

Effect of hydrogen peroxide topical application on the enamel and composite resin surfaces and interface

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ABSTRACT

Objectives: The objective of the present study was to analyze the superficial roughness and the interface between enamel and composite resin restorations after dental bleaching procedure.

Materials and Methods: Black's class V cavities were made and restored with composite resin, and the whole set, enamel–restorative material, was treated with 35% hydrogen peroxide. Seven procedures of 30 min each were performed. A profilometric assessment was carried out before and after the treatment of each sample, and roughness scores were obtained. Treated and untreated samples were analyzed under scanning electronic microscope and images of their surface were obtained.

Results and Conclusion: The treatment with 35% hydrogen peroxide caused no alteration in the interface between enamel and composite resin, Tetric Ceram, fillings and the topical application of 35% hydrogen peroxide on enamel and composite resin, Tetric Ceram, caused an alteration of their surface topography, featuring a predominance of depressions after the bleaching treatment.

Key words: Composite resin, dental material, hydrogen peroxide

Dental bleaching is a conservative alternative to restore the esthetics of either stained or darkened teeth. Although exogenous bleaching has been long used and widely accepted as a clinical procedure, little is understood about its interaction with hard and soft tissues of the oral cavity, besides the possible consequences of these reactions in the long term.^[1] Many authors have demonstrated alterations of the enamel after topical application of bleaching agents, presenting as major consequences are: ions release, increased superficial roughness, stronger bacterial attachment, hardness alteration, color alteration, and adhesion to resinous materials.^[2-10]

Several studies report alterations of composite resin restorations after contact with bleaching agents. These restorations may show an increase in superficial roughness, color alteration, presence of clefts, microhardness alteration, and as a consequence, an increase in marginal microleakage.^[10-13]

When a bleaching treatment is performed on a restored tooth, alterations may occur in both the enamel and restorative material, although the restoration would not necessarily need to be replaced after bleaching. Thus, the analysis of the surface and the interface between the

enamel and restorative material after dental bleaching is fundamental, considering that the quality of this interface is of great relevance to the longevity of the restorations.

Due to the divergence among the results shown in dental literature, the objective of this study was to analyze the superficial roughness as well as the interface between the enamel and composite resin restorations after 35% hydrogen peroxide application, through bidimensional profilometry and scanning electronic microscopy (SEM).

MATERIALS AND METHODS

Six human maxillary premolar teeth were used in this study, newly extracted under orthodontic indication, which were stored in artificial saliva solution at $37 \pm 2^\circ\text{C}$. The use of these teeth were authorized by the ethics and research committee of Federal University of Minas Gerais. The roots of these teeth were amputated 2 mm from the cemento-enamel junction using a carborundum disc. The coronary remnant was polished with pumice and Robinson bristle brush (KG Sorensen Ind. Com., Brazil) in a contra-angle (Intramatic - Kavo, Brazil).

Standardized cavities were made in the center of the teeth buccal surface, measuring 2 mm in diameter and 2 mm in depth. Cylindrical diamond burs #2294 were used (KG Sorensen Ind. Com., Brazil) as recommended by Shinohara

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et al.^[14] This diamond bur features a particular penetration cutoff, therefore providing standardized cavities.

The cavities were then restored with light-cured hybrid composite resin (Tetric Ceram, Vivadent Ets., Liechtenstein). Before the placement of the filling material, the cavity was conditioned with 37% orthophosphoric acid (Vivadent Ets., Liechtenstein) for 30 sec and washed with tap water for 30 sec. Excess water was removed using absorbent paper and a single component adhesive system with ethanol-based solvent was applied (Excite, Vivadent Ets., Liechtenstein). After the adhesive application, a mild air jet was blown from a distance of 20 cm in order to accelerate the solvent evaporation. The adhesive system was light cured for 20 sec using a halogen light-curing unit DCL 401 (Kerr Dental, CA, USA) with luminous intensity higher than 400 mW/cm². Before each light-curing cycle, the appliance luminous intensity was measured, using a radiometer (Model 100 cure radiometer, Demetron Research Corporation). The composite resin was laid in the cavity in two increments. The first increment covered the pulpal and occlusal walls of the cavity, while the second increment covered the rest of the cavity. Each increment was light cured for 20 sec. Soon after the restorations were placed, the samples were immersed in artificial saliva solution, pH 7.2, and stored in biological kiln at 37 ± 2°C. Twenty-one days after their fabrication, the restorations were polished manually with sandpapers of granulation 400, 600, 800, and 1000, and, which were always used with tap water between them and the restoration surface. The final polishing was performed with a felt disc soaked in 3-mm alumina. The use of a sandpaper of granulation 400 was necessary to plane the surface to be analyzed through the profilometer, since premolar teeth surfaces feature a slight curvature. At the end of each polishing sequence, the samples were visualized under a stereomicroscope (magnification x100), so as to assure a polishing uniformity among them. After being polished, the samples were immersed again in artificial saliva solution and stored in biological kiln at 37 ± 2°C.

A profilometric analysis of five samples was carried out soon after all the restorations were polished, with the use of a profilometer, Rank Taylor-Hobson model from Talysurf Series. The samples were then washed in distilled water, dried with air jet, and finally set at the profilometer plate. Four regions of each sample were analyzed, and the measurements were initiated on the buccal surface occlusal portion, went through the interface between enamel-restorative material, then throughout the whole restorative material, and ended on the cervical portion enamel of the same surface. The scanning probe went through the crown buccal surface toward its long axis. A 0.10-mm-roughness cutoff and a 0.50-mm-waviness cutoff were used. The data provided by topography, enamel roughness patterns, and restorative materials were obtained and analyzed by the software Talymap Universal, version 3.0 (Taylor Hobson).

The treatment with 35% hydrogen peroxide (Whiteness HP, FGM Ind. Com., Joinville, Brazil) was performed in the five samples submitted to profilometric analysis. Enamel and restorations were previously dried with air jet, so that the treatment could be performed. The treatment consisted application of the bleaching agent, using a dropper, onto the whole buccal surface of the crowns for 30 min. After this period, the samples were washed in tap water for 60 sec and returned to the artificial saliva solution at 37 ± 2°C. This procedure was carried out seven times, with an interval of 24 hours between the sessions. The samples were never touched by any material, therefore, any alteration on their surface must have been only due to the action of the bleaching agent.

Soon after the treatment period, the samples were stored in artificial saliva solution for 24 hours at 37 ± 2°C, and then submitted to a new profilometric analysis, with the same criteria used for the former analysis.

Two samples were analyzed under SEM: one was treated [Figures 1, 3 and 5] and the other one was untreated [Figures 2, 4 and 6]. The experimental sample was submitted to a profilometric analysis both before and after the treatment, while the control sample was not. The samples were metallized with a gold coating in a metallizing appliance, Desk II Cold Sputter / Etch Unit (Denton Vacuum, LLC), and analyzed under SEM JSM5310 (JEOL).

RESULTS

Roughness average (Ra) and skew average (Rsk) of each sample were obtained and the data were statistically analyzed. In order to perform the statistical analyses, softwares SPSS 9.0.01 and Minitab 13.02 were used. The data normality was tested by means of Anderson–Darling’s normality test at the significance level of 5% ($\alpha = 0.05$). Likewise, to test the existence of difference between the averages, paired *t* test was used to compare averages at a significance level of 5% ($\alpha = 0.05$).

After paired *t* test was applied to the data of enamel Ra to compare the averages, [Table 1], [Figure 7], it was possible to observe that, although the average had decreased after the treatment, ($X_{\text{before}} - X_{\text{after}} = 0.014 \pm 0.017$ mm), there was no statistically significant difference between the Ra before and after the treatment ($P = 0.135$).

Table 1: Results of paired *t* test

	Variable	Average	Standard deviation	<i>P</i>
Ra (enamel)	$X_{\text{before}} - X_{\text{after}}$	0.014	0.017	0.135
Rsk (enamel)	$X_{\text{before}} - X_{\text{after}}$	1.021	0.356	0.003
Ra (resin)	$X_{\text{before}} - X_{\text{after}}$	0.036	0.077	0.352
Rsk (resin)	$X_{\text{before}} - X_{\text{after}}$	3.918	1.189	0.002

For comparisons between the averages of roughness average (Ra) and skew average (Rsk) before and after the treatment

Paired *t* test was then applied to the data of enamel Rsk to compare the averages, [Table 1], [Figure 8], and it was possible to observe a statistically significant difference between the Rsk before and after the treatment ($P = 0.003$). As this difference was positive, ($X_{\text{before}} - X_{\text{after}} = 1.021 \pm 0.356 \text{ mm}$), it

is possible to state that the Rsk is lower after the treatment.

When paired *t* test was applied to the resin Ra data in order to compare the averages, it was possible to observe that, although the Ra had decreased after the treatment,

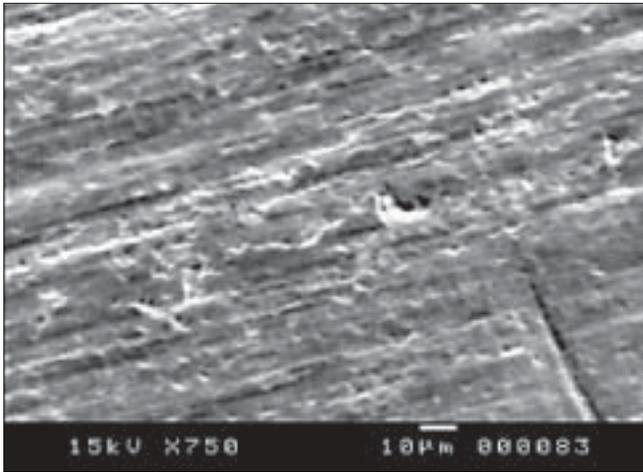


Figure 1: Control enamel

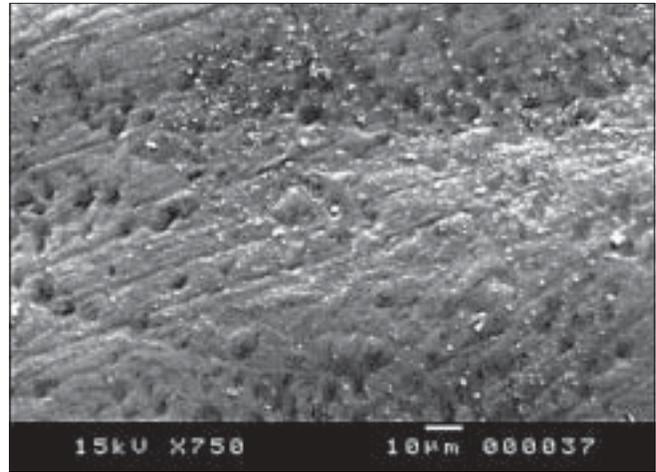


Figure 2: Experimental enamel

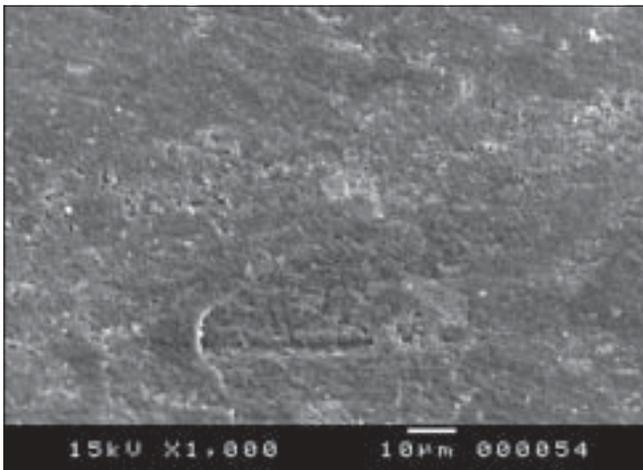


Figure 3: Control restoration

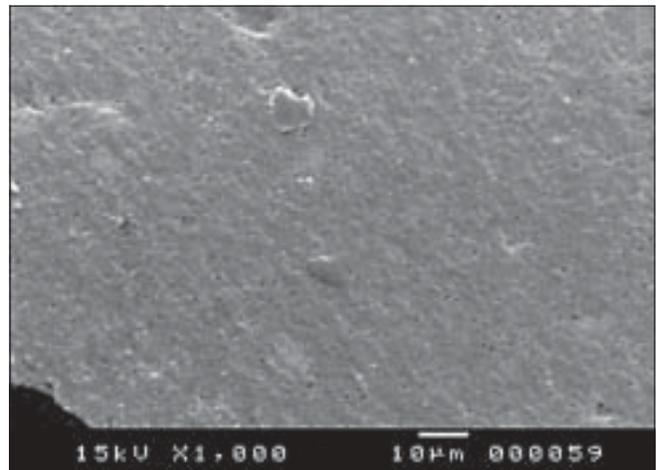


Figure 4: Experimental restoration

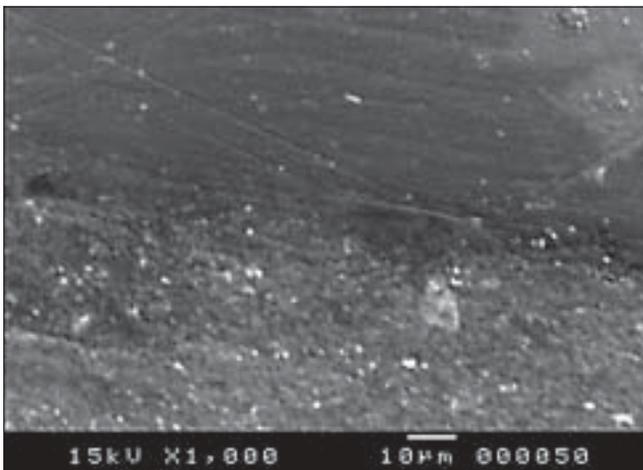


Figure 5: Control interface

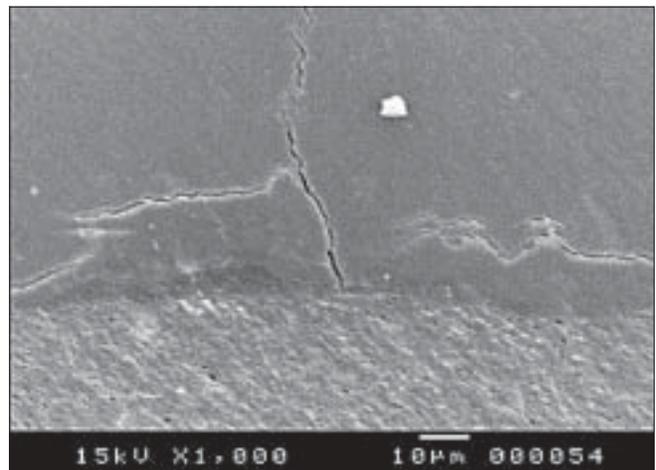


Figure 6: Experimental interface

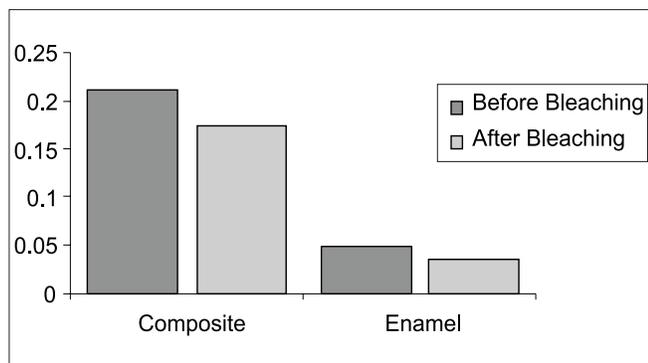


Figure 7: Comparison of mean roughness (Ra) of the enamel before and after the treatment

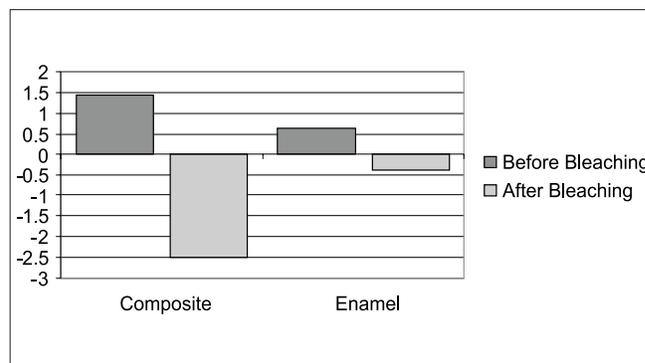


Figure 8: Comparison of mean skew average (Rsk) of the enamel before and after the treatment

($X_{\text{before}} - X_{\text{after}} = 0.036 \pm 0.077$ mm), there is no statistically significant difference between the Ra before and after the treatment ($P = 0.352$).

The application of paired *t* test to compare the averages of the resin Rsk data showed statistically significant difference between the Rsk before and after the treatment ($P = 0.002$). As this difference was positive, ($X_{\text{before}} - X_{\text{after}} = 3.918 \pm 1.189$ mm), it is possible to state that the Rsk is lower after the treatment.

The photomicrographs and the profilometric images of the restorations showed that there were no alterations of the interface between enamel–restorative material after the bleaching treatment. On surface treatment with hydrogen peroxide, it was possible to observe the surface plaining with decrease in the wavy irregularities and pores appearing in isolated regions.

The images of composite resin restorations obtained through profilometric analysis and SEM showed no alterations of their surfaces after the bleaching treatment.

DISCUSSION

Contrary to the samples analyzed under SEM, the samples to be analyzed in the profilometer need no special preparation, such as desiccation and metallization. Thus, the image obtained through profilometric analysis is not influenced by any previous procedure.^[8] The association of the two imaging techniques, as performed in this present study, permitted the profilometric analysis quantitative data to be complemented by the SEM qualitative data.

Among the various types of roughness parameters, only the data from Ra and Rsk would provide relevant information. The Ra shows the average between the peaks and depressions of the surface, and the higher its score the more remarked the roughness will be. The Rsk shows the predominance of peaks or depressions on the surface, where the depressions are featured with negative scores and the peaks with positive scores. The latter parameter becomes

quite relevant considering that an area which features a predominance of depressions tends to accumulate a larger amount of materials on its surface. In the case of dental fillings and dental enamel, it is possible to theorize that the alterations which lead to a rise in Ra as well as to Rsk negative score may cause a greater accumulation of food remainders and bacterial plaque on their surfaces.

In the images obtained through SEM it is possible to observe enamel alterations after the treatment with 35% hydrogen peroxide. There were small orifices in some regions indicating an erosive effect of the bleaching agent upon the surface. There was also a decrease in the surface Rsk associated with the orifices. These data are compatible with those found in the profilometric analysis; the score of the enamel Ra after the treatment showed no statistically significant variation, however, there was a downward trend. The Rsk scores showed a statistically significant decrease that indicates a rise in the ratio of depressions to peaks, considering the material topography. This finding showed to be compatible with the orifices featured in the SEM images.

McGuckin *et al.*,^[15] used solutions of 10% carbamide peroxide and 30% hydrogen peroxide onto the enamel surfaces of human premolar teeth, and observed them under SEM and through the profilometer. The images showed that the carbamide peroxide caused a decrease in the enamel superficial roughness scores. In the teeth treated with hydrogen peroxide, rougher and more conditioned surfaces were observed due to the acid etching performed before the treatment. Ben-Amar *et al.*^[16] also observed an increase in the enamel smoothness after the treatment with 10% carbamide peroxide, besides the presence of pores in some specific areas. These findings are in accordance with those obtained in the present study.

The reactions caused by free radicals are not specific and may modify various organic structures. Therefore, when a bleaching treatment is carried out the enamel organic matrix may be broken and damaged. This would lead to a higher enamel permeability and lower resistance to

fracture propagation. These structural alterations induce modifications of the enamel's mechanical properties.^[3] However, Leonard, Jr.^[17] reported a 7-year clinical follow-up of patients who underwent dental bleaching, where no tooth showed fracture of either the enamel or the fillings.

The pH of bleaching agents is closely related to the alterations of the enamel surface caused by them.^[4,10,15] According to several authors, the more neutral the bleaching agent's pH the slighter the enamel alterations will be. Some products contain small amounts of phosphoric and citric acid in their formulae. These acids are used in order to stabilize and preserve the materials, however, they may cause dental structure conditioning.^[18] We used a bleaching agent with pH 4.2, while the enamel demineralization process initiates when the pH reaches the critical rate between 5.2 and 5.8.^[18] The enamel alterations found in this study can be related to the low pH, however Tames *et al.*^[7] used a solution of 10% carbamide peroxide with pH 6.2, and found alterations in the enamel after the treatment period. Thus, it is possible to state that the pH may influence in the action of bleaching agents onto hard tissues, although it is not the only factor which can cause the alterations of these tissues.

A rougher surface not only facilitates bacterial attachment but also makes it difficult to be removed by mechanical procedures. According to Gürkan *et al.*^[19], oral bacterial attachment to both the enamel and restorative materials is notoriously harmful to these structures, being a relevant factor for the development of caries and periodontal disease. The same authors did not observe an increase in the superficial roughness of the enamel after treatment with bleaching agent, however, they could observe an increase in bacterial attachment. The authors conclude that bleaching agents cause alterations in the enamel microstructure, which enhances bacterial attachment. Although the present study has featured no increase in the enamel Ra, the results of the study carried out in 1997 are in accordance with those presented in the current study, for the presence of pores, which can enhance bacterial attachment.

Moreover, the results shown in this study confirm the results obtained by Silva *et al.*^[20], when they observed no statistically significant increase in the Ra of a hybrid composite resin after treatment with hydrogen peroxide solution, and oppose those found by Cooley and Burger^[21] and Morales *et al.*^[10] when they observed a statistically significant increase in the Ra of a hybrid composite resin after treatment with 10% carbamide peroxide and 35% hydrogen peroxide. In the images obtained under SEM and profilometry, it is not possible to observe alterations in the surface of this material either.

Nevertheless, in regard to Rsa, the current study showed a statistically significant decrease in composite resin fillings after being treated with bleaching agents. This phenomenon

is explained by an increase in the predominance of depressions on their topography due to the action of the hydrogen peroxide onto the fillings surface. It must be remarked that a surface featuring a negative Rsk shows a greater facility to accumulate debris, which in regard to composite resins may represent a greater accumulation of bacteria and extrinsic pigments. This hypothesis is strongly related to the results of the study performed by Mor *et al.*,^[22] when they found a stronger bacterial attachment to composite resin fillings submitted to a previous treatment with carbamide peroxide.

Bailey and Swift, Jr.^[12] reported a microhardness decrease in hybrid and microparticle composite resins after topical application of bleaching agents consisting in 10% carbamide peroxide. According to the authors, the microparticle resins are more susceptible to the adverse effects caused by bleaching agents due to their higher concentration of organic matrix. The softening of composite resins due to the bleaching agent's action is, according to the authors, difficult to explain since no component of the bleaching agent used in their study features parameters of solubility that can interact with Bis-GMA. In SEM analysis, the authors could observe the presence of cracks between the resin matrix and the prepolymerized particles of the microparticle resin, which is also a result of the bleaching agent's action onto this material. The decrease in Rsk, as found in the present study, may confirm this kind of alteration because the higher predominance of depressions on the topography may express the presence of cracks in the surface. Therefore, dental care providers should be concerned about the performance of a resin filling after dental bleaching procedure.

Due to the discrepancy between the results found in dental literature and those found in the current study, it is fair to presume that the amounts of organic matrix as well as the size of load particles of a composite resin are determining factors for the susceptibility of this material to the action of bleaching agents.

CONCLUSIONS

The treatment with 35% hydrogen peroxide caused no alteration in the interface between enamel and composite resin, Tetric Ceram, fillings and the topical application of 35% hydrogen peroxide on enamel and composite resin, Tetric Ceram, caused an alteration of their surface topography, featuring a predominance of depressions after the bleaching treatment.

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