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Effect of a 37% carbamide peroxide bleaching agent activated by halogen light or light emiting diodes (LEDs) on enamel surface roughness microhardness

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Bacci ACL, Flório FM, Basting RT. Efeito de um agente clareador de peróxido de carbamida a 37% ativado por luz halógena ou diodos emissores de luz (LEDs) na rugosidade e microdureza de superfície do esmalte. Rev Odontol UNESP. 2007; 36(4): 351-355.

Abstract: The purpose of this in vitro study was to evaluate the surface roughness and microhardness of human enamel treated with an in-office dental bleaching agent containing 37% carbamide peroxide – Magic Bleaching/ Vigodent - (CP) activated by either a halogen light (HL) or light emitting diodes (LED). Sixty enamel fragments were randomly divided into 5 groups were treated accordingly: 1) CP; 2) CP+HL; 3) CP+LED; 4) placebo agent (PLA) which contained carbopol and glycerin; 5) saliva (CON). During and after 4 weekly applications, enamel fragments were washed and kept in artificial saliva at 37 °C. Surface roughness was determined before and after treatments with a profilometer and cut-off set to 0.08 and microhardness was determined with a Knoop indenter with 25 g. Analysis of variance and Tukey test ($p < 0.05$) showed significant increase in surface roughness for all groups. There were no statistical significant changes in enamel microhardness after treatment with CP associated or not with light curing units. PLA group had higher roughness and lower microhardness. Irrespective of the treatment, enamel surface roughness was altered and enamel microhardness was unaffected by the experimental treatments. Halogen light curing units or light emitting diodes may be used in combination with in-office dental bleaching without significantly altering the enamel surface roughness and microhardness when compared to a control.

Keywords: *Tooth bleaching; dental enamel; hydrogen peroxide.*

Resumo: O objetivo deste estudo foi avaliar in vitro a rugosidade e a microdureza do esmalte dental humano submetido a um agente clareador de consultório contendo peróxido de carbamida a 37% - Magic Bleaching/ Vigodent (MB) - ativado por luz halógena (LH) ou por diodos emissores de luz (LED). Sessenta fragmentos de esmalte foram divididos aleatoriamente em 5 grupos que receberam diferentes tratamentos: 1) MB; 2) MB + LH; 3) MB + LED; 4) agente placebo (PLA) composto por carbopol + glicerina; e 5) saliva (CON). Durante e após 4 aplicações semanais, os corpos de prova foram lavados, imersos em saliva artificial e mantidos a 37 °C. A rugosidade foi mensurada antes e ao final dos tratamentos com um perfilômetro e *cut-off* de 0,08 e os testes de microdureza foram realizados com penetrador Knoop e carga de 25 g. A análise de variância e o teste de Tukey ($p < 0,05$) mostraram aumento significativo na rugosidade superficial para todos os grupos. O grupo que recebeu a aplicação de PLA apresentou maior valor de rugosidade e menor valor de microdureza. Independentemente do método de fotoativação, a rugosidade superficial do esmalte é alterada e a propriedade de microdureza apresenta as mesmas características após a utilização de um agente clareador de consultório contendo peróxido de carbamida a 37%.

Palavras-chave: *Clareamento de dente; esmalte dentário; peróxido de hidrogênio.*

Introduction

Dental bleaching has been used in Dentistry for a long time. However, only in 1960 was carbamide peroxide accidentally discovered by Klusmier as a bleaching agent. Its use and application in vital tooth bleaching techniques was described by Haywood, Heymann[1] in 1989.

Modifications of the original home technique included the use of thickening agents, such as carbopol and glycerin, which prolong the time for releasing the active ingredient and increasing the product adherence to the tooth[2]. The concentration of carbamide peroxide agents in the home technique ranges between 10 to 22%[2]. Higher concentrations (up to 37%) are used in the in-office bleaching techniques and these products are capable of bleaching teeth in a shorter period of time. This product might or might not include the use of a source of heat to accelerate the carbamide peroxide degradation and to release hydrogen peroxide[3,4].

Scanning electronic microscopy and microhardness evaluations showed the presence of erosions, porosities and alterations of the mineral content after enamel treatment with bleaching agents containing 10% carbamide peroxide[5-11]. Analysis of the tooth structure using X ray diffraction and infrared spectroscopy have shown the presence of alterations in the enamel with the use of carbamide peroxide at 35%[12], although the concentrations of 10 and 16% did not produce any modification. In vitro, such surface micromorphology alterations have led to an increase in surface roughness[10].

With respect to the in-office bleaching technique, the use of specific lamps to activate the degradation of the bleaching agent has been evaluated[3,4,13,14]. Such lamps are available through conventional curing units systems (halogen light) which may promote a rise in intra-pulpar temperature[13,15]. The purpose of this light is to minimize the time required for tooth bleaching[4] by activating or accelerating the effect of bleaching agents.

Currently, new light curing systems have used Light Emitting Diodes - LED, making the polymerization process of certain dentistry materials take place with a minimum of heat[16-19]. Their use may also be extended to the in-office tooth bleaching technique. Consequently, the enamel surface and the bleaching agent itself may undergo less exposure to heat, minimizing some of the undesirable effects, such as sensitivity, alterations to microhardness and enamel surface roughness.

Thus, the purpose of this in vitro study was to evaluate the surface roughness and microhardness of human enamel treated with an in-office dental bleaching agent containing 37% carbamide peroxide activated by either a halogen light or light emitting diodes.

Material and method

This study had the approval of the local Ethical Committee (process n. 074/02). Human third molars were used in this study and were stored in a thymol solution at 0.1% until the cleaning procedures were carried out with periodontal curettes and scalpel blades. Longitudinal cuts were made with diamond disks in the crown and resulted in slabs measuring 4 x 4 mm. The slabs that were cracked or fractured on analyses under stereoscopic microscope were discarded from the experiment. Using 2.0 cm diameter PVC molds, the slabs were embedded into polystyrene resin. After 24 hours, they were removed from the mold and kept under relative humidity.

The external surface of each enamel fragment was flattened in a rotary electrical polisher with aluminum oxide discs of decreasing granulation (400, 600 and 1000) under constant water cooling and polished with diamond pastes of 6, 3, ½ and ¼ µm under mineral oil cooling. The slabs that presented dentin exposure were eliminated after observation under stereoscopic microscope.

The sixty specimens were randomly divided into five groups (n = 12), as showed in Table 1.

The bleaching agent evaluated (Magic Bleaching/ Vigodent, Bonsucesso, RJ, Brazil – lot Nº 06302) contains 37% carbamide peroxide, thickening agents - carbopol and glycerin - in amounts not specified by the manufacturer. The placebo agent was in the form of a neutral gel (pH = 7.0) with the same characteristics of consistency and odor as

Table 1. Experimental and control groups of the study

Group		Treatment	Light curing unit/ Manufacturer
1	CP	In-office bleaching agent containing 37% carbamide peroxide	-
2	CP+HL	In-office bleaching agent containing 37%carbamide + photoactivation by a conventional halogen light curing unit	Degulux/Degussa, Postfach, Hanau, Germany
3	CP+LED	In-office bleaching agent containing 37%carbamide peroxide + photoactivation by a light emitting diodes curing unit	Ultraled/ Dabi Atlante, Ribeirão Preto, SP, Brazil
4	PLA	Placebo agent (carbopol gel + glycerin) used as positive control group to promote equal hydration to experimental units	-
5	CON	Immersion in artificial saliva used as a negative control group	-

the bleaching agent Magic Bleaching/ Vigodent containing carbopol (1.2%) and glycerin (5%) in its composition, but without the presence of 37% carbamide peroxide. The artificial saliva solution used is described by Featherstone et al.[20], and modified by Serra, Cury[21].

The light curing units used for activating the 37% carbamide peroxide were a light emitting diodes (LED) light curing unit (Ultraled/ Dabi Atlante) with mean intensity of 110 $mW.cm^{-2}$ (ranging from 120 to 105 $mW.cm^{-2}$) and the conventional halogen light curing unit (Degulux/ Degussa) with mean intensity of 570 $mW.cm^{-2}$ (ranging from 595 to 560 $mW.cm^{-2}$). They were measured by a radiometer (CL150/DMC, São Carlos, SP, Brazil).

The amount of bleaching agent or placebo agent used was 0.02 mL for each fragment that remained on the surface for 40 minutes. The groups CP+HL and CP+LED at the times of 15 and 30 initial minutes received the photoactivation by their respective appliances for 40 seconds on each specimen. The CON group remained at ambient temperature for the same period of time as the other groups receiving their treatments. When the total application was completed, the specimens of all groups were washed with distilled water and immersed in an artificial saliva solution at 37 °C ± 2 °C for the period of 7 days. The saliva solution was changed every 2 days. These procedures were repeated for 4 weeks, according to the manufacturer bleaching agent instructions.

The surface roughness of each enamel specimen was measured before (baseline values) and after the application of the treatment agents with a profilometer Surf-Corder SE 1700 (Kozaka lab. Ltd., Tokyo, Japan). A cut-off of 0.08 was used, in sequential mode, using the ISO BS parameter. To assess the mean roughness, 3 readings were taken of each test specimen (in the mesio-distal direction, occlusal-cervical direction and transversal direction). The profilometer needle run speed was 0.05 mm/s.

To assess the microhardness of the specimens after treatment, a microhardness meter Future Tech (FM-1e, Tokyo, Japan) was used. Five microhardness indentations were made in each specimen, using a Knoop penetrator and static load of 25 g for the period of 5 seconds. For the microhardness tests, the means in micrometers of 5 replicas of the microhardness meter indentation measurements were also considered. This measurement was used for calculating the Knoop hardness value.

For the roughness data, the means were considered in Ra of the 3 roughness readings before and after application of the treatment agents. The statistical analysis of the roughness and microhardness data was done by the parametric technique of the Analysis of Variance (ANOVA). The Tukey test was used in the comparisons among the results of each treatment agent.

Result

The results of the analysis of variance (ANOVA) and the Tukey test for the roughness tests may be seen in Table 2. For all treatment groups, there was a statistically significant increase in surface roughness from baseline ($p < 0.01$).

After the application of the different treatment agents, the placebo agent had the highest significant surface roughness values. Specimens treated with carbamide peroxide associated or not with light curing units (CP, CP+HL and CP+LED) showed no significant differences in surface roughness compared to the control group (CON).

The results obtained from the statistical analysis for the microhardness test are presented in Table 3. Specimens treated with the placebo agent (PLA) presented lower microhardness values, differing statistically from the other groups ($p < 0.05$).

Discussion

Studies that evaluate enamel surface roughness and micromorphology following treatment with home bleaching agents containing carbamide peroxide have shown the presence of erosions and porosities[5-8,10-11,22]. Other studies showed a greater roughness[10] and a greater adherence of *Streptococcus mutans* to the surface treated with carbamide peroxide[23]. Fur-

Table 2. Mean surface roughness values (Ra in µm) and standard deviations before and after the application of the different treatment agents

Group	Treatment	Surface roughess	
		Before	After
1	CP	0.057 ± 0.003 aA	0.079 ± 0.012 bA
2	CP+HL	0.057 ± 0.003 aA	0.066 ± 0.007 bA
3	CP+LED	0.057 ± 0.004 aA	0.069 ± 0.010 bA
4	PLA	0.057 ± 0.004 aA	0.096 ± 0.017 bB
5	CON	0.059 ± 0.003 aA	0.069 ± 0.010 bA

The means followed by different lower case letters in the horizontal line and different capital letters in the vertical line indicate statistical difference ($p < 0.01$)

Table 3. Mean Knoop microhardness values for the enamel submitted to the different treatment agents

Group	Treatment	Knoop microhardness values
1	CP	339.85 A
2	CP+HL	330.91 A
3	CP+LED	357.68 A
4	PLA	79.15 B
5	CON	337.53 A

The means followed by different capital letters indicate statistical difference ($p < 0.05$)

thermore, alterations in the enamel mineral content may be expected due to the acid properties of these materials and their components[2,6,8,11,22], and capable of being assessed through microhardness tests[20]. These effects are noted in spite of the presence of saliva, fluorides or other remineralizing solutions which are capable of maintaining the balance between the demineralization and remineralization processes.

In this study, a quantitative and non-destructive evaluation of the enamel surface was measured by means of a roughness meter or profilometer. After application of all the treatment agents, a statistically significant increase of enamel surface roughness was seen. However, this increase was also seen for the control group, which did not receive the treatment agents. This fact was intriguing, since care was taken during the procedures of manipulating the specimens (insertion, removal and drying of the storage mediums) and that the artificial saliva has no acidic properties.

However, observing the roughness values after treatment, specimens treated with bleaching agents whether or not associated with the sources of photoactivation, as well as the control group, did not present significant differences. This may be explained by the application regime of the agents containing 37% carbamide peroxide to the enamel for a relatively short time (four 40-minute applications). Although the higher concentrations of the bleaching agent seem to be a factor that changes enamel surface micromorphology[12], longer periods of exposure to carbamide peroxide in more prolonged treatments may also be pointed to as important factors for the appearance of alterations to the surface characteristics[5-8,10,11,19]. Furthermore, the absence of alterations in roughness may be a characteristic inherent to the methodology used, since a micromorphological analysis under a scanning electronic microscope[24] or an X ray diffraction analysis[12] could show such alterations.

However, it was observed that specimens treated with the placebo agent showed greater roughness values than the experimental and control groups. This fact may be explained by the presence of carboxylic acid (carbopol) which composed the placebo product. Polyacrylic acid may lead to demineralization[25], especially in a remineralizing solution, inhibiting the growth of hydroxyapatite crystals due to the chelation with calcium[26]. In spite of glycerin also being one of the placebo agent components, when associated with carbopol it also leads to demineralization of the enamel, although in isolation, it presents a more inert characteristic[3]. This may lead to alterations in roughness, as observed in the results of this study. As carbopol is presented in association with carbamide peroxide in commercial bleaching agents, in addition to other products that the manufacturer does not mention in the description of the material, its effect seems to be masked[25].

Microhardness alterations may be observed when using home bleaching agents containing carbamide peroxide[22,27-29]. Such alterations may be justified due to the experimental delineation and study model used, application time, agents used and concentration. However, in this study, 37% carbamide peroxide was used, showing that the high concentration used may have been compensated by the low frequency of application, leading to statistically similar microhardness values among the groups treated, the bleaching agent and the control group. Furthermore, the placebo agent presented lower microhardness, this fact is explained due to the presence of carbopol (carboxylic acid) and glycerin which, when associated, promote enamel demineralization[25].

Within the treatment regime used in this study, sources of light curing units evaluated – conventional halogen light curing or light emitting diodes (LED) - activated the degradation of the bleaching agent[30] and thus minimizing the time necessary for bleaching the tooth[30]. This may be done without significantly altering the enamel surface roughness and microhardness when compared to the control group.

Conclusion

Irrespective of the method of photo activation and in the presence of artificial saliva, the properties of enamel roughness and microhardness presented the same characteristics for the human dental enamel whether or not submitted to a bleaching agent containing 37%carbamide peroxide.

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