Bleaching Agent Action on Color Stability, Surface Roughness and Microhardness of Composites Submitted to Accelerated Artificial Aging

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ABSTRACT

Objectives: The purpose of this study was to evaluate the bleaching agent action on color stability, surface roughness and microhardness of composites (Charisma, Filtek Supreme and Heliomolar - A2) submitted to accelerated artificial aging (AAA).

Methods: A Teflon matrix (12 x 2 mm) was used to fabricate 18 specimens (n=6) which, after polishing (Sof-Lex), were submitted to initial color reading (Δ E), Knoop microhardness (KHN) (50 g/15 s load) and roughness (R_a) (cut-off 0.25 mm) tests. Afterwards, the samples were submitted to AAA for 384 hours and new color, microhardness and roughness readings were performed. After this, the samples were submitted to daily application (4 weeks) of 16% Carbamide Peroxide (NiteWhite ACP) for 8 hours and kept in artificial saliva for 16 hours. New color, microhardness and roughness readings were made at the end of the cycle, and 15 days after bleaching.

Results: Comparison of the ΔE means (2-way ANOVA, Bonferroni, P<.05) indicated clinically unacceptable color alteration for all composites after AAA, but without significant difference. Statistically significant increase in the KHN values after AAA was observed, but without significant alterations 15 days after bleaching. For R_a there was no statistically significant difference after AAA and 15 days after bleaching.

Conclusions: The alterations promoted by the bleaching agent and AAA are material dependent. (Eur J Dent 2011;5:143-149)

Key words: Bleaching agent; Accelerated aging; Color stability; Microhardness; Surface roughness.

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INTRODUCTION

Color alteration of composites can be caused by intrinsic or extrinsic factors. Intrinsic factors are related to physical chemical reactions in the deepest portions of the restoration, in addition to changes in temperature and humidity, and extrinsic factors are related to adsorption or absorption of staining substances.¹

Bleaching agents act efficiently in removing intrinsic stains from the tooth and can be used under professional supervision in a dental office (professional bleaching); or without supervision (home bleaching), performed by the patient. This procedure involves the use of carbamide peroxide solutions at concentrations between 10% and 16%, and generally provides satisfactory results after a period of 2 to 6 weeks.² These solutions are very unstable, which, in contact with saliva, immediately dissociate into 3% to 5% hydrogen peroxide and 7% to 19% urea. After this, the hydrogen peroxide is degraded into oxygen and water, while urea is degraded into ammonia and carbon dioxide.³⁻⁵ The hydroxyl radicals formed combine with the intrinsic and extrinsic stains present in the teeth and remove them through an oxidation reaction.^{1,6}

Frequently, teeth restored with esthetic materials are submitted to bleaching, which may cause an alteration in the restorative material color, so that it no longer mimics the color of the tooth, making it one of the most frequent reasons for replacing restorations after bleaching.⁷

Several studies have tested the effect of bleaching agents on the properties of dental materials. As regards to microhardness, studies have reported an increase,⁸ reduction⁹ or even no alteration¹⁰⁻¹⁴ in the surface hardness of composites after the carbamide peroxide gel application.

Regarding the surface texture of the restorative material, some studies^{9,15} have observed small alterations in the surface of composites after daily immersion in bleaching gel, without statistically significant results among control and bleached groups. Nevertheless, Wattanapayungkul et al¹⁶ demonstrated the presence of several gaps in the restorative material surface after home bleaching, which may favor esthetic material staining after bleaching.⁷

Although bleaching agents are frequently used, there isn't a consensus about the effects of these products on restorative materials, particularly those that have already been in function for some time.¹⁰⁻¹⁶ Therefore, the aim of this study was to evaluate the effect of home bleaching agent on the color stability, surface roughness and microhardness of direct composites submitted to the Accelerated Artificial Aging procedure (AAA). Two hypotheses were tested in this study: the bleaching agent would be effective regarding bleaching the aged specimens, and its use would not alter the surface roughness and microhardness of the composites.

MATERIALS AND METHODS Specimen preparation

Three composite resins with different filler size were used in the current study (Table 1). A Teflon matrix (12 x 2 mm) was used to fabricate 18 specimens (n=6) according to the incremental technique. Light activation was performed with a light emitting diode device (Flash Lite 1401, Discus Dental, Culver City, Ca, USA, 465/475 nm, 1400 mW/cm²) for 40 seconds, in accordance with the manufacturer's recommendations.

Once removed from the matrix, the specimens were polished with abrasive paper disks in a descending order of granulation (Sof-Lex, 3M do Brasil, Sumaré, SP, Brazil). Next, the specimen's thickness was checked with a digital pachymeter (Digimess, São Paulo, SP, Brazil). After this, color, surface roughness and initial microhardness readouts were performed.

Assessment procedures

To color analysis, a Spectrophotometer (PCB 6807 BYK GARDNER, Geretsried, Germany) was used. The standard observation simulated by the Spectrophotometric colorimeter was in accordance with the CIE L*a*b* system, described by Pires-de-Souza et al,¹⁷ and the readings were performed against a white standard background (White Standard Sphere for 45°, 0° Reflectance and Color Gardner Laboratory Inc. Bethesda, Geretsried, Germany). Knoop microhardness of the specimens (KHN) was determined using a microhardness tester (HMV 2000, Shimadzu, Kyoto, Japan) in three different points, with a 50 g/15 s load. Surface roughness (R) was measured with a Rugosimeter Surfcorder SE 1700, cut-off - 0.25 mm (Kosakalab, Tokyo, Japan). The rugosimeter needle was positioned over each test specimen, performing three readings in different locations of the sample surface. After the three readings, the mean surface roughness values were obtained.

Aging procedure

After initial color, surface roughness and microhardness analyses, the specimens were submitted to Accelerated Artificial Aging (AAA) using an Accelerated Aging System for non-metallic objects C-UV (Comexim Matérias Primas Ltda., São Paulo, Brazil), under the action of UV light and condensation, which were activated in separate cycles repeated successively and automatically. The AAA is achieved in a laboratory environment that indicates the behavior of materials under certain conditions and it is widely used for development and control of different materials properties.¹⁸ This system is composed of a network of eight fluorescent light tubes of 40 watts with emission concentrated in the ultraviolet B region; with radiation concentrated at 280/32 nm and the exposure temperature is automatically controlled according to the programs established by the UV/ condensation cycles. The working program was standardized for 4 hours of exposure to UV-B at 50°C and 4 hours of condensation at 50°C. The distance from the light source was 50 mm and the maximum aging time 384 hours¹⁷ corresponding to a year of clinical use,¹⁹ in agreement with the guidelines recommended by ASTM.¹⁸

After AAA, new color, surface roughness and microhardness readings were performed, following the same methodology. After this, the specimens were submitted to daily application

Table 1. Tested composites.

(4 weeks) of 16% Carbamide Peroxide (NiteWhite ACP Discus Dental, Culver City, CA, USA, pH 5.9 to 6.6) for 8 hours in oven at 37°C. After this period, the specimens were washed under running water and kept in artificial saliva at 37°C for 16 hours. At the end of the cycle, new color, microhardness and roughness readings were performed, and again, 15 days after bleaching. In this period, the specimens were kept in artificial saliva at 37°C, changed every day.

The color stability (ΔE) of the materials was calculated using the formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{\frac{1}{2}}$ where ΔE represents the color change in all dimensions (L*a*b*) and ΔL^* , $\Delta a^* \Delta b^*$ represent color changes along the individual axes.²⁰ Values of $\Delta E \ge 3.3$ were considered clinically unacceptable.²¹

The initial and after-treatment values of ΔE , surface roughness and microhardness were statistically analyzed (2-way ANOVA, Bonferroni's test, P<.05).

RESULTS Color spectrophotometry

The comparisons between the intervals of aging and baseline, bleaching and baseline; and 15 days before bleaching and baseline are shown in Table 2. It was verified that Charisma and Heliomolar presented high Δ E values after AAA, an alteration considered clinically unacceptable,²¹ with statistically similar results between them (P>.05). After AAA, the composite Filtek Supreme presented color alteration with Δ E considered within the acceptable limit,²¹ with statistically significant

Composite	Monomers	Loads		Manufacturer
		Size	% by volume	
Charisma	Bis-GMA, TEGDMA	2 - 0.04 µm	61%	Heraus Kulzer, Hanau, Germany
Filltek Supreme Z350	Bis-GMA, Bis-EMA, UDMA, TEGDMA	0.6 - 1.4 µm (Agglomerated), 5 – 20 nm (Nanoagglomerated)	59.5%	3M ESPE, Irvine, CA, USA
Heliomolar	Bis-GMA, UDMA	0.04 - 0.2 μm	46%	Ivoclar/Vivadent AG, Schaan, Liechtenstein

Bis-GMA Bisphenol glycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: Urethane Dimethacrylate; Bis-EMA: Bisphenol A diethoxy methacrylate.

Table 2. Means (standard deviation) of ∆E of aged samples (2-way ANOVA, Bonferroni, P<.05).

	Post-aging	Post-bleaching	15 days after bleaching
Charisma	5.83 (1.7) ^{a,A}	3.91 (0.8) ^{b,A}	3.79 (0.9) ^{b,A}
Filltek Supreme Z350	2.04 (0.5) ^{a,B}	5.17 (0.8) ^{a,A}	3.54 (0.8) ^{a,A}
Heliomolar	4.48 (3.1) ^{a,A}	5.17 (0.8) ^{a,A}	5.18 (0.8) ^{a,A}

*Different letters, lower case letters on the line and capital letters in the column indicate statistically significant difference (P<.05).

difference in comparison with the other materials (P<.05). After bleaching, Charisma presented lower ΔE than after aging, a statistically significant result (P<.05). For the other materials, there was an increase in ΔE , without statistically significant difference (P>.05). When comparing the performance of composites after bleaching, there were no statistically significant differences among them (P>.05). Fifteen days after bleaching, the ΔE values were very similar in comparison with the postbleaching values, without statistically significant differences among the materials, in comparison with the previous treatment (P>.05).

Surface roughness

The results after AAA (Table 3) indicated that there was no statistically significant difference (P>.05) in roughness for any of the tested materials. When the materials were compared among them, there was statistically significant difference (P<.05) only for Heliomolar. After bleaching, Filtek Supreme and Heliomolar presented an increase in surface roughness values, with statistically significant difference (P>.05) in comparison to the post AAA values. Fifteen days after bleaching, there was not statistically significant difference (P>.05) in comparison with the post bleaching situation.

Microhardness

As regards the microhardness values (Table 4), a large increase was verified after AAA, results differing statistically from the initial values (P<.05). After bleaching, there was an increase in the microhardness of Heliomolar (P<.05). For the other composites, there was a decrease, with statistically significant results (P>.05). After 15 days, there were no significant alterations (P>.05).

DISCUSSION

There is a consensus among researchers that direct composite resin restorations undergo color alteration with the passage of time,²¹⁻²⁸ and that one of the greatest challenges in modern dentistry is to find a material that has a color stability similar to that of the dental structure; and that this stability can be maintained in the oral environment as the years pass.²⁹⁻³⁰

As regards the color stability, this study tested the hypothesis that the bleaching agent would promote bleaching of the studied composites. The results demonstrated that the color of composites could be significantly altered by AAA³¹ and bleaching, and this alteration is material dependent, results that are similar to other studies.^{7,32} This susceptibility to color alteration can be attributed to the composition of the resin matrix and the type and volume of load particles of the composite.⁷

When bleached, the aged specimens did not presented significant alteration for ΔE (P>.05), with the exception of the composite Charisma, and the same behavior was maintained 15 days after bleaching. This fact demonstrated that the action of the bleaching agent was more effective on the aged specimens, allowing one to agree with the tested hypothesis. After being bleached, the specimens remained without significant color alteration (P>.05). This may have occurred due to the stability of bleaching agents and their prolonged action.³¹

Differences in the chemical structure of composites, such as the type of oligomers or monomers used; concentration/type of activators, initiators, inhibitors; oxidation of carbon doublebonds; size/type of load particles and the load

Table 3. Means (standard deviation) of surface roughness (Ra) of aged samples (2-way ANOVA, Bonferroni, P<.05).

	Initial	Post-aging	Post-bleaching	15 days after bleaching
Charisma	0.18 (0.1) ^{a,A}	0.32 (0.1) ^{ab,AB}	0.35 (0.1) ^{b,A}	0.41 (0.2) ^{b,A}
Filltek Supreme Z350	0.12 (0.1) a,A	0.12 (0.0) ^{a,A}	0.53 (0.1) ^{b,A}	0.51 (0.1) ^{b,A}
Heliomolar	0.27 (0.2) ^{a,A}	0.42 (0.1) ^{a,B}	1.35 (0.4) ^{b,B}	1.33 (0.2) ^{b,B}

*Different letters, lower case letters on the line and capital letters in the column indicate statistically significant difference (P<.05).

Table 4. Means (standard deviation) of microhardness (KHN) of aged samples (2-way ANOVA, Bonferroni, P<.05).

	Initial	Post-aging	Post-bleaching	15 days after bleaching
Charisma	74.9 (8.2) ^{a,A}	92.8 (2.3) ^{b,A}	89.2 (6.0) ^{ab,A}	86.2 (3.4) ^{ab,A}
Filltek Supreme 350	86.2 (8.2) ^{a,A}	134.8 (6.8) ^{b,B}	115.3 (8.5) ^{с,В}	114.5 (8.1) ^{с,В}
Heliomolar	43.0 (6.1) ^{a,B}	61.2 (10.7) ^{b,C}	107.0 (23.1) ^{с,В}	93.6 (15.4) ^{c,A}

*Different letters, lower case letters on the line and capital letters in the column indicate statistically significant difference (P<.05).

particle/resinous matrix bonding system may interfere in its properties.³³ In that way, the significant color alteration of the Charisma composite can be related to the largest size and the largest concentration of the load particles in its composition. Smaller particles, with smaller load concentration in a composite, promote a larger light reflection among the particles.³⁴ The size of the inorganic particles is also related to the color alteration, once composites formulated with large particles are more susceptible to water sorption and color alteration. Several studies have demonstrated that the composites allow solvents to penetrate into the resinous matrix or in the interface matrix/particles. When hydrolytic degradation of the polymeric network occurs, this interface can be altered and, consequently, to modify the light dispersal.^{20,35}

According to Ferracane,³⁵ as greater the volume of particles in the composite formulation, lower the degree of conversion presented. Consequently, the polymer formed would have a larger quantity of remaining double-bonds and lower quantity of formed bonds. Therefore, this composite will be more predisposed to the action of the solvent (water), as there will be a greater free volume for water action, which will penetrate into the resinous matrix, causing "swelling" or relaxation of these bonds, in an effect known as plasticization. The solvent inside may cause resinous matrix and particle/matrix interface deterioration.³⁵ Plasticization will promote, besides the decrease of the composite hardness, greater color alteration, due to the presence of water inside the resinous matrix after AAA procedure. It was what happened with the Charisma composite in this study, which presented greater color alteration and smaller microhardness after bleaching, result that remained after 15 days.

In the present study, the clinical acceptance value of ΔE <3.3 for esthetic restorative materials was used; a value adopted by other authors,³⁶ and one exceeded by all the materials, with the exception of Filtek Supreme.

The other hypothesis tested in the study was that the use of the bleaching agent would not alter the surface roughness or microhardness of the composites. With regards to surface roughness, bleaching caused an increase in roughness, with statistically significant results in comparison with the initial situation (P<.05). Nevertheless, the changes were small and material dependent, and the results are in agreement with other studies.^{5,16} Bailey & Swift⁹ suggested that the increase in roughness could be as a result of loss of resinous matrix rather than load particles.

The bleaching agent used in the study was carbamide peroxide-based, which breaks down into urea and hydrogen peroxide. This in turn forms free radicals that can eventually form water and accelerate the hydrolytic degradation of the composite, resulting^{35,37} in bonding failure between the resinous matrix and load particles, increasing the surface roughness of the composite.¹⁶

Zanin et al³⁸ verified the color stability and surface roughness of indirect composite resins submitted to AAA for 384 hours. The authors verified that all the studied composites presented both color alteration and increase in surface roughness, and concluded that these properties are closely related. However, in the present study, it could be observed that the composites evaluated did not present statistically significant increase (P>.05) in the surface roughness values after AAA. The relationship between surface roughness and color stability described by Zanin et al,³⁸ did not occur in this case, since the composites Charisma and Heliomolar presented high ΔE values,²¹ but low levels of surface roughness.

Based on these findings, it could be concluded that there was no direct relationship between surface roughness and color stability of the composites. Both AAA and the bleaching agent were capable of producing rougher surfaces, which would cause a more significant color alteration. It could be observed that the color alteration that occurred in all of the composites could be more related to the intrinsic alterations that occurred within the specimens, rather than on their surfaces.¹¹

With regards to microhardness, there was a significant increase (P<.05) after AAA, result of a process of post-polymerization caused by the action of temperature (50°C) and water condensation.^{1,39,40} After bleaching, there was a significant reduction (P<.05) in microhardness for Filtek Supreme, which was expected, since this composite presents a high concentration of resinous matrix to be oxidized by the hydrogen peroxide.^{9,10} However, there are discrepancies between these results and those of other studies, due to the diversity of

studied materials; since some are more susceptible to alterations than others.^{41,42} Moreover, the effect of bleaching may be different, as one of the limitations of the study was that the bleaching agent was not diluted in saliva, which could have diminished its action. Therefore, the second hypothesis of the study could not be accepted, as the bleaching agent produced surface alterations in the studied composites.

CONCLUSIONS

From the results found and considering the limitations of this study, it was concluded that:

• AAA promoted color alterations considered clinically unacceptable for Charisma and Heliomolar and bleaching promoted significant color alteration only for Charisma.

• The home bleaching agent produced an increase in the surface roughness values of the studied composites, an alteration that remained 15 days after bleaching.

• There was an increase in the microhardness values of the composites after AAA. After bleaching there was a significant reduction (P<.05) for the nanoparticle composite. Fifteen days after bleaching, there was no significant alteration in microhardness (P>.05).

• Composite alterations promoted by the bleaching agent are material dependent.

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