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Original Article

Surface degradation of composite resins under staining and brushing challenges

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Abstract *Background/purpose:* The primeval evaluation of the properties of composite resins with different compositions that impact clinical behavior is very important in guiding use in clinical settings. The aim of this study was to evaluate color stability (ΔE) and Knoop microhardness (KHN) of composite resins containing different compositions.

Materials and methods: Forty-five disks were made composite resins: Filtek Z350 XT/3MESPE and Beautifill II/SHOFU and divided into 3 subgroups: Control - immersed in artificial saliva; OB – brushing with Oral B 3D White; CT - brushing with Colgate Total 12. The OB and CT groups had the disks brushed daily with 120 cycles after immersion in coffee solution (10 min) for a period of 30 days. ΔE and KHN was obtained at baseline and after the treatments. Data were analyzed ANOVA and Tukey tests ($p < 0.05$).

Results: ANOVA revealed significant differences for ΔE and KHN. ΔE: the highest mean was observed in Beautifil II composite resin group, which differed significantly from Z350 group. For the Beautifil II, the treatments were significantly different from each other. For the Z350, the control group showed significant differences in relation to OB e CT groups. KHN: the highest mean was observed in Z350 group, which differed significantly from Beautifil II group. For Z350, the control group showed significant differences in relation to OB e CT groups. For Beautifil II, the treatments did not differ significantly among themselves.

Conclusion: The composite resin containing fluoride in the composition showed higher color alteration (ΔE) and lower Knoop microhardness (KHN), thus demonstrating that composition is an important factor in the clinical performance of esthetic restorative materials.

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Introduction

The increasing in the esthetic demanding associated with the improvements in the properties and power of bonding to the tooth substrate result in the supremacy of composite resin over other restorative materials.[1] Composite resins aim to mimic the natural features of the tooth, such as the color, translucency, and texture similar to the teeth.

The composition of the composite resin evolves significantly over the years. Alterations in the matrix composition, e.g. type and size of filler particles, exemplifies the constant evolution of this restorative material.[2] Recently, a nanohybrid resin composite with surface pre-reactive glass (S-PRG) ionomer cement promises to release and uptake fluoride in the oral environment.[3,4]

Since the discovery of a therapeutic agent in preventing carious lesion, fluoride is largely used in Dentistry with different vehicles (gel, varnish, dentifrice, and mouthrinse) and incorporated into water and food (salt, milk, juice, soda, and fluoride supplements). The fluoride ability to increase the resistance against demineralization, decrease the microbial activity, and consequently also decrease the occurrence of secondary carious lesions is very known in current Dentistry. Therefore, composite resins containing fluoride, aiming to obtain the advantages promoted by this ion in tooth/restoration interface, would be considered as restorative material of great clinical applicability, due to the capability of fluoride to influence caries risk of tooth structure adjacent to the restoration. Previous studies show similar results regarding mechanical properties of fluoride and conventional composite resin.[5]

However, the drawbacks of composite resin restoration are surface degradation, technique, fracture, and color alteration.[5] These are the main reasons for composite resin replacement,[1] probably because the continuous exposure to saliva and beverages/food stains within oral conditions. Previous studies report that direct esthetic restorative materials present *in vitro* and *in vivo* color instability with staining.[6–8]

At long term, the surface alterations of composite resin may occur according to intrinsic and extrinsic factors which restorations are routinely exposed[6]: insufficient polymerization, water sorption, and staining from diet and hygiene habits of the patient.[8,9] Some studies correlate the size and distribution of filler particles and the matrix composition of composite resin as factors to interfere in color alteration. The discoloration of composite resin might be related to the degree of the water sorption and the hydrophilic property of the resin matrix.[10,11]

Additionally to the effect on composites color, the composition of the food and beverage may degrade the surface of the restorative materials and affect the organic phase of the resin matrix, promoting the disintegration of the disperse phase and altering the surface hardness of composite resin.[12–14] Furthermore, the hygiene habits, e.g. toothbrushing, also influence on the material's longevity. Abrasion may result in alterations in the material surface affecting in the contour, coloration and favoring the plaque retention due to the surface roughness.[15]

The literature affirm that the mechanical properties of the current fluoride composites are similar to those of the conventional composites.[16] On the other hand, new questions arise on the profile of these materials in relation to the daily habits of feeding and hygiene compared with other nanohybrid composites. Thus, this study aimed to evaluate the color alteration (ΔE) and Knoop surface microhardness (KHN) of nanoparticle-reinforced composite resins containing different compositions, after staining induced by coffee solution and abrasion challenge through simulated toothbrushing. The null hypothesis was that no significance differences occur in color and superficial microhardness between the studied composite resins regardless of the surface treatment to which they were submitted.

Materials and methods

Specimen preparation

Forty-five cylindrical specimens were fabricated (shade A2), using a metallic matrix with 2 mm in height and 3 mm in diameter, of each composite resin brand: Filtek Z350 XT (Nanoparticle; 3M/ESPE, St. Paul, MN, USA) and Beautifil II (Nanohybrid; Shofu Inc., Kyoto, Japan). A polyester matrix was placed over the composite resin and pressed with a glass slide to provide a flat surface. The composite resin was inserted in increments of 2-mm and cured on the top surface using LED curing unit (Elipar Freelight 2, 3M/ESPE, St. Paul, MN, USA) at 1200 mW/cm^2 power density, activated for 40 s. After curing, the specimens were stored individually in deionized water for 24 h. Then, the specimens were polished using a sequence of 1200, 2400 and 4000 grit aluminum oxide abrasive disks (Extec, Enfield, CT, USA) in a polishing device (DP-10, Panambra, São Paulo, Brazil). After polishing, all specimens were stored in deionized water at 37 °C for 24 h.

The specimens of each composite resin ($n = 45$) were randomly divided into 3 subgroups ($n = 15$), according to the type of toothpaste used: Control Group - The samples were immersed in artificial saliva, at 37 °C, throughout the period of study; Colgate Total 12 - The samples were submitted daily to brushing cycles with Colgate Total 12 toothpaste (Colgate-Palmolive Industrial Ltda, São Bernardo do Campos, SP, Brazil), after immersion in coffee solution for 10 min, under agitation, for 30 days; Oral B 3D Group - The samples were submitted daily to brushing cycles with Oral B 3D toothpaste (Procter & Gamble Brazil, Louveira, SP, Brazil) after immersion in coffee solution for 10 min, under agitation, for 30 days.

Color measurement (ΔE)

Prior to surface treatments, baseline color of each specimen was assessed under standardized ambient conditions according to the CIE L*a*b* system, using a spectrophotometer (CM2600d, Konica Minolta, Osaka, Japan) and an integrating sphere. The device was adjusted to use the D65 standard light source with 100% UV included or 100% UV excluded and specular reflection included (SCI). The observer angle was set at 2° and the device was adjusted to a small reading area. The color of each specimen was measured three times and averaged. The results of the color measurements were quantified in terms of three coordinate values (L*, a*, b*) as established by the Commission Internationale de l'Eclariage (CIE), which locates the color of an object in a three-dimensional color space. From the color measurement at baseline and those after the surface treatment, the values of the changes of L* (ΔL), a* (Δa), and b* (Δb) were calculated. The total change in color or the variation in perception of color of each specimen was calculated, designated by the abbreviation ΔE*ab. This parameter was calculated according to the following formula: $\Delta E^*ab = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}$.

Knoop microhardness analysis (KHN)

The microhardness measurement was performed with a microhardness tester (FM-700, Future-Tech, Tokyo, Japan), Knoop tip, under 50 g load for 15 s. Three indentations were performed 100, 200 and 300 μm apart from each other, on the surface of the specimens. The means were determined as Knoop Hardness Number (KHN).

Surface treatments: staining in coffee solution and brushing simulation

In each daily cycle, the samples were first immersed in 2 ml of coffee solution, at 37 °C for 10 min, under constant agitation. The coffee solution was prepared with 1 tsp. of soluble coffee (Nescafé Original, Nestlé, Araras, São Paulo, Brazil) dissolved in 50 ml of boiling water. Subsequently, the specimens were subjected to brushing abrasion in an automatic toothbrushing (TB) machine (ODEME Biotechnology, Joaçaba, SC, Brazil), which imparted reciprocating motion to 6 soft bristle toothbrush heads (Sanifill Ultraprofissional, Hypermarcas, São Paulo, SP, Brazil). This apparatus provides linear brushing movements across the specimens at a speed of 120 cycles per min at 37 °C, with a double pass of the brush head over the surface, simulating 3 brushings of 40 cycles per day, which corresponds to 3 daily brushings in oral cavity for one month.[17] The abrasive slurry consisted of toothpaste and artificial saliva, in a ratio of 1:3, by weight. The toothpastes used in this study are specified in Table 1. After brushing, the specimens were kept in deionized water at 37 °C. At the end of this protocol, the specimens were rinsed with deionized water and microhardness and color measurement were performed.

Statistical analysis

Data analysis was carried out using the software program Minitab for Windows (version 16.1, College State, PA, USA), Statistica for Windows (version 9.1, StatSoft Inc., Tulsa, OK, USA) and Statistix 9 (version 9.1, Tallahassee, FL, USA). Statistical analysis was submitted to two-way analysis of variance (ANOVA; composite resin and surface treatments) and Tukey tests, with a significance level set at 5% ($p < 0.05$).

Results

Color measurement (ΔE)

According to ANOVA, the treatment ($F = 72.98$; $p = 0.0001$), composite resin ($F = 49.98$; $p = 0.0001$), and the interaction ($F = 2.92$; $p = 0.0016$) were statistically significant. Table 2 displays the mean values of the color change (ΔE) for the composite resin factor for each treatment. It is observed that the greatest mean values were presented by Beautifil resin group, which differed significantly from the Filtek group. For the Beautifil group, the treatments differed significantly from each other. For the Filtek group, the control group presented significant differences in relation to the treatment groups (Colgate and Oral B). Regarding surface treatments, all groups (Control, Colgate, and Oral B) exhibited significant differences between the studied composite resins (Filtek and Beautifil II).

Knoop microhardness (KHN)

ANOVA showed statistically significant differences for treatment ($F = 21.76$; $p = 0.0001$), composite resin ($F = 38.96$; $p = 0.0001$), and interaction ($F = 6.96$; $p = 0.0016$). Table 3 presents the mean values of the Knoop microhardness (KHN) for the factor composite resin in each treatment. The composite resin Filtek exhibited the greatest mean value, statistically different from that of the resin Beautifil. In relation to surface treatments, the control group and the Oral B group statistically differ between the studied composite resins, while the group Colgate did not statistically differ. It could be observed that the composite resin containing fluoride Beautifil II and the composite resin fluoride-free Filtek reduced the microhardness values after the treatment with the dentifrices compared with the control groups. However, in spite of this reduction, the microhardness values presented by the Filtek composite resin were higher than those of the Beautifil II composite resin.

Discussion

This study evaluated the effect of the immersion into coffee solution associated to simulated toothbrushing on

Table 1 Toothpastes used.

Toothpaste	Compositions
Colgate Total 12 (Colgate-Palmolive Ind.Ltda)	Sodium fluoride (1100 ppm fluoride), water, hydrated sílica, pentasodium triphosphate, PEG-12, tetrapotassium pyrophosphate, sodium lauryl sulphate, polyethylene, cocamidopropyl betaine, sodium saccharin, sodium hydroxide, titanium dioxide.
Oral-B 3D White (Procter&Gamble GmbH)	Sodium fluoride (1450 ppm fluoride), water, hydrated sílica, sorbitol, sodium lauryl sulphate, sodium hydroxide, sodium saccharin

Table 2 Mean values ± standard deviation of ΔE and the results of Tukey tests (5%).

Composite resin	Treatments		
	Control	Colgate	Oral B
Filtek Z350 XT	1.73 ± 1.19 Ab	7.00 ± 2.15 Aa	7.27 ± 2.55 Aa
Beautifil II	4.50 ± 2.96 Ba	9.29 ± 2.33 Bb	12.11 ± 1.56 Bc

Different letters show statistically significant differences ($p < 0.05$): uppercase letters refer to columns; lowercase letters refer to lines.

Table 3 Mean ± standard deviation of KHN values and the results of Tukey tests (5%).

Composite resin	Treatments		
	Control	Colgate	Oral B
Filtek Z350 XT	27.16 ± 10.85 Ab	8.23 ± 6.27 Aa	15.48 ± 9.66 Aa
Beautifil II	11.21 ± 5.17 Ba	6.25 ± 5.08 Aa	4.35 ± 4.69 Ba

Different letters show statistically significant differences ($p < 0.05$): uppercase letters refer to columns; lowercase letters refer to lines.

changing the color and surface microhardness of nanoparticle-reinforced composite resins. The surface treatments significantly reduced the properties of the studied composite resins, showing differences in color and superficial microhardness. Thus, the null hypothesis was rejected.

The samples not subjected to the surface treatments were immersed into artificial saliva, as a positive control. According to this present study, the artificial saliva slightly altered the color of the resin Filtek Z350 XT ($\Delta E = 1.73 \pm 1.19$), a result similar to that of previous studies.[1,18] However, when compared to the Beautifil II samples, it was observed that artificial saliva had a significant color change ($\Delta E = 4.50 \pm 2.96$), since the ΔE value equal or below to 3.3 is considered clinically acceptable in Dentistry.[19,20] This color change could be related to the long immersion period to which the samples were immersed. According to Domingos et al.[1] the immersion into artificial saliva significantly influenced the color stability of composite resin after 30 days, but not at the initial period. Also, the artificial saliva components and the water sorption by the resin matrix could result in plasticization, softening, and hydrolysis of the material, thus promoting greater susceptibility to color change.[1]

On the other hand, coffee was the substance used to stain the material because of its greater consumption by the population and potential to stain both the tooth and the composites.[9] Furthermore, the coffee can significantly alter certain resin properties at high temperatures.[10,21,22] Routinely, food and beverage have short contact with tooth and restorative material surfaces before either the saliva wash-out or toothbrushing. Notwithstanding, as far as we are concerned, this is the first study that immersed the samples for a shorter period to mimic daily habits, that is, each sample was immersed into the coffee solution for 10 min per day, followed by simulated toothbrushing, and kept in distilled water to copy the neutralizing effect of the saliva, but without the saliva components.[23]

The results of the color change showed significant differences among the evaluated treatments and between the composites. Both composite resins increased the ΔE values after the surface treatment, regardless of the toothpaste used. The rationale behind this color change could be the daily immersion into coffee solution, similarly to the studies of Patel et al.[22] and Ertas et al.,[10] in which the coffee solution had the greatest ΔE values, mostly influencing on the resin color.

Also, the simulated toothbrushing could have favored some changes in the composite resin surface. The *in vitro* simulated toothbrushing is a parameter to evaluate the restorative material's ability to maintain the smoothness, brightness, and to avoid staining.[24] The greater the number of toothbrushing cycles and period, the highest is the degradation of the composite resin with higher surface roughness and lower brightness.[24] Similarly, in this present study, the simulated toothbrushing significantly altered the color of the evaluated composite resins. The increase of the roughness due to the gradual removal of the filler particles, during the toothbrushing process, may account for this result,[24,25] thus favoring the staining of the composite surface. This effect in color change differs depending on the composite resin composition: particle size, resin matrix composition, and conversion after polymerization.[24] This would explain the difference observed by this present study, in which the nanohybrid resin Beautifil II had the highest ΔE variation than the nanoparticulate resin Filtek Z350 XT, in agreement with other studies.[26]

The abrasiveness of the dentifrices would have affect the surfaces of the studied composites. The greater the dentifrice abrasiveness, the greater is the surface roughness and the material's weariness.[17,27] This present study employed two dentifrices with different compositions. The nanoparticulate composite without fluoride did not show statistically significant differences in color alteration after the treatment with both toothpastes. However, the nanohybrid composite with fluoride exhibited a higher ΔE variation after the treatment with dentifrice Oral-B than with the dentifrice Colgate Total 12. Probably, the different dentifrice compositions and the different composite resin compositions explain this difference. The different methodologies regarding the dentifrice slurry, toothbrushing type, hardness and rigidity of the bristles, and number of the toothbrushing cycles[25] make difficult to compare our results with those of the literature.

The surface hardness is the mechanical property related to the material's resistance to wear. Generally, the hardness alterations of the composite resin occurred within the first seven days after the exposure to chemical solutions.[28] In this study, the microhardness was evaluated just after the polymerization and after the surface treatments. In this present study, both composites statistically decreased the microhardness after the immersion into artificial saliva without simulated toothbrushing. The chemical degradation on the composite surfaces related to the resin matrix would explain the differences in the microhardness values.[29] Still, the hydrolytic instability of the organic matrix, considered the most fragile point of the composites, could have accounted for the changes due to the high sensitivity of the material to water absorption.[23]

We found differences in the microhardness between the two composites after immersion into artificial saliva, related to the composition and content of the particles. Despite of the similar matrix compositions, the size and amount of the filler particles are different. The resin Filtek Z350 XT has nanoparticles of approximately 0.6 μm and smaller filler content (78.5%) than the resin Beautifil II (nanohybrid particles of 0.8 μm and 83% of filler content). Probably, the smaller filler content of the resin Filtek Z350 XT favored the significant wear of the surface hardness after the immersion in artificial saliva for 30 consecutive days. However, the KHN values of the resin Filtek Z350 XT were higher than those of the tested composite resin containing fluoride.

After the surface treatments, the resin Beautifil II exhibited KHN values smaller than that of resin Filtek Z350 XT, at all time periods. Beautifil II is composed by S-PRG ionomer particles with relatively greater size (0.8 μm), resulting in a rougher surface profile than Filtek Z350 XT (0.6 μm). Moreover, the greater filler content favors smoother surfaces,[30] and accounts for greater ease in surface weariness. In other words, the differences in the mechanical properties between the nanoparticulate and nanohybrid composite with fluoride would be explained by the different composition and hybrid nature of the latter. The surface microhardness exhibited the same profile after toothbrushing. No statistically significant differences occurred after the use of both dentifrices, for both studied composite resins Filtek Z350 XT and Beautifil II. Due to the samples were daily immersed into the coffee solution followed by the simulated toothbrushing, the dentifrice type did not alter the surface weariness.

Although some studies verify separately the effect of liquid immersion to simulate feeding and the effect of toothbrushing, the evaluation of this association is clinically relevant. Clinically, the consumption of food or beverages occur prior to the oral hygiene habits. The long immersion in coffee solution at high temperatures may reduce the Knoop microhardness.[21] This immersion may alter the resin matrix, causing the exposure to filler particles and then, the toothbrushing may alter the mechanical properties of the resin surfaces.[31] The results of this present study showed difference in the resin profile after the treatment, with smaller KHN values for the fluoride composite resin. Thus, we hypothesize that the superficial microhardness value after toothbrushing varied according to the number, size and type of the filler particles, which are determining factors for a satisfactory clinical outcome.

Despite of the fluoride presence, the composite resin Beautifill II showed unsatisfactory color and hardness outcomes after daily food and hygiene habits compared with the composite resin Filtek 350 XT without fluoride. Within the limitation of this study, the fluoride release and uptake was not evaluated. Further studies are necessary to verify this property of fluoride release and uptake claimed by the manufacturer, as well as the effects of this restorative material with fluoride in patients with high caries prevalence.

Based on the results of this study, it can be concluded that the fluoride-containing composite resin presented higher color changes and microhardness reduction than the non-fluoride composite resin; after daily treatments with immersion in coffee solution associated with brushing cycles. Thus, demonstrating that composition is an important factor in the clinical performance of esthetic restorative materials.

Conflicts of interest

None.

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