regular (RT) or whitening (WT) toothpastes on enamel color and surface in comparison to carbamide peroxide (CP).

Methods: Dental blocks (n = 10/group) were randomly divided into COAL, COAL/RT, COAL/WT, CP, CP/RT, CP/WT, RT, WT, and CONT (without treatment). Simulated toothbrushing and whitening treatments were followed by colorimetric (ΔE_{00}, L^*, a^*, b^*), surface roughness (Ra), and enamel topography assays. ΔE_{00} was submitted to two-way ANOVA and Tukey test. Color coordinates and Ra were tested with three-way repeated measures ANOVA (α = 5%).

Results: COAL exhibited greater ΔE_{00} than CONT (P = .048), but it did not enhance ΔE_{00} promoted by RT or WT (P > .05). COAL alone increased Ra (P < .001) and altered enamel topography. COAL did not increase Ra caused by RT and WT (P > .05). CP exhibited the highest ΔE_{00} (P < .05), but it raised Ra and changed enamel topography to a less extent than COAL.

Conclusion: Even though charcoal powder did not increase enamel Ra when combined with toothpastes, the topography was negatively impacted by COAL. Also, COAL was unable to enhance the color change of RT and WT, or reach the effectiveness of CP.

Clinical Significance: The use of activated charcoal-based product, claimed as a natural whitener, before brushing with toothpastes is not only ineffective to change the color of teeth, but also it might result in alterations on the enamel surface. Whitening with CP, instead, was effective during the same period of treatment, which still represents a more appropriate technique to whiten teeth.

KEYWORDS
carbamide peroxide, charcoal, toothbrushing, toothpastes, whitening

1 | INTRODUCTION

At-home tooth whitening supervised by dentists is a technique widely described in the literature as effective and safe for patients.[1]

Part of this work was presented during poster session of the Annual Meeting of the Brazilian Division of the IADR (SBPqO) in 2019.

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v1.10 Last updated September 2015

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