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ORIGINAL ARTICLE

Microhardness change of enamel due to bleaching with in-office bleaching gels of different acidity

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Abstract

Objective. The aim of this study was to assess the enamel microhardness treated with three in-office bleaching agents, containing 35% hydrogen peroxide with different acidity. ***Materials and methods.*** Bovine incisors were divided into three groups that received the following bleaching agents: Whiteness HP, Total Bleach and Opalescence Xtra. Three gel applications/10-min each, totaling 30-min of bleaching treatment, were made on the teeth and activated with a blue LED (1000 mW/470 nm) combined to a LASER (120 mW/795 nm) device (Easy Bleach–Clean Line). Vickers hardness (VH) was evaluated at baseline and after the bleaching procedure. The values of Hardness loss [HNL] (% reduction) were calculated. The two-sample t-test was used for comparison of the HNL of the three bleaching products (5% level of significance). ***Results.*** The Opalescence Xtra, which had the lowest pH value (pH = 4.30), showed a significant increase of HNL when compared with Total Bleach bleaching agent, which had the highest pH value (pH = 6.62). ***Conclusions.*** The 35% hydrogen peroxide bleaching agents resulted in a reduction in surface enamel microhardness and bleaching with the most acid agent resulted in a significant enamel hardness loss compared to the less acid agent (4.30 vs 6.62). Strategies proposed to reduce the enamel loss after bleaching treatment may include the use of daily fluoride therapy, mouth rinsing (fluoride, milk and sodium bicarbonate solution), fluoride/bicarbonate dentifrices without abrasives, do not toothbrush immediately after bleaching, fluorides and calcium add to bleaching agents.

Key Words: *bleaching, enamel, hydrogen peroxide, microhardness, pH*

Introduction

The in-office bleaching technique is a method of tooth bleaching treatment that uses a hydrogen peroxide-based gel at a high concentration of ~ 35%, which is applied to the teeth for a period of 30–60 min. However, some studies have shown that bleaching solution can cause deleterious effects on the tooth structure such as erosion, roughness, loss of mineral substance and tissue irritation [1–3]. These deleterious effects depend on the composition and concentration of the bleaching agents [4,5], exposure time [5] and acid pH of the bleaching solution [3,5].

The in-office bleaching gels presented in one bottle have an acid pH, ~ 2 [6,7], which purposes to remain stable and to avoid degradation, increasing its storage time and validity. However, the pH value of ~ 2 is below the critical pH for enamel demineralization, ~ 5.5 [6]. The critical pH of 5.5 refers to average human saliva with 0.8 mol/L of total non-protein bound calcium, 4.3 mol/L total phosphate and an ionic strength of 0.0275 mol/L, as described by Schmidt-Nielsen [7].

To avoid deleterious effects on the tooth structure due the acid pH of the bleaching solution, two-bottle bleaching gels were developed. The first bottle has the hydrogen peroxide solution with an acid pH stable at ambient temperature. The second bottle has an alkaline agent capable of raising the pH of the gel. According to the manufacturer's specifications,

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after these components are mixed, the pH value of the final solution results in values between 6.5–7, above the critical value of enamel demineralization [6,7].

The microhardness test has been used as a parameter to assess whether a demineralization process occurs. *In vitro* evaluations have reported alterations of the enamel microhardness after exposure of the surface enamel to hydrogen peroxide by in-office technique [4,5,8]. This alteration depends on the brand of the product used and particularly its acidity [3,5]. Some studies have shown that, with the use of more acid bleaching gels, more alterations in the enamel surface have been observed [3,5,9].

The aim of the current study was to evaluate the effect of bleaching agents containing 35% hydrogen peroxide with different acidity on enamel microhardness.

Materials and methods

Preparation of specimens

Thirty bovine incisor teeth were selected. The roots were sectioned with a flexible diamond steel disk in a handpiece until the distance of 2 mm from the cement–enamel junction was standardized. The pulps were extirpated from the pulp chamber and the crowns were embedded in self-polymerizing acrylic resin (Classic, São Paulo, Brazil). The enamel of the buccal surfaces were worn using abrasive papers (grit 600) coupled to a circular polishing machine (PA-10, Panambra, São Paulo, Brazil) under water cooling, to expose an area standardized in 3 × 3 mm. The exposed enamel portion was polished using abrasive papers (grit 800, 1200 and 4000) coupled to a circular polishing machine.

Microhardness test

Vickers microhardness, at a load of 200 g, with an indentation time of 10 s, was determined using a microhardness tester (FM-700, Future-Tech, Tokyo, Japan). Three indentations were performed on the surface of each specimen, with a distance of 50 μm between them and the mean Vickers hardness (VH) calculated. This first measured VH is the VH baseline that was used to calculate the values of Hardness loss.

Bleaching treatment

The 30 specimens were divided into three groups containing 10 specimens each according to the bleaching gel used (Table I):

- *Group 1*: The specimens received 35% Hydrogen Peroxide bleaching gel (Whiteness HP, FGM; Joinville, SC, Brazil), according to the manufacturer's specifications. The Whiteness HP bleaching agent presents two bottles, which are mixed at the time of use;
- *Group 2*: The specimens received 35% Hydrogen Peroxide bleaching gel (Total Bleach, Clean Line, Taubaté, São Paulo, Brazil), according to the manufacturer's specifications. The Total Bleach bleaching agent presents two bottles, which are mixed at the time of use; and
- *Group 3*: The specimens received 35% Hydrogen Peroxide bleaching gel (Opalescence Xtra, Ultradent, South Jordan, UT), according to the manufacturer's specifications. The Opalescence Xtra bleaching agent is presented in one bottle.

A 2 mm layer of bleaching gel was applied on the enamel surface of the specimens. The gel received five repetitive activations, using a 1 min switched and 1 min resting time (1 min ON–1 min OFF), with the hybrid device Easy Bleach (Clean Line, Taubaté, SP, Brazil) that has two blue light-emitting LEDs, each with a power of 500 mW, totaling an optical power of 1000 mW and a wavelength of 470 nm. This unit also presents an infrared diode LASER, with power of 120 mW and a wavelength of 795 nm (power density = 177 mW/cm^2).

After every 1 min period, the gel was mixed on the surface. After 10 min, the specimens were washed with an air/water spray for 30 s and dried with blown air. The specimens received two new applications of bleaching gel following the same treatments described above.

After bleaching treatment, three further indents were taken near to the baseline indents and the mean VH calculated, which is the VH measured that was used to calculate the values of Hardness loss.

pH measurement

For pH measurements, the pH meter M-20 model (Digicrom analítica Ltda, São Paulo, SP, Brazil) and a

Table I. Bleaching products used.

Product	Composition	pH values	Manufacturer
Whiteness HP	35% hydrogen peroxide, thickener, coloring, glycol, water	6.4	FGM, Joinville, SC, Brazil
Total Bleach	35% hydrogen peroxide, water, stabilizers, coloring, thickener, pH regulator and surfactant	6.62	Clean Line, Taubaté, SP, Brazil
Opalescence Xtra	35% hydrogen peroxide, carotene, thickener	4.3	Ultradent, South Jordan, UT, USA

double junction glass electrode DMCV-8 (Digimed, São Paulo, SP, Brazil) were used. The pH meter was initially calibrated using standard buffered solutions of pH 6.8 and 4.0 which are provided by the manufacturer. The bleaching gels were placed in 30 ml graduated plastic cups. The pH electrode was immersed inside the gel to allow uniform contact with the electrode tip. The bleaching gels were in contact with the pH electrode for 10 min at room temperature (25°C) and the pH values were recorded.

Statistical analysis

From the microhardness at baseline and after bleaching procedure, the values of Hardness loss [HNL] (% reduction) of each specimen were calculated according to the following formula: HNL = [(VH baseline – VH measured)/VH baseline] *100.

The two-sample *t*-test was used for comparison of the HNL values of the three bleaching products, at a 5% level of significance.

Results

Table I showed the pH values of different bleaching gels: Whiteness HP bleaching gel had a pH value of 6.40; Total Bleach bleaching gel had a pH value of 6.62; Opalescence Xtra bleaching gel had a pH value of 4.30.

Figure 1 shows the mean and standard-deviation of HNL for all three bleaching agents. It can be observed that the Opalescence Xtra showed the highest values of HNL (11.117% reduction) after the bleaching procedure compared to Total Bleach (6.91% reduction) and Whiteness HP (7.50% reduction).

Table II shows the comparison of the HNL between different groups. The application of the two-sample *t*-test revealed significant differences between Opalescence Xtra and Total Bleach bleaching agents ($p = 0.03$). The Opalescence Xtra, which had the lowest pH value, showed a significant increase of HNL when compared with Total Bleach bleaching agent, which had the highest pH value.

Discussion

Although some authors [10,11] affirm that dental bleaching is a safe procedure, many controversies are observed with regard to the adverse effects produced on the enamel tissue [1–3]. Some studies have shown changes in porosity and surface morphology of enamel, such as: erosion, roughness, mineral content loss and reduction in enamel microhardness after bleaching treatment [1–4,12]. Microhardness changes are related to a loss (demineralization) or gain of mineral (remineralization) and the microhardness test is suitable for determining small changes in surface enamel microhardness [13].

Attin et al. [5,14] showed a reduction in surface enamel microhardness after the in-office bleaching treatment with 35% hydrogen peroxide (Opalescence Xtra - Ultradent) activated with the light of a halogen lamp. Lewisntein et al. [15] also found that enamel and dentin microhardness diminished significantly after bleaching treatment with 35% hydrogen peroxide (Opalescence Xtra). Pinto et al. [4] showed a reduction in surface enamel microhardness after bleaching treatment with 35% hydrogen (Whiteness HP).

The highest HNL (% reduction) of Opalescence Xtra bleaching product are probably related to their lowest pH value compared to the other bleaching products (Figure 1). The results of this study indicated a significant reduction in microhardness of bleached enamel treated with the most acid bleaching gel (Opalescence Xtra/pH = 4.3) compared to the group treated with less acid bleaching gel (Total Bleach/pH = 6.62).

Some authors reported that the reduction in bleached enamel microhardness depends on the composition or concentration of hydrogen peroxide [4,5,16]. However, Attin et al. [5] attributed the reduction in enamel microhardness with 35%

Figure 1. Graph of mean Hardness Loss (HNL, % reduction) for the different bleaching agents. Hardness loss values which are not significantly different are marked with the same capital letters.

Table II. Results of two sample *t*-test.

Bleaching agent	HNL (% reduction) ± SD	*t*	*p*
Opalescence Xtra	11.117 ± 4.53	2.34	0.0305*
Total Bleach	6.91 ± 3.37		
Opalescence Xtra	11.117 ± 4.53	1.53	0.1429
Whiteness HP	7.50 ± 5.92		
Total Bleach	6.91 ± 3.37	−0.27	0.7895
Whiteness HP	7.50 ± 5.92		

Hardness loss (HNL) (% reduction); Vickers Hardness (VH). HNL were calculated according to the following formula: HNL = [(VH baseline – VH measured)/VH baseline] *100.
*Significant differences.

hydrogen peroxide (Opalencence Xtra) to exposure time and the acid pH of this bleaching agent. Additionally, Sulieman et al. [3] have reported that, following treatment with 35% hydrogen peroxide, there was no evidence of deleterious effects on enamel or dentin. They believe that studies reporting adverse effects on bleached enamel and/or dentin do not reflect the bleach itself; instead, they reflect on the pH of the formulation used.

In our study, the acid pH measured for Opalescence Xtra was 4.3, below the critical level for enamel, which is between 4.5–5.5 [6,7], therefore allowing enamel demineralization. The other gels used were less acid (Total Bleach = 6.62; Whiteness HP = 6.4), but they also caused a reduction in enamel microhardness, even with their pH levels being above the critical level for enamel demineralization.

The microhardness alterations in groups 1 and 2 are probably attributed to the reaction ability of the bleaching agent in relation to the organic phase of enamel, consequently affecting its mineral content. The strong oxidizing effect of hydrogen peroxide on the organic matrix plays a predominant role in the post-bleaching alterations observed in bleached teeth; this can be enhanced by the low pH of the bleaching agent, leading to a decrease in enamel and dentin microhardness [5,17,18]. Additionally, Shannon et al. [9] observed micromorphologically that all bleaching agents lead to loss of enamel mineral content and the more acid pH resulted in the greatest mineral loss.

Another possible theoretical speculation about why the two gels (Total Bleach and Whiteness HP) with higher pH induced softening of enamel is that the saturation of these gels in respect of the minerals of enamel might have an influence on the demineralization/softening of the enamel. Low concentrations of calcium and phosphate and high concentrations of sodium and chloride present in these gels due to the bleaching agent can cause under-saturation with respect to hydroxyapatite to be able to dissolve and soften enamel.

Acid substances on the surface of the teeth for an extended period of time may lead to dental erosion, defined as a chemical process that leads to irreversible loss of dental hard tissue [19]. The alteration of the external enamel structure after bleaching treatment may be worse after cleaning with tooth brushing and/or abrasive dentifrices as they might remove the degraded enamel and enhance the phenomena of erosion and wear [20].

Strategies proposed to reduce the enamel loss after bleaching treatment may include the use of daily fluoride therapy [21,22], mouth rinsing with fluoride and neutral solutions (milk or sodium bicarbonate) [23], fluoride/bicarbonate dentifrices without abrasives [20], avoiding tooth brushing immediately after the bleaching procedure (remineralizing effect of saliva) are important methods to prevent possible erosion caused on the enamel due to the use of superficial cleaning treatments after bleaching agents [20,24]. In addition, the attempt to increase the microhardness means of the bleached enamel has been successfully accomplished by means of adding fluorides and calcium to bleaching agents [22,25,26].

Bleaching procedures should be carried out correctly, respecting the time and concentration of agents, according to the manufacturer's instructions and as substantiated by scientific knowledge, thereby ensuring the safety of the treatment. In addition, the use of the acidic bleaching agents that can cause a significant loss of enamel microhardness in bleached teeth should be avoided.

Conclusion

According to the limitations of the current study, it can be concluded that the 35% hydrogen peroxide bleaching agents resulted in a reduction in surface enamel microhardness and bleaching with the most acid agent (Opalescence Xtra/pH = 4.3), resulting in a significantly higher enamel hardness loss compared to the less acid agent (Total Bleach/pH = 6.62).

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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