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# Decomposition Rate, pH, and Enamel Color Alteration of At-Home and In-Office Bleaching Agents

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This study evaluated the decomposition rate (DR), pH, enamel color alteration ( $\Delta E$ ) and whiteness index ( $\Delta WI$ ) promoted by at-home and in-office bleaching. Enamel surface was submitted to (n=10): at-home (10%, 15%, 20% carbamide peroxide - CP, 6% hydrogen peroxide -HP) and three 35% HP agents with light irradiation (LED, laser, and halogen) or no treatment (control). The DR and pH of agents were measured after 0, 2, 4, 6 and 8 h (at-home) or after 5, 15, 20, 30 and 40 min (in-office). Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $\Delta E$ ,  $\Delta WI$ ) were determined at baseline and after bleaching. DR, pH,  $L^*$ ,  $a^*$ ,  $b^*$  data were analyzed by one-way (at-home) or two-way (in-office) repeated measures ANOVA and Tukey test.  $\Delta E$  and  $\Delta WI$ , by one-way (at-home) or two-way (in-office) ANOVA and Tukey test. DR of at-home agents was similar after 6 and 8 h ( $p>0.05$ ), with pH close to neutral (6.5 to 6.9, CP) or acid 5.9 (6% HP). From 4 to 8 h,  $\Delta E$  was higher for 15% and 20% CP compared with 10% CP ( $p<0.05$ ). After 40 min, DR of 35% HP agents was similar and all exhibited significant  $\Delta E$  in one application ( $p<0.05$ ), regardless light irradiation.  $\Delta WI$  indicated whitening effect with no differences among groups ( $p>0.05$ ). One 35% HP showed alkaline pH, and the others,  $pH < 5.5$ . At-home agents could be applied for 2 h (15%, 20% CP, 6% HP) and 4 h (10% CP) and the in-office agents, up to 40 min in one application, without light.

Key Words: hydrogen peroxide, hydrogen-ion concentration, color.

## Introduction

The transenamel/dentinal diffusion of hydrogen peroxide (HP) is a concern due to possible damages promoted by bleaching agents on vital pulp tissues (1). Some reports indicate that even low-concentrated HP infiltrates enamel and dentin structures, reaching the pulp chamber (2) and promoting cytotoxic effects to odontoblastic-like cells (3,4). These studies indicate that the cytotoxicity is dependent on the concentration of the HP byproducts that reach the pulp, and even low-concentrated (or at-home) carbamide peroxide (CP) agents could promote intense cytopathology effects, reducing cell viability (3,4). Moreover, the intrapulp concentration of the HP is dependent on dentin permeability (5), pulp and osmotic pressure (6), heat applied as a catalyst (7), quality and thickness of enamel/dentin (8,9), and, finally, time of application (10).

Carbamide peroxide was originally designed to be used overnight (6 to 8 h) (11), however, the application time recommended in literature for low concentrations of CP (10-16%) varies from one (12) to 4 h (2). The in-office technique, using high HP concentrations (35-38%), indicates three consecutive 15-min applications, repeated for up to four appointments, with seven-day intervals. Other in-office products indicate the continuous application of HP for 20 min, but manufacturers do not have scientific backgrounds on the decomposition behavior of HP. Overextending HP

application times on enamel could cause alterations on enamel surface (5) and lead to HP transenamel/dentin diffusion, increasing the occurrence of sensitivity (13).

The application of light, such as lasers, light-emitting diodes (LEDs), plasma arc and quartz-tungsten-halogen (QTH) lamps, have been proposed to, catalyze the HP decomposition by theoretically, increase the decomposition rate of oxygen to form oxygen-free radicals (14). This reaction could accelerate bleaching, decrease the clinical application time of whitening agents and possibly, decrease tooth sensitivity (14,15). The authors of an in vivo randomized, split-mouth clinical study observed that using a hybrid light (laser/LED) concomitant with high-concentrated HP agents reduced bleaching sensitivity and promoted similar whitening effect compared with conventional in-office bleaching (15). However, the use of light is still controversial as some authors suggest that lights could increase the intrapulpal temperature and the risk of post-operative sensitivity (14).

As scarce information is available regarding bleaching agent's decomposition on enamel surface, the aim of the present study was to evaluate the decomposition rate of low and high-concentrated bleaching agents and the effects of LED, laser, and halogen lamps in the decomposition of the high-concentrated agents. Additionally, enamel color and pH were evaluated as the optimal time of

decomposition should correspond to an effective enamel color alteration, and pH would indicate the products' safety. The null hypothesis tested were that (I) application times of low-concentrated bleaching agents could follow manufacturer's instructions, (II) the decomposition rate of high-concentrated agents is not influenced by the application of light (LED, laser or halogen) and (III) application times of high-concentrated bleaching agents could follow manufacturer's instructions.

## Material and Methods

### Specimen Preparation and Black Tea Staining.

Figure 1 displays the schematic representation of methodology. One hundred and sixty bovine incisors were collected, stored in 0.1% thymol solution (Labsynth, Diadema, SP, Brazil) and cleaned of gross debris. Teeth free of defects and with similar color and size were chosen. Prior to the chemical analyses, the bovine teeth were immersed and stained with black tea solution (Matte Leão, São Paulo, SP, Brazil) under agitation for 24 h (6 sachets; 500 mL of water). The staining method was adapted from that of Sulieman et al. (16). The crown of the stained teeth was isolated with nail varnish leaving a 16 mm<sup>2</sup> area of exposed enamel for the bleaching treatment.

### Group Division

The stained bovine teeth (160) were divided into low and

high-concentrated bleaching treatments: Bleaching with low-concentrated agents were treated with (n=10): 10% CP (Opalescence, Ultradent, South Jordan, UT, USA); 15% CP (Opalescence, Ultradent); 20% CP (Opalescence, Ultradent); 6% HP (Whiteness Class, FGM, Joinville, PR, Brazil). High concentrations of hydrogen peroxide (HP) agents (35% HP) were also evaluated (HP Maxx - Whiteness HP Maxx, FGM; HP Blue - Whiteness HP Blue: FGM or PO - Pola Office, SDI, Bayswater, Victoria, Australia) and each of these three agents were subdivided into four groups according to the light activation methods (n=10): LED, laser, halogen lamp or without light activation (Control - C). Table 1 displays the composition and application mode of the bleaching

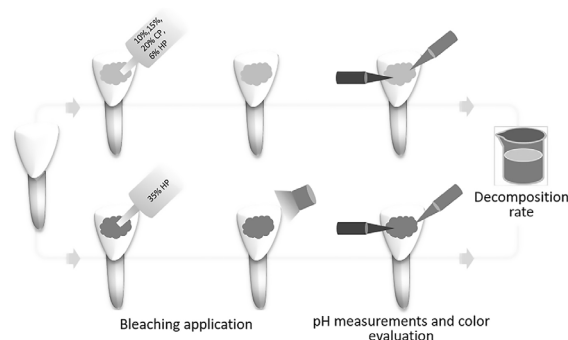


Figure 1. Schematic representation of methodology.

Table 1. Commercial name, composition, manufacturer directions, and experiment application protocol

Groups	Commercial name and manufacturer	Composition*	Manufacturer Directions*	Experimental application protocol
10% CP 15% CP 20% CP	Opalescence 10%, 15%, 20% (Ultradent Products Inc, South Jordan, UT, USA)	Carbamide peroxide, glicerín, carbopol, distilled water, 1100 ppm sodium fluoride	Bleaching for 6-8 h/d; 14-28 d	One application for 8 h.
6% HP	White Class (FGM Dental products, Joinville, SC, Brazil)	6% HP, neutralized carbopol, potassium nitrate, calcium gluconate, sodium fluoride, distilled water	Bleaching for 90 min/d; 14-28 d.	
HP Maxx	Whiteness HP Maxx (FGM Dental products, Joinville, SC, Brazil)	35% HP (after mixture), thickener, pigment, neutralizing agents, glycol, distilled water	After mixture, the agent is applied on the surface for 15 min and stirred 3-4 times to promote oxygen release; repeat twice. Light sources can be used to accelerate bleaching.	One application for 40 min.
HP Blue	Whiteness HP Blue Calcium (FGM Dental products, Joinville, SC, Brazil)	35% HP (after mixture), thickener, violet pigment, neutralizing agents, calcium gluconate, glycol, distilled water.	After mixture, apply on the surface for 40 min and stir every 5-10 min to promote oxygen release. Repeat the application up to 4 times with a 7-d interval. No light sources needed to accelerate bleaching.	
PO	Pola Office (SDI, Southern Dental Industries, Bayswater, Victoria, Australia)	35% HP and stabilizing agents; thickener, catalyzing agent, desensitizers, blue pigment.	The powder/liquid are mixed (1:4) and applied on dental surface for 8 to 20 min and repeated up to 3 times. Light activation is optional.	

\*MSDS data sheet.

agents according to the manufacturers. Table 2 presents the light sources, product features, and application protocol.

### Bleaching Treatment

The low-concentrated agents (10%, 15%, 20% CP, and 6% HP) were applied on the buccal surface of the bovine incisors and decomposition rate and pH were measured immediately (0) and every two hours until the end of the bleaching treatment (2, 4, 6 and 8 h). Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $\Delta E$ ,  $\Delta WI$ ) were measured at baseline and after 8 h of bleaching. Samples were stored over sterile cotton gauze soaked with distilled water at 37 °C throughout the bleaching treatment. The decomposition rate and pH of the 35% HP agents were evaluated at 0, 5, 10, 20, 30, and 40 min throughout the bleaching treatment, at room temperature (20 °C) to simulate the in-office technique. Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $\Delta E$ ,  $\Delta WI$ ) were measured at baseline and after 40 min of bleaching. The flowchart (Fig. 2) exhibits the group division and the time line of

the evaluations.

### Decomposition of Bleaching Agents

A known amount of the bleaching agent (0.25 g) was applied on the enamel, and at each evaluation, an aliquot was collected, weighed (0.01 g) and diluted in 10 mL of water. The diluted bleaching was added to concentrate sulfuric acid (20 mL). The concentration of hydrogen peroxide in this solution was determined by titration with 0.1 N potassium permanganate. This method is based on a reduction-oxidation reaction and quantifies the amount of hydrogen peroxide in the solution (17). Potassium permanganate ( $KMnO_4$ : 0.2 g of  $Na_2C_2O_4$  + 250 mL of  $H_2O$  + 15 mL of  $H_2SO_4$ ) was added to the diluted bleaching agent at a rate of 0.1 mL/s until a violet color was observed. The color change corresponds to the equivalence point, when all the  $H_2O_2$  had been consumed. The known amount of  $KMnO_4$  and the known amount of the bleaching agent were applied to the formula:  $C = V \times C_f \times 1,701 \times 100 / mg$  (Where:  $C = H_2O_2$

concentration (w/w);  $V =$  volume of  $KMnO_4$  added during titration;  $C_f =$  correction factor for the 0.1 N potassium permanganate solution; and  $m =$  mass of the bleaching product in milligrams) (17). Titration was performed at the intervals 0, 2, 4, 6, and 8 h of bleaching with low-concentrated agents, and at 0, 5, 10, 20, 30, and 40 min of bleaching with high-concentrated agents.

### Color Evaluation and pH Measurement

Simultaneously with the decomposition evaluations, pH of the bleaching agent was determined and color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) were measured at baseline and after bleaching. Prior to the color evaluation, the bleaching material was removed with a resin composite instrument (8AL,

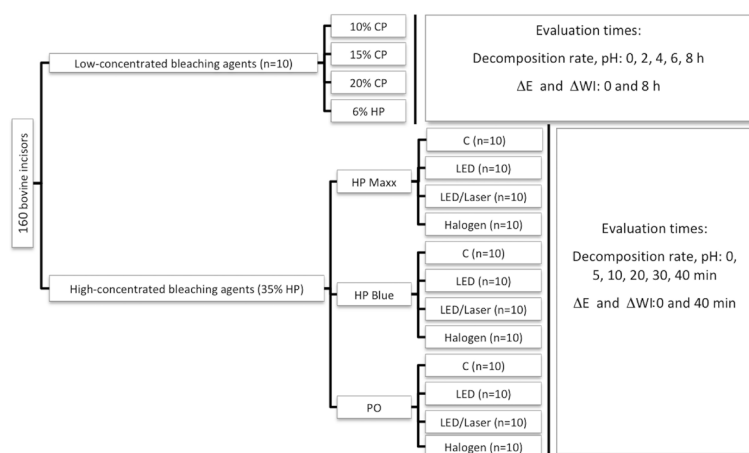


Fig. 2 Flowchart of the group division and the time line of decomposition rate,  $\Delta E$ ,  $\Delta W$  and pH evaluations.

Table 2. Light sources, product features, and application protocol

Light sources	Commercial names	Characteristics	Application protocol
LED	Valo (Ultradent Products Inc, South Jordan, UT, USA)	Wavelength: 395-480 nm. Light intensity: 790 mW/cm <sup>2</sup> .	The bleaching gel remains 1 min without agitation and is light irradiated for 2 min. The procedure must be repeated 3 times with 1-min interval among irradiations.
Laser diode	Whitening Lase Light Plus (DMC Equipment, São Carlos, SP, Brazil)	LED wavelength: 470 nm. Infrared diode laser wave length (3): 810 nm and power of 0.2 W.	The bleaching gel remains 1 min on surface without agitation and is light irradiated for 2 min. The procedure can be repeated 3 times with 1-min interval among irradiations.
Halogen	Optilux 501 (Demetron/Kerr, Danbury, CT, USA)	Wavelength: 560 nm. Light intensity: 600-800 mW/cm <sup>2</sup> .	The bleaching agent is applied on the surface for 2 min and irradiated for 30 s. The irradiation is repeated 3 times with a 2-min interval.

MSDS data sheet.

Cosmedent Restorative Dental Products, Chicago, IL, USA) and the enamel surface was cleaned with distilled water and air dried. The same gel that was removed was re-applied after color measurements. Color evaluation was determined with a spectrophotometer (Vita Easyshade Advance, VITA Zahnfabrik H. Rauter GmbH & Co. KG, Bad Sackingen, Germany) and the tip of the device was placed perpendicularly, in contact with the exposed enamel area. A silicon mold matrix was used to standardize the position of the tip. In each evaluation, three measurements were recorded according to the color parameters  $L^*$ ,  $a^*$ , and  $b^*$ , of the CIE Lab (Commission Internationale de l'Eclairage,  $L^*$ ,  $a^*$ ,  $b^*$ ) system. The CIE color  $L^*$  parameter corresponds to the value or degree or lightness, whereas  $a^*$  and  $b^*$  co-ordinates designate positions on red/green ( $+a^*$ =red,  $-a^*$ =green) and yellow/blue ( $+b^*$ =yellow,  $-b^*$ =blue) axes. The color parameters were averaged and color differences ( $\Delta E$ ) calculated using the following equation at each interval:  $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ .

The whiteness index for Dentistry was calculated using the  $L^*$ ,  $a^*$ ,  $b^*$  parameters of transmittance analysis, according to the equation:  $WI = 0.511L^* - 2.3424a^* - 1.100b^*$  (18), in which, higher WI values indicate whiter samples and lower WI (including negative values), indicate darker samples. According to this index, the whiter the material, the higher and more constant is the reflectance across the visible wavelength range (near to 100% of reflectance value of 1) (18). The baseline and final (after bleaching) whiteness index was calculated according to:  $\Delta WI = WI_{\text{after bleaching}} - WI_{\text{baseline}}$ .

The pH of bleaching agents was measured throughout the bleaching treatment at the intervals previously established, with a potentiometer (Equilam, Diadema, SP, Brazil) previously calibrated with buffered solutions (pH 4.0 and pH 7.0).

### Statistical Analysis

Data was submitted to exploratory analysis using SAS software (Realease 9.1, SAS Institute, Cary, NC, USA). The mean values were submitted to one-way (at-home bleaching agents) or two-way (in-office agents and light activation) repeated measures ANOVA followed by the Tukey test, at a 5% level of confidence, for the variable responses decomposition rate and pH,  $L^*$ ,  $a^*$  and  $b^*$ . Color alteration ( $\Delta E$ ) and whiteness index ( $\Delta WI$ ) were evaluated by one-way (at-home bleaching agents) and two-way (in-office agents and light activation) ANOVA, followed by the Tukey test ( $\alpha=0.05$ ).

## Results

### Decomposition of Low-Concentrated Bleaching Agents.

From 0 to 4 h groups presented different HP

concentrations, according to their initial HP amount. However, after 6 and 8 h no differences were detected among the groups ( $p>0.05$ ). The HP percentage of 10% CP decreased after 4 h and remained constant up to 8 h. The HP amount of 15% CP decreased after 2 and 8 h, whereas for 20% CP the concentration declined after 2, 4 and 6 h and no statistical differences were observed in the decomposition rate from 6 to 8 h of bleaching ( $p>0.05$ ). The HP concentration of 6% HP decreased after 2 and 4 h and remained until 8 h of bleaching. Figure 3 displays the decomposition rate of the low-concentrated agents as a function of time.

### Decomposition of High-Concentrated Bleaching Agents

The HP rate of HP Maxx decreased after 5 min of bleaching regardless of light activation, and after 40 min no differences were observed among HP Maxx groups regardless light activation. The decomposition of HP Blue without light activation (C) significantly decreased only after 30 min of bleaching. Contrarily, LED, laser, and halogen lamp hastened the decomposition rate, as it was significant after 5, 10, and 20 min of bleaching, respectively. After 40 min, the HP rate was lower in the HP Blue laser and halogen lamp than in the LED and C groups. Decomposition of PO was observed after 5 min of the LED and laser groups and after 10 min for the halogen and C groups. After 40 min of bleaching, no differences were observed among the PO decomposition rates (regardless of light activation). The average decomposition of the high-concentrated agents was 15%. Consequently, most groups reached a final average concentration of 20% HP. Figure 4 displays the decomposition rates of the high-concentrated agents as a function of time and light activated methods.

### Color Alteration of Low-Concentrated Bleaching Agents

At baseline, bleaching agents exhibited similar  $L^*$ ,  $a^*$ ,  $b^*$  (Table 3). After 8 h of bleaching, lightness ( $L^*$ ) increased,

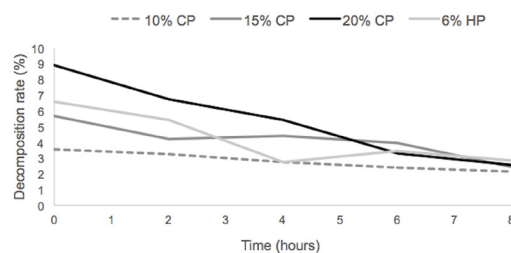


Figure. 3 Decomposition rate of hydrogen peroxide (%) of 10% CP, 15% CP, 20% CP, and 6% HP as a function of time.

b\*(yellow-blue) values decreased and a\* (red-green) values decreased for 15% CP and 20% CP groups. In the first 2 h, ΔE was similar among groups (p>0.05) (Table 4). The ΔE values of 20% CP at the time points 4h - 2h, 6h - 4h, 8 h - 6 h and 8h - Baseline were greater than that of 10% CP (p<0.05) and similar to 6% HP and 15% CP (Table 4). ΔWI exhibited positive values and no differences were detected in the whiteness index among groups (p>0.05) (Table 4).

**Color Alteration of the High-Concentrated Bleaching Agents**

At baseline, bleaching agents exhibited similar L\*, a\*, b\* (Table 5). After 40 min of bleaching, lightness (L\*) increased, b\* and a\* values decreased. No differences in ΔE were detected among bleaching agents at the time points 5 min - Baseline and 10 min - 5 min, regardless light activation (p>0.05) (Table 6). The use of light associated

with HP Maxx (regardless the light source) increased ΔE at the interval 40 min - 30 min compared to control group. However, no differences were observed in ΔE of HP Maxx, HP Blue and PO- treated groups at 40 min - Baseline. ΔWI exhibited positive values and no differences were found in the whiteness index among groups, regardless bleaching protocols or light activation modes (p>0.05) (Table 6).

**pH of Low-Concentrated Bleaching Agents.**

Bleaching agents exhibited different pH values during evaluations (p<0.05). The pH of 10%, 15%, and 20% CP at the intervals (0, 2, 4, 6, and 8 h) were close to 7.0, while 6% HP was lower, ranging from 5.8 to 6.1 (p<0.05). No differences were observed for 10%, 15%, and 20% CP and no significant variation was observed from baseline to 8 h of treatment for any of the low-concentrated bleaching agents tested (p>0.05). Figure 5 displays pH variations of the low-concentrated agents as a function of time.

**pH of High-Concentrated Bleaching Agents.**

High-concentrated agents exhibited differences in pH throughout the evaluation (p<0.00001). However, light activation did no change the pH of such agents (p=0.3496). HP Blue demonstrated the greatest pH values (8.3 - 8.5)

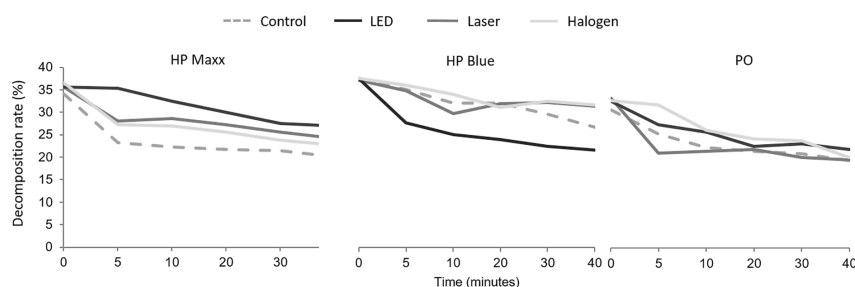


Fig. 4 Decomposition rate (%) of 35% HP agents as a function of time and light activation methods.

Table 3. L\*, a\*, b\* mean values (standard deviation) according to the bleaching agents

Groups	L*		a*		b*	
	Baseline	After treatment	Baseline	After treatment	Baseline	After treatment
6 % HP	64.6 (3.6) Aa	71.8 (2.9) Ab	7.6 (1.1) Aa	5.9 (0.7) Aa	19.3 (1.6) Aa	15.1 (1.5) Ab
10% CP	65.1 (2.2) Aa	72.4 (2.1) Ab	6.5 (2.2) Aa	4.4 (1.7) Aa	19.6 (1.9) Aa	14.9 (1.5) Ab
15% CP	61.3 (3.8) Aa	69.1 (3.1) Ab	8.0 (1.7) Aa	4.2 (1.3) ABb	17.4 (1.6) Aa	13.1 (2.1) Ab
20% CP	65.3 (3.6) Aa	72.1 (2.6) Ab	5.9 (1.7) Aa	3.0 (1.2) Bb	17.8 (1.8) Aa	13.8 (1.6) Ab

Means followed by distinct letters differ statistically at 5%, according to one-way repeated measures ANOVA and Tukey test (α=0.05). Uppercase letters compare bleaching; lowercase letters compare time After treatment: 8h of bleaching.

Table 4. Color alteration (ΔE) and whiteness index (ΔWI) mean values (standard deviation) according to the bleaching agents at different time points.

Group	2 h - Baseline	4 h - 2 h	6 h - 4 h	8 h - 6 h	8 h - Baseline	ΔWI
6% HP	4.5 (0.4) A	4.9 (1.3) AB	5.7 (1.2) AB	7.3 (1.1) AB	9.3 (2.2) AB	12.3 (3.6) A
10% CP	3.3 (0.8) A	3.7 (2.2) B	4.8 (2.1) B	5.3 (3.2) B	7.8 (2.1) B	14.0 (6.0) A
15% CP	3.8 (0.6) A	4.4 (0.9) AB	6.7 (2.5) A	8.4 (2.5) A	11.5 (1.8) A	17.6 (4.4) A
20% CP	4.9 (0.7) A	5.5 (2.7) A	6.9 (2.9) A	8.8 (2.3) A	13.8 (2.1) A	14.6 (7.1) A

Means followed by distinct letters comparing differ statistically at 5% according to one-way ANOVA and Tukey test (α=0.05). Uppercase letters compare bleaching treatment.

Decomposition rate of pH of bleaching agents

followed by HP Maxx (5.1 - 6.0) and PO, which had the lowest pH values (2.6 - 3.7) among the groups ( $p < 0.0001$ ). After 5 min of bleaching, the pH of PO laser increased from 2.6 to 3.1 and reach the pH of 3.5. Figure 6 displays pH variations of the high-concentrated agents as a function of time and light activation methods.

## Discussion

The agents 6% HP, 15% CP, and 20% CP exhibited

significant decomposition at the beginning of the bleaching therapy (after 2 h), whereas the decomposition rate of the 10% CP agent decreased only after 4 h of bleaching. This might indicate that the agents with the lowest HP concentration present a more time-consuming decomposition rate than the most concentrated ones. In fact, this could be observed in the color alteration ( $\Delta E$ ) results, which are a consequence of the HP decomposition.

Table 5. L\*, a\*, b\* mean values (standard deviation) according to the bleaching agents and light activation

Bleaching agent	Group	L*				a*				b*			
		Baseline		After treatment		Baseline		After treatment		Baseline		After treatment	
HP Maxx	Control	62.2 (3.8)	Aa	79.6 (3.5)	Aa*	6.8 (0.9)	Aa	4.7 (1.3)	Aa*	19.5 (2.3)	Aa	13.5 (2.6)	Aa*
	Led	62.4 (2.3)	Aa	80.6 (2.7)	Aa*	5.2 (1.8)	Aa	1.5 (0.8)	Ba*	19.3 (1.9)	Aa	9.4 (1.0)	Bb*
	Laser	61.3 (3.8)	Aa	80.8 (2.5)	Aa*	5.8 (0.9)	Aa	2.0 (1.3)	Ba*	17.4 (1.6)	Aa	10.6 (2.4)	Ba*
	Halogen	59.0 (1.9)	Aa	78.9 (1.9)	Aa*	5.9 (1.8)	Ab	3.1 (0.6)	AB*	17.8 (1.8)	Aa	8.9 (1.0)	Ba*
HP Blue	Control	58.8 (5.1)	Aa	76.2 (3.1)	Ab*	6.4 (1.2)	Ab	2.7 (1.4)	Ab*	19.0 (1.1)	Aa	9.9 (1.9)	Ab*
	Led	60.0 (3.0)	Aa	76.3 (4.3)	Ab*	6.6 (1.7)	Aa	1.5 (0.5)	Aa*	19.6 (2.0)	Aa	12.2 (1.5)	Aa*
	Laser	62.8 (3.6)	Aa	74.4 (1.9)	Ab*	6.3 (1.4)	Aa	2.9 (0.8)	Aa*	17.5 (3.8)	Aa	9.7 (0.9)	Aa*
	Halogen	62.5 (4.1)	Aa	78.5 (4.3)	Aa*	5.3 (1.1)	Ab	2.2 (0.8)	Ab*	18.2 (1.5)	Aa	9.8 (1.2)	Aa*
PO	Control	62.8 (1.8)	Aa	78.2 (3.3)	Aa*	4.6 (1.4)	Ab	1.9 (0.8)	Ab*	17.8 (1.8)	Aa	9.5 (1.9)	Ab*
	Led	62.1 (2.5)	Aa	77.6 (3.6)	Aa*	6.0 (2.0)	Aa	2.0 (0.5)	Aa*	17.9 (1.1)	Aa	8.9 (1.3)	Ab*
	Laser	61.5 (3.0)	Aa	77.9 (3.4)	Aa*	7.0 (1.4)	Aa	3.3 (1.3)	Aa*	17.8 (1.4)	Aa	8.9 (0.6)	Aa*
	Halogen	60.1 (2.0)	Aa	75.6 (2.4)	Ab*	7.5 (1.5)	Aa	3.9 (2.0)	Aa*	19.2 (1.9)	Aa	9.9 (1.8)	Aa*

Means followed by distinct letters differ statistically at 5%, according to two-way repeated measures ANOVA and Tukey test ( $\alpha = 0.05$ ). Uppercase letters compare lights for the same bleaching condition and time; lowercase letters compare bleaching agents for the same light condition and time. \*Indicate differences between baseline and after treatment. After treatment: 40 min of bleaching.

Table 6. Color alteration ( $\Delta E$ ) and whiteness index ( $\Delta WI$ ) mean values (standard deviation) according to the bleaching agents and light activation.

Group	$\Delta E$												$\Delta WI$		
	5 min - Baseline		10 min - 5 min		20 min - 10 min		30 min - 20 min		40 min - 30 min		40 min - Baseline				
HP Maxx	C	7.0 (0.2)	Aa	8.0 (0.3)	Aa	10.1 (0.1)	Aa	10.6 (0.3)	Bb	10.2 (0.2)	Cb	19.3 (3.6)	Ab	21.6 (8.4)	Aa
	LED	11.0 (0.4)	Aa	11.5 (0.4)	Aa	15.7 (0.3)	Ba	19.2 (0.3)	Aa	21.0 (0.3)	Ab	20.7 (3.7)	Ab	24.8 (2.7)	Aa
	Laser	8.2 (0.5)	Ab	8.9 (0.5)	Ab	13.4 (0.3)	ABb	14.2 (0.7)	ABb	15.7 (0.3)	Bb	20.9 (4.9)	Aa	22.5 (3.7)	Aa
	Hal	10.0 (0.2)	Aa	10.3 (0.4)	Aa	12.5 (0.4)	Ab	15.9 (0.5)	ABb	16.4 (0.3)	Bb	21.9 (2.4)	Aa	22.4 (2.9)	Aa
HP Blue	C	9.6 (0.4)	Aa	10.2 (0.5)	Aa	15.5 (0.6)	Aa	19.2 (0.4)	Aa	22.3 (0.4)	Aa	22.3 (5.6)	Ab	27.5 (4.5)	Aa
	LED	8.9 (0.3)	Ab	9.3 (0.3)	Ab	15.6 (0.2)	Aa	16.7 (0.5)	Ab	18.7 (0.7)	Ba	21.1 (3.8)	Ab	28.4 (2.8)	Aa
	Laser	9.8 (0.5)	Ab	10.0 (0.3)	Aa	16.7 (0.7)	Aa	18.2 (0.3)	Aa	19.5 (0.6)	ABa	20.4 (2.8)	Aa	22.6 (5.9)	Aa
	Hal	10.2 (0.2)	Aa	11.0 (0.6)	Aa	17.9 (0.6)	Aa	18.6 (0.5)	Aa	18.2 (0.4)	Ba	22.8 (3.9)	Aa	24.7 (6.1)	Aa
Pola Office	C	10.9 (0.3)	Aa	10.7 (0.5)	Aa	17.4 (0.1)	Aa	19.7 (0.4)	Aa	19.2 (0.4)	Ba	24.9 (2.6)	Aa	23.3 (3.3)	Aa
	LED	7.0 (0.5)	Ab	7.8 (0.2)	Ab	15.5 (0.6)	Aa	18.7 (0.5)	Aa	22.7 (0.4)	Ab	23.9 (0.2)	Aa	27.1 (5.7)	Aa
	Laser	11.2 (0.6)	Aa	12.1 (0.3)	Aa	17.7 (0.3)	Aa	17.5 (0.4)	Aa	20.9 (0.5)	ABa	20.7 (0.3)	Aa	26.7 (4.2)	Aa
	Hal	10.7 (0.4)	Aa	11.2 (0.4)	Aa	17.4 (0.5)	Aa	19.1 (0.9)	Aa	22.3 (0.3)	Aa	22.6 (0.3)	Aa	26.8 (6.6)	Aa

Means followed by distinct letters differ statistically at 5%, according to two-way ANOVA and Tukey Test ( $\alpha = 0.05$ ). Uppercase letters compare lights for the same bleaching condition; lowercase letters compare bleaching agents for the same light conditions. After treatment: 40 min of bleaching

The  $\Delta E$  values of 20% CP (at the time points 4h - 2h, 6h - 4h, 8h - 6h and 8h - Baseline) were greater than that of 10% CP ( $p < 0.05$ ) and similar to 6% HP and 15% CP. However, it should be noted that after 8h, all groups exhibited increase in luminosity ( $L^*$ ), decrease in the yellow appearance of enamel ( $b^*$ ) and exceeded the value for clinical color perception ( $\Delta E > 3.7$ ) (19). The whiteness index used (WI) was specifically formulated for Dentistry and is a simple linear formulation using the three CIELAB chromatic coordinates ( $L^*$ ,  $a^*$ ,  $b^*$ ), aiming to avoid subjective visual factor in the dental color analysis (18). According to this method, the high positive values of the WI index indicate higher whiteness values. Low or even negative values disclose lower values of whiteness of the sample (18). In this study, only positive values were found for the low and high-concentrated agents, which indicated that even one bleaching application was able to whiten the black tea-stained enamel surface.

A single eight-hour application of 10% CP promoted lower  $\Delta E$  compared to 15% and 20% CP groups (Table 4). Contrary to these findings, Delfino, et al. (20) observed that color changes promoted by 16% CP were similar to those of 10% CP and this agent exhibited  $\Delta E$  values superior to the 6.5% HP bleaching strips. However, the authors evaluated

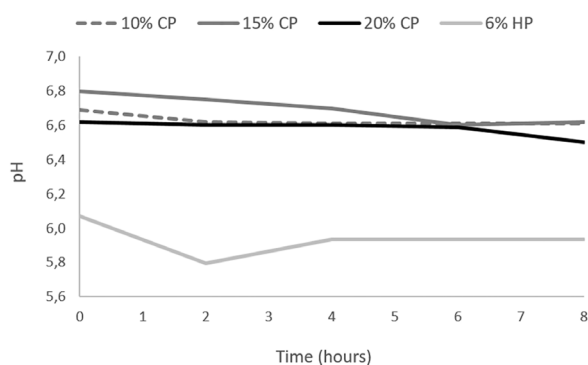


Fig. 5 pH analysis of 10% CP, 15% CP, 20% CP, and 6% HP as a function of time.

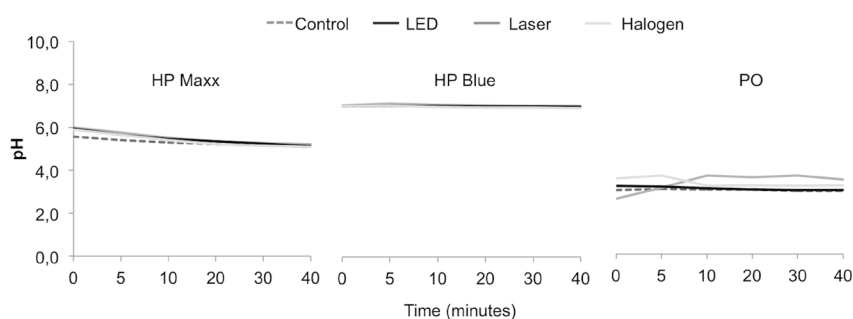


Fig. 6 pH analysis of 35% HP agents as a function of time and light activation method.

color after 7, 14, and 21 days of bleaching instead of the eight-hour decomposition rate/ $\Delta E$  follow up, as this study aimed to evaluate. In a clinical trial, Cardoso, et al. (12) evaluated the effectiveness promoted by 10% CP used for 15, 30 min, 1 h and 8 h/day for 16 days. Patients who experienced bleaching for 8 h/day exhibited a slightly faster bleaching result than patients that bleached for 1 h/day, but both application times were considered satisfactory (12). According to these authors (12,20), 10% CP could be recommended for lower application times and lower frequency (14 days) (20).

The CP agents (10%, 15%, and 20%) presented pH values close to neutral (6.5-6.9), whereas 6% HP displayed lower pH values (6.1 - 5.9, from the beginning to the end of the bleaching treatment). Although 6% HP is not below the critical 5.5 pH value to promote enamel mineral loss (21), according to manufacturer's it should be used for 90 min/day.

These findings provide background to reject the first null hypothesis and suggest a protocol change for the low-concentrated bleaching therapy based on the decomposition rate, color change and pH of the bleaching agents. Instead of 8 h of bleaching, 10% CP agent can be used for 4 h and 6% HP, 15% CP, and 20% CP agents can be used for up to 2 h, but the bleaching therapy is extended for up to 14 days (12). Amongst the bleaching agents, we recommend the lowest concentrated agent (10% CP) for home applications, in order to prevent possible cytotoxicity effects and lower sensitivity (22).

Some manufacturers of high-concentrated HP agents for in-office application indicate light activation (LED, Laser, or halogen lamp), with the purpose to increase the efficacy and accelerate decomposition rate of the agent, based on the thermocatalytic theory of HP decomposition (23). This theory supports the idea that the production of hydroxyl radicals is hastened by temperature increase and therefore, when light energy reaches the bleaching agent, a fraction of this energy is absorbed and converted into heat. In order to increase light absorption, some agents contain

a specific photosynthetic pigment such as carotene. This agent has a reddish color and increases blue light absorption. Another method used by manufacturers to increase light absorption is the addition of nanometer silica particles, which provides the blue color to the bleaching agents (23).

The decomposition of HP Maxx began 5 min after



application, irrespective of light activation and after 40 min, no differences were observed among HP Maxx groups, regardless light activation. Similar to these results, Hein et al. reported that the application of light and heat were unable to increase the decomposition rate of HP (24). However, these authors observed that carotene-containing agents considerably increased the temperature of these agents, which is a concern for pulp safety and pulp damage (23,24). According to manufacturer's directions, HP Maxx could have a better performance when light-activated; however, based on the decomposition rate results, HP Maxx directions could change, as no differences were noted among groups regardless of light activation. In addition, it must be pointed out that HP Maxx is a pigment-containing agent and the possibility of a temperature rise reaffirms that there is no need for light activation.

Contrarily, HP Blue was influenced by the light activation method as LED, laser and halogen lamp decrease the HP DR after 5, 10 and 20 min, respectively, while C decreased only after 30 min of bleaching. In a previous study, it was observed that LED or laser irradiation increased the final concentration of 35% HP in the pulp chamber (8), which might indicate a higher decomposition rate when the agent is light activated. However, after 40 min, HP Blue LED and halogen exhibited lower HP rates than laser-activated group, but all groups were similar to C. Since no differences were observed between C and the light-activated groups, we support the manufacturer's recommendation, as no light sources is needed to accelerate bleaching. Moreover, although PO was accelerated by LED and laser sources after 5 min of bleaching, after 10 min, C group lower the HP rate. Therefore, we upkeep PO directions that state that the product can be used without light activation. Based on these results, we reject the second null hypothesis as decomposition rate is influenced by light sources; however, we do not indicate the use of light, as no differences could be observed among light- and non-activated groups at the end of bleaching.

The manufacturers of most in-office bleaching agents indicate three applications of 15 min each. In the current study, the authors observed that one single application of 40 min promotes approximately 15% of the decomposition rate, and most groups reached a final averaged concentration of 20% HP. These results are comparable to those of Marson et al. (17), in which the authors observed that the decomposition rate of 38% HP and 35% HP agents reached 29-33% in one single 45-min application. The authors concluded that there is no need to remove the agent after 15 min and repeat the application, as long as pH is kept neutral (17).

Changes in the protocol of high-concentrated bleaching agents have recently been suggested (13). That investigation

compared different concentrations of HP (17.5%, 35%, or 37%) with distinct application protocols (1 or 3 applications for 5/15 min or 10/20 min for the 37% HP) and concluded that all procedures effectively changed the tooth color, but shortening the contact time or reducing the concentration of the 35% HP gel decreases enamel-dentin diffusion. Therefore, the reduction in the frequency of the application (from 3 to 1 time in one single session) could decrease the amount of product reaching the pulp chamber and the possible side effects promoted by HP in pulp cells, as the amount of HP that diffuses through enamel and dentin is directly related to the toxic effects to cells (13).

Although some differences were observed in the decomposition rate among groups, all of them were able to change enamel color after 40 min of bleaching, even in one single application. It is important to observe that  $\Delta E$  obtained by all the groups exceeded the value for clinical color perception ( $\Delta E > 3.7$ ) (19). Therefore, decrease the application time and frequency of in-office agents without light activation seems to be an effective and possibly safe option, as HP diffusion could be diminished.

Among the high-concentrated agents, HP Blue presented high pH values (approximately 8.0, from the beginning to the end of bleaching), but HP Maxx and PO exhibited pH values below the critical pH value for enamel (mean values of 5.0 and 3.0, respectively). These pH values are a warning that in-office bleaching treatment should be carefully indicated due to the potential enamel mineral loss triggered by bleaching (24), especially with the high frequency of applications (13). For that reason, PO manufacturer's directions to apply the agent for 8 to 20 min is consistent with our findings and HP Blue could be used for up to 40 min; however, preferably both agents in one single application. On the other hand, HP Maxx directions could be reduced to one single 15-20 min application, due to the low pH, decomposition rate and color alteration behavior. Therefore, the third null hypothesis could also be rejected as high-concentrated agents could be applied once for up to 20 (PO and HP Maxx) or 40 min (HP Blue).

Since this is an in vitro assessment, it must be kept in mind that saliva would clinically balance enamel mineral loss (21), but the quality of the surface on which the bleaching agent is applied (enamel with minor fissures or cervical dentin expositions) is an important factor to consider since this could also increase HP diffusion and bleaching sensitivity (13). Future clinical studies should be conducted to assess whether the reduced application time would be able to reach the desired color change and decrease the possibility of tooth sensitivity.

In conclusion, the application mode of in-office and at-home bleaching agents suggested by manufacturers could

decrease without compromising bleaching effectiveness, particularly those with low pH.

## Resumo

Este estudo avaliou a taxa de decomposição (DR), pH, alteração da cor do esmalte ( $\Delta E$ ) e índice de clareamento ( $\Delta WI$ ) promovido por agentes clareadores caseiros e de consultório. A superfície do esmalte foi submetida a ( $n=10$ ): agentes caseiros (10%, 15%, 20% de peróxido de carbamida - CP e 6% de peróxido de hidrogênio - HP) e três agentes a base de HP a 35% com irradiação por luz (LED, laser e halógena) ou sem tratamento (controle). A DR e pH dos agentes foi mensurada após 0, 2, 4, 6 e 8 h (agentes caseiros) e após 5, 15, 20, 30 e 40 min (agentes de consultório). Parâmetros de cor ( $L^*$ ,  $a^*$ ,  $b^*$ , DE e  $\Delta WI$ ) foram determinados inicialmente e após clareamento. Os dados da DR, pH,  $L^*$ ,  $a^*$ ,  $b^*$  foram analisados por ANOVA um fator (agentes caseiros) ou dois fatores (agentes de consultório) de medidas repetidas e teste Tukey.  $\Delta E$  e  $\Delta WI$  por ANOVA um fator (agentes caseiros) e dois fatores (agentes de consultório) e teste Tukey. A DR dos agentes clareadores caseiros foi similar após 6 e 8 h ( $p>0.05$ ), com pH próximo ao neutro (6,5 a 6,9 - CP) ou ácido (5,9- HP 6%). De 4 a 8 h, a  $\Delta E$  foi maior para CP 15% e 20% comparado ao CP 10% ( $p<0.05$ ). Após 40 min, a DR dos agentes HP 35% foi similar e todos exibiram  $\Delta E$  significativa em uma aplicação ( $p<0.05$ ), independente da aplicação de luz.  $\Delta WI$  indicou efeito clareador, sem diferenças entre os grupos ( $p>0.05$ ). Um agente HP 35% exibiu pH alcalino, e os outros,  $pH<5.5$ . Os agentes clareadores caseiros poderiam ser aplicados por 2 h (15%, 20% CP e 6% HP) e 4 h (CP 10%), e os agentes clareadores de consultório, até 40 min em uma aplicação, sem luz.

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