



Titanium dioxide nanotubes in a hydrogen peroxide-based bleaching agent: physicochemical properties and effectiveness of dental bleaching under the influence of a poliwave led light activation

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Abstract

Objectives The effects of different concentrations of titanium dioxide (TiO₂) into 40% hydrogen peroxide (HP) were evaluated as regards the effectiveness of dental color change either associated with activation by polywave LED light or not.

Materials and methods TiO₂ (0, 1, 5, or 10%) was incorporated into HP to be applied during in-office bleaching (3 sessions/40 min each). Polywave LED light (Valo Corded/Ultradent) was applied or not in activation cycles of 15 s (total time of 2 min). The color of 80 third molars separated into groups according to TiO₂ concentration and light activation (*n* = 10) was evaluated at baseline and at time intervals after the 1st, 2nd, and 3rd bleaching sessions.

Results WI_D value was significantly higher when using HP with 5% TiO₂ in the 2nd session than the values in the other groups (*p* < 0.05). After the 2nd and 3rd sessions, the ΔE_{ab} value was significantly higher when activated with light (*p* < 0.05) for all agents containing TiO₂ or not. Zeta potential and pH of the agents were not modified by incorporating TiO₂ at the different concentrations.

Conclusions The 5% TiO₂ in the bleaching agent could enhance tooth bleaching, even without light application. Association with polywave LED light potentiated the color change, irrespective of the presence of TiO₂ in the bleaching gel.

Clinical significance HP with 5% TiO₂ could lead to a greater tooth bleaching response in the 2nd clinical session, as well as the polywave light can enhance color change.

Keywords Dental bleaching · Titanium dioxide · Hydrogen peroxide · Poliwave LED light

Introduction

Smile esthetics has been desired by patients, and dental procedures need to provide the natural characteristics of the teeth, reproducing the shape and esthetics in a

harmonious and beautiful way [1, 2]. From this aspect, the color of the teeth can be changed with the use of dental bleaching techniques, which are performed under the supervision of the dentist, using trays (used at home by the patient with the low concentrations of hydrogen peroxide

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or carbamide peroxide), in-office (applied by the dentist with high concentrations of hydrogen peroxide), or the combined technique (consisting of in-office bleaching associated with the at-home technique) [1, 3]. However, painful symptoms have been reported during the bleaching procedure, in which this sensitivity can be influenced by the time of use, concentration of the bleaching agent, and technique applied [3–6].

With the purpose of accelerating the degradation reaction of the bleaching agent and minimizing the time of exposure of dental tissues to the bleaching agent, titanium dioxide (TiO_2) can be incorporated into bleaching agents to promote electron excitation and generation of oxygen ions, thus generating superoxide that contributes to the breakdown of chromophores present in the dental structure [4, 7, 8]. By incorporating TiO_2 into the composition of bleaching agents, the concentration of hydrogen peroxide can even be reduced (between 3.5 and 23% in different studies [9, 10]), with similar bleaching effectiveness when compared with the use of high concentration agents, and the time of application of the bleaching gel may even be reduced [8–10]. Addition of this catalyst agent to the in-office hydrogen peroxide-based bleaching gel has been used at concentrations of approximately 1% [10–12] and has demonstrated a positive bleaching effect. However, different concentrations of TiO_2 in the bleaching agent have not yet been evaluated, and it is necessary to assess their physicochemical properties on being incorporated into the gel and their influence on the degree of bleaching in the periods between the different clinical sessions.

TiO_2 is considered a photocatalytic agent, as it allows acceleration of the reaction rate when excited by light [13]. It also acts as a catalyst and photocatalyst semiconductor that reacts to blue and violet light [10, 13, 14]. When using a visible light source in the blue–violet range with a wavelength of 405 to 480 nm, it was shown that TiO_2 showed reactivity, suggesting that violet light (in the range of 405 nm) would increase the catalytic reaction of TiO_2 [13].

However, professionals do not always have a specific violet LED light device in their offices to perform bleaching procedures, while the presence of photoactivating devices for polymerization of polywave-type composite resins is more common. Polywave light activation devices have LEDs (light emitting diodes) that emit blue light between 445 and 460 nm, as well as violet light close to 407 nm, and the totality of these emissions leads to the possibility of improved excitation of photoinitiators present in part of the contemporary composite resins [15]. Although this is a light-curing device used by professionals when they perform restorative procedures [16, 17], there are no studies that have evaluated polywave LED light as an activating agent for TiO_2 when it is incorporated into the bleaching agent. Therefore, the association

between the bleaching agent, incorporation of TiO_2 , and the use of polywave LED light could result in requiring a shorter time for performing the in-office technique or even in fewer sessions, which could reduce dentin sensitivity.

Thus, the aim of this study was to evaluate the influence of different concentrations of TiO_2 incorporated into 40% hydrogen peroxide on the physicochemical properties of the bleaching agent and on the color change of dental enamel, either associated with the polywave LED light or not. The following null hypotheses were studied: (H01) the incorporation of TiO_2 at different concentrations would not influence the physicochemical properties of pH, particle size, polydispersity, and zeta potential of 40% hydrogen peroxide; (H02) the incorporation of TiO_2 at different concentrations into 40% hydrogen peroxide would not influence the change in dental color; (H03) activation of 40% hydrogen peroxide with or without polywave LED light would not influence the dental color change.

Materials and methods

Specification of the bleaching agent and the polywave LED, obtainment and incorporation of the TiO_2 nanotubes and incorporation

A bleaching agent containing 40% hydrogen peroxide was used (Opalescence Boost, Ultradent, Salt Lake City, USA); composition by weight according to the Material Safety Data Sheet: Hydrogen peroxide $\leq 40\%$, glycerin $< 12\%$, potassium nitrate $\leq 3\%$, potassium hydroxide $< 3\%$, sodium fluoride 1.1%, and polyethylene glycol $< 1\%$. The TiO_2 nanotubes were incorporated into the different concentrations (1, 5, and 10%) of this bleaching agent.

TiO_2 nanotubes formed by using a single sheet of material wound in a spiral (diameter ~ 10 nm and size ~ 200 nm) were synthesized by the alkaline method [18]. Then, they were weighed on a precision balance with readability of 0.0001 g (Engenharia BEL, Monza, Milan, Italy) and manually added to the recently manipulated bleaching agents at concentrations of 1, 5, and 10%. The incorporation procedure recommended by Monteiro et al. [12] was used and performed by using a plastic spatula for spatulating the products on a sheet of impermeable paper for 1 min. Subsequently, the content obtained was deposited in a sterile, disposable syringe. The TiO_2 was always incorporated into the bleaching agents immediately before each application.

The polywave LED device (Valo, Ultradent Products INC, Utah, USA) was used in standard mode with power of 1000 mW/cm². The device has 3 different colored chips in the single matrix set: two blue (emitting around 460 nm), one blue (emitting around 445 nm), and one violet (emitting around 400 nm) [15].

Physicochemical characterization of gels: evaluation of pH, average particle size, polydispersity, and zeta potential

The pH value of the bleaching agent incorporated into the different concentrations of TiO₂ was measured in triplicate immediately after the manipulation and incorporation of TiO₂, using a table pH meter (MS Tecnoyon Equipamentos Especiais LTDA, RbPH-210, Piracicaba, SP, Brazil).

The mean size of the bleaching gel particles with or without the addition of TiO₂ nanotubes, polydispersity (which measures the heterogeneity of the molecule or particles in the solution), and zeta potential (colloidal stability) was evaluated by dynamic light scattering (Zetasizer Nano ZS, Malvern Instruments, Malvern, UK) in triplicate at 25 °C, immediately after manipulation and incorporation of TiO₂ [12]. The zeta potential was evaluated to measure the colloidal stability, using the Helmholtz–Smoluchowski [19] model, to measure the electrophoretic mobility of the dispersed particles in the electric field applied. The electrostatic potential of particles directly related to their dispersion stability by the electrostatic repulsion. Therefore, when the potential was small, attractive forces could exceed this repulsion and the dispersion could break and flocculate [20].

Zeta potential analysis was performed by laser electrophoresis with 30 runs per measurement at 25 °C. Zeta potentials were automatically calculated based on the electrophoretic mobility, using the Smoluchowski approximation: $UE = 2 * \epsilon * z * f(ka) / 3 * \eta \rightarrow z \approx UE * \eta / \epsilon$, where UE was the electrophoretic mobility, ϵ was the dielectric constant, z was the ZP, f(ka) was the function of Henry, and η was the viscosity.

Preparation of teeth and application of bleaching treatments

The sample size calculation was dimensioned with the use of G*Power program [21] considering the experimental design (TiO₂ concentration, presence of light, and time). Eighty teeth randomly distributed among 8 groups ($n = 10$) provided a power of 0.80 ($\beta = 0.20$) for a level of significance of 5% ($\alpha = 0.05$) and large effect size for TiO₂ concentration and presence of light (effect size > 0.35), and average effect size (effect size > 0.20) for time and interactions among factors under study.

After approval by the Research Ethics Committee (CAAE number 12317519.7.0000.5374), 100 freshly extracted healthy human third molars were selected. The teeth were kept frozen until the beginning of the experiment, when they were cleaned with a scalpel blade and submitted to prophylaxis with pumice stone and Robinson brush. All teeth were evaluated throughout their full length, under a stereoscopic magnifying glass (Eikonal Equipamentos

Óticos e Analíticos, model EK3ST, São Paulo, SP, Brazil) with 20× magnification. Any teeth with cracks, wear, and/or fractures were discarded.

The teeth were placed in PVC molds 20 mm in diameter and 20 mm high, and the root portion, up to the cemento-enamel limit, was fixed in the mold with self-curing acrylic resin (VipiFlash, Vipi, Pirassununga, SP, Brazil). The initial color of teeth was then evaluated by means of a spectrophotometer (VITA Easyshade® Advance, Vita, Germany), using the Vita Classical scale as a criterion. Teeth with very light or very dark colors were excluded from the study, so that 80 teeth that had different colors (A3, A3.5, A4, B2, B3, and B4) were selected for homogeneous distribution among the eight groups under study ($n = 10$) according to the percentage of TiO₂ incorporated (absent, 1, 5, and 10%) and use of polywave LED light (absence or presence of light).

Subsequently, the teeth were kept individually immersed in Eppendorf flasks containing artificial saliva for 7 days, as recommended by Featherstone et al. [22] and modified by Serra & Cury [23] composed of (1.5 mMol/L Ca; 50 mMol/L KCl; 0.9 mMol/L PO₄; 20 mMol/L Tris Buffer; $pH = 7$). For the application of the treatments, the teeth were removed from the saliva and dried with air jets for 5 s. An amount of 0.03 mL of the bleaching gel corresponding to each group was applied on the buccal surface of the teeth by means of a syringe. The bleaching treatment was performed in three clinical sessions (40 min each) with an interval of 7 days between sessions.

In the groups in which treatment was associated with the polywave LED light, this light was turned on immediately after application of the gel, then applied in light-activation cycles of 15 s, waiting for 5 min, new light-activation for 15 s and so on until 40 min of bleaching treatment were concluded. Therefore, when adding up the light-activation times, each tooth received a total activation time of 2 min distributed in the different time intervals in each session. This protocol was based on the study by Kishi et al. [13] which proved the effect of TiO₂ photocatalysis on enamel color change when associated with blue LED light devices (7 blue LEDs — 470 nm) and 1 violet LED (405 nm) with a power of 650 mW/cm² and application time of 1 min while the bleaching agent was left on the specimens for 5 min. As no other study had been employed polywave LED light to perform bleaching treatment, our protocol was also based on and adapted from other studies that used LED curing units, as those shown by Torres et al. [24] — who applied different types of LED light for a total time of 5 min with intervals of 1 min each — and by Ferreira et al. [25] who applied blue LED light (470 nm) and green LED light (430 nm) for 3 min associated with bleaching with 35% hydrogen peroxide. On conclusion of the recommended time, the gel was sucked up with an aspirator cannula, and the teeth were cleaned with gauze and washed with water for 10 s and then immersed in

an artificial saliva solution for a period of 7 days, with the solution being changed every 2 days.

Color evaluation

The teeth were evaluated by means of a spectrophotometer (Easyshade® Advance, Vita, Germany) to analyze the color according to the parameters of the Vita Classical scale and CIEL*a*b* (L^* = luminosity; a^* = green–red axis; b^* = blue–yellow axis). The color was evaluated in the following time intervals: initial (baseline); 7 days after the first bleaching session (after 1st session); 7 days after the second bleaching session (after the 2nd session); 7 days after the third bleaching session (after the 3rd session). For color evaluation, the teeth were placed in a box with a white background in order to standardize lighting. The procedure was always performed by the same operator and in duplicate, in each time interval, to ensure accuracy.

The tooth color obtained according to the Vita Classical scale was converted into numbers according to previously established numerical values [12], by organizing them from number 1 (B1 shade) to 16 (C4) in order of luminosity: B1, A1, B2, D2, A2, C1, C2, D4, A3, D3, B3, A3.5, B4, C3, A4, and C4. Thus, the lower the numerical value, the greater the luminosity and the lighter the tooth appeared to be.

Color changes were evaluated according to the CIEL*a*b* parameter, and the values of ΔL^* , Δa^* , and Δb^* were obtained for each group and study period, in which the color change (ΔE_{ab}) was evaluated and estimated by formula [26]: $\Delta E_{ab} = \sqrt{((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)}$. The perceptibility and acceptability limits considered for ΔE_{ab} were 1.2 and 2.7, respectively [26, 27]. The color change was also evaluated by CIEDE2000 (ΔE_{00}), which uses the h (hue) and C (chroma) values [28]. ΔE_{00} values of 0.8 and 1.8 were adopted as the perceptibility and acceptability limits [26]. Monitoring of the dental bleaching was evaluated by the Whiteness Index for Dentistry (WI_D), in which the parameters L^* , a^* , and b^* were used in the following equation [29]: $WI_D = 0,511L^* - 2,324a^* - 1,100b^*$. Differences in WI_D between the initial and final assessments were evaluated (ΔWI_D), using threshold values for ΔWI_D of 0.72 for perceptibility and 2.6 for acceptability [30].

Statistical analysis

Descriptive and exploratory analyses of all color change variables were performed. Exploratory analyses indicated that the variables did not meet the assumptions of an analysis of variance (ANOVA). The pH and zeta potential data were analyzed by the non-parametric tests of Kruskal–Wallis and Dunn. Particle size and polydispersity data were analyzed by generalized linear models. As regards the color analysis, the a^* data did not fit a known distribution and were analyzed

by the Kruskal–Wallis and Dunn non-parametric tests for comparisons between TiO_2 concentrations; Mann–Whitney for comparisons between groups with and without light activation; and Friedman and Nemenyi tests for comparisons among time intervals. For L^* , b^* , WI_D , ΔE_{ab} , ΔE_{00} , and ΔWI_D , generalized linear models were used, considering an asymmetric distribution, considering the factors under study “ TiO_2 concentration” and “activation with light” with repeated measures in time, as well as the interactions between them. The analyses were performed using the R Core Team (2021) program, with a significance level of 5%.

Results

The pH values of the bleaching agents (Table 1) show that the incorporation of 1% TiO_2 led to significantly higher pH values than when 10% was incorporated. The higher percentage of TiO_2 led to a reduction in pH; however, even with the incorporation of 10%, the pH of all agents remained close to neutrality and without significant differences in comparison with the gel without TiO_2 . The particle size was significantly larger in the product with 10% TiO_2 , and only the presence of 1% TiO_2 did not influence the particle size of the product in comparison with that of the bleaching agent without its incorporation. Polydispersity was significantly higher in the absence of TiO_2 than in its presence. The zeta potential was significantly less negative with 10% than with 5%, with no significant differences between groups.

Irrespective of activation with or without LED light, there was no significant difference between bleaching agents with different concentrations of TiO_2 for the Vita Classical, L^* , and a^* scale ($p > 0.05$) (Table 2). For b^* , 5% TiO_2 incorporated into the bleaching agent showed lower values than those of the other concentrations, but without significant differences in comparison with the agent without incorporation throughout the course of the bleaching sessions. For WI_D , after the second clinical session, 5% TiO_2 led to higher values than the bleaching agent without incorporation, but there were no differences between the groups after the third bleaching session. When activated with LED light, all bleaching agents with or without incorporation of TiO_2 had higher L^* values than those without light-activation at all time intervals ($p = 0.0172$). For all bleaching agents whether they contained TiO_2 or not, there was a significant decrease in the colors of the Vita Classical scale, a^* , and b^* throughout the clinical bleaching treatment sessions, with significant differences in comparison with baseline ($p < 0.05$). There was also a significant increase in L^* and WI_D values in all groups after 3rd bleaching session compared with baseline ($p < 0.05$).

For b^* , there was no significant difference between groups at baseline ($p > 0.05$). There was no significant difference between

Table 1 pH, particle size, polydispersity, zeta potential, and pH of the bleaching product with different concentrations of TiO₂

Variable	TiO ₂	Mean (standard deviation)
pH	Absent	7.38 (0.02) ab
	1%	8.01 (0.08) a
	5%	7.27 (0.02) ab
	10%	6.97 (0.02) b
	<i>p</i> -value	0.0148
Particle size	Absent	515.93 (± 141.11) ^c
	1%	485.63 (± 44.93) ^c
	5%	836.73 (35.29) b
	10%	1043.67 (29.87) a
	<i>p</i> -value	< 0.0001
PDI (polidispersibility)	Absent	0.67 (0.02) a
	1%	0.49 (0.09) b
	5%	0.42 (0.07) b
	10%	0.41 (0.04) b
	<i>p</i> -value	0.0008
Zeta potential (mV)	Absent	− 29.63 (2.21) ab
	1%	− 32.87 (0.47) ab
	5%	− 44.53 (0.40) b
	10%	− 18.87 (0.72) a
	<i>p</i> -value	0.0156

Different letters indicate statistically significant difference ($p \leq 0.05$)

groups at baseline time intervals for WI_D ($p < 0.05$). However, after the 1st clinical bleaching session, the WI_D was significantly higher for bleaching agents with 1% and 5% TiO₂ than those with 10% ($p < 0.05$). After the 2nd clinical bleaching session, the WI_D was significantly higher with 5% TiO₂ than the other groups ($p < 0.05$), while after the 3rd bleaching session, the WI_D was significantly higher with 5% TiO₂ than with 10% ($p < 0.05$), with no differences in comparison with the group without incorporation or with 1% TiO₂.

At all assessment time intervals, there were no significant differences between groups relative to ΔE_{ab} ($p > 0.05$) (Table 3). However, in the assessment time intervals after the 2nd session ($p = 0.0280$) and 3rd bleaching session ($p = 0.0272$), the ΔE_{ab} value was significantly higher in groups with light activation, irrespective of the concentration of TiO₂ either incorporated into the bleaching agent or not. There was no significant difference between the groups regarding ΔE_{00} and ΔWI_D ($p > 0.05$) irrespective of activation with or without LED light.

Discussion

Modifications in the composition of bleaching agents could favor a reduction in adverse effects on mineralized dental substrates, pulp, and periodontal tissues, as well as

potentiate the beneficial effects with regard to color change. From this aspect, the incorporation of TiO₂ leads to the possibility of decreasing the concentration of the bleaching agent, especially when it is associated with activation by diode laser light, with effective bleaching being obtained [11], or even reducing the time of the bleaching treatment by a reduction of the number of the sessions. The higher generation of hydroxyl radicals may also result from the incorporation of TiO₂ into the bleaching agents, especially because it affects the physicochemical properties of the bleaching agent, which led to rejection of the first null hypothesis of the present study. The presence of 1% of TiO₂ in the bleaching agent was able to raise the pH from 7.38 to 8.01; however, there was no statistically significant difference, and the pH of the bleaching agent became slightly alkaline, which may have had an impact on the higher rate of release and speed of free radicals during the oxidation–reduction reaction of the bleaching agent [24, 31]. The increase in pH is capable of accelerating the rate of degradation of hydrogen peroxide, triggering the release of free radicals that promote improvement in the effectiveness of bleaching [24, 31–33]. When 5% TiO₂ was added to the pH, the pH remained practically the same (7.27) as that of the bleaching gel without its incorporation (7.38). However, a higher concentration of TiO₂ (such as the 10% used) led to a decrease in the pH of the bleaching agent (6.97), which could, to a certain extent, reduce the release of reactive free radicals and affect bleaching efficacy [24, 34].

When using a concentration of 1% of TiO₂ incorporated into the 40% hydrogen peroxide bleaching agent, Monteiro et al. [12] observed no differences in the effectiveness of tooth enamel bleaching, suggesting that other concentrations should be evaluated. From this aspect, although the present study showed that there were no differences between the concentrations of TiO₂ incorporated into the bleaching agent when evaluating the color evaluation parameters with reference to the Vita Classical scale, L*, and a*, both in the presence and absence of light, differences could be observed between the parameters b* and WI_D, which led to rejection of the second hypothesis of the present study. The concentration of 5% TiO₂ led to lower b* values after the first clinical session compared with the other concentrations, which means that there was a higher degree of degradation of yellow pigments present in the tooth at the time of evaluation. Evaluation of the b* parameter has been pointed out as being an important factor in the evaluation of color changes during tooth bleaching because it is the parameter that has a faster and more extensive influence on color than that of the other components of the CIELab system [35], whereas it was only when evaluating the WI_D parameter that a significant difference in the presence of 5% TiO₂ was observed in comparison with the control group (bleaching agent without

Table 2 Median (minimum value; maximum value) for Vita Classical shade guide and a* and mean (standard deviation) for L*, b*, and WI_D according to concentration of TiO₂, activation with LED light, and time of evaluation

Variable	Activation with light	TiO ₂	Time				
			Baseline	After 1st bleaching session	After 2nd bleaching session	After 3rd bleaching session	
Vita classical	Absent	Absent	11 (3; 15) Aa	6 (2; 13) ABa	6 (2; 11) Ba	3 (2; 11) Ba	
		1%	11 (3; 13) Aa	4 (2; 9) ABa	3 (2; 11) Ba	3 (2; 3) Ba	
		5%	11 (3; 12) Aa	3 (2; 11) ABa	3 (1; 3) Ba	3 (2; 3) Ba	
		10%	11 (3; 12) Aa	10 (2; 11) ABa	7 (2; 11) Ba	4 (2; 11) Ba	
	<i>p</i> -value		0.9999	0.1370	0.0628	0.2600	
	Present	Absent	11 (3; 13) Aa	3 (2; 9) ABa	3 (2; 3) Ba	2 (1; 3) Ba	
		1%	11 (3; 12) Aa	3 (2; 11) Ba	3 (1; 9) Ba	* 3 (2; 11) Ba	
		5%	11 (3; 12) Aa	3 (2; 9) ABa	2 (2; 3) Ba	* 2 (2; 3) Ba	
		10%	11 (3; 12) Aa	9.7 (2;11) ABa	3 (2; 11) Ba	3 (2; 11) Ba	
	<i>p</i> -value		0.9999	0.1400	0.1880	0.1280	
	L*	Absent	Absent	80.76 (3.89) Ca	84.66 (1.70) Ba	85.20 (2.62) Ba	86.58 (2.04) Aa
			1%	80.46 (4.29) Ba	85.42 (2.48) Aa	84.65 (2.49) Aa	86.40 (3.25) Aa
			5%	80.20 (5.79) Da	84.40 (1.97) Ca	85.34 (1.98) Ba	86.10 (2.80) Aa
			10%	81.69 (3.30) Ca	81.82 (6.16) Ba	84.55 (3.55) Aa	85.07 (4.05) Aa
Present		Absent	* 81.06 (2.79) Ca	* 85.67 (1.76) Ba	* 85.32 (2.37) Ba	* 87.90 (1.28) Aa	
		1%	* 80.59 (2.67) Ba	* 86.02 (1.85) Aa	* 87.21 (1.77) Aa	* 86.96 (1.73) Aa	
		5%	* 81.65 (3.85) Da	* 85.82 (2.37) Ca	* 87.91 (2.41) Ba	* 88.26 (2.01) Aa	
		10%	* 80.45 (2.77) Ca	* 84.89 (3.10) Ba	* 86.28 (2.58) Aa	* 87.12 (3.93) Aa	
<i>p</i> -value			p(TiO ₂)=0.7053; p(Light)=0.0172; p(TiO ₂ ×Light)=0.8090; p(Time)<0.0001; p(TiO ₂ ×Time)=0.0615; p(Light×Time)=0.2809; p(TiO ₂ ×Light×Time)=0.2762				
a*		Absent	Absent	-0.42 (-2.53; 1.73) Aa	-1.70 (-3.10; -0.27) Aa	-1.93 (-2.83; -1.27) ABa	-2.48 (-3.43; -1.87) Ba
			1%	-0.12 (-1.63; 0.97) Aa	-1.98 (-2.47; -0.37) ABa	-2.50 (-3.20; -1.73) BCa	-2.87 (-3.50; -1.47) Ca
			5%	-0.57 (-2.17; 1.87) Aa	-2.23 (-2.87; -0.47) ABa	-2.53 (-3.17; -1.30) Ba	-2.83 (-3.73; -1.60) Ba
			10%	-0.12 (-3.23; 2.83) Aa	-1.32 (-3.03;0.00) ABa	-2.12 (-3.33; -0.33) Ba	-1.97 (-3.47; -0.60) Ba
		<i>p</i> -value		0.9788	0.5030	0.4780	0.2140
	Present	Absent	0.02 (-1.63; 2.77) Aa	-1.93 (-2.43; -1.27) ABa	-2.38 (-2.70; -1.53) BCa	-2.62 (-3.23; -1.07) Ca	
		1%	-0.77 (-2.63; 3.30) Aa	-1.82 (-3.30; -0.37) ABa	-2.08 (-3.40; -1.27) Ba	-2.50 (-3.20; -1.37) Ba	
		5%	-0.18 (-2.90; 2.57) Aa	-1.93 (-3.47; -0.80) ABa	-2.43 (-3.50; -1.57) BCa	-2.77 (-3.60; -1.93) Ca	
		10%	0.47 (-1.77; 2.23) Aa	-1.17 (-3.07;0.80) ABa	-1.80 (-3.03; -0.50) BCa	-2.22 (-3.57; -0.23) Ca	
	<i>p</i> -value		0.8843	0.3503	0.4254	0.5837	
	b*	Absent	Absent	30.08 (6.48) Aa	24.83(6.04) Bab	22.93 (5.37) Cab	21.91 (5.70) Dab
			1%	27.27 (2.86) Aa	23.30 (1.76) Ba	22.04 (2.45) Ca	20.47 (1.55) Da
			5%	27.44 (3.64) Aa	21.60 (2.37) Bb	19.92 (1.82) Cb	19.99 (1.73) Db
			10%	29.22 (4.58) Aa	24.55 (3.28) Ba	22.72 (3.15) Ca	22.39 (3.08) Da
Present		Absent	29.97 (6.96) Aa	21.71(2.53) Bab	19.68 (2.30) Cab	18.27 (3.34) Dab	
		1%	29.17 (5.13) Aa	22.72 (3.05) Ba	21.59 (3.19) Ca	20.88 (4.40) Da	
		5%	28.67 (3.39) Aa	21.43 (2.40) Bb	19.29 (2.83) Cb	17.98 (2.51) Db	
		10%	29.33 (5.51) Aa	24.48 (4.32) Ba	22.02 (4.66) Ca	21.18 (4.86) Da	
<i>p</i> -value			p(TiO ₂)=0.0525; p(Light)=0.1524; p(TiO ₂ ×Light)=0.5892; p(Time)<0.0001; p(TiO ₂ ×Time)=0.1189; p(Light×Time)=0.0155; p(TiO ₂ ×Light×Time)=0.3846				

Table 2 (continued)

Variable	Activation with light	TiO ₂	Time			
			Baseline	After 1st bleaching session	After 2nd bleaching session	After 3rd bleaching session
WI _D	Absent	Absent	9.08 (11.00) Da	19.79 (8.92) Cab	23.01 (7.65) Bb	26.06(7.59) Aab
		1%	11.75 (6.44) Da	22.27 (3.83) Ca	24.36 (4.71) Bb	28.04(3.13) Aab
		5%	11.61 (8.07) Da	23.94 (4.39) Ca	27.25 (2.85) Ba	28.17 (3.51) Aa
		10%	9.92 (9.79) Da	18.20 (6.64) Cb	22.89 (5.68) Bb	23.29 (6.20) Ab
	Present	Absent	8.20 (10.30) Da	24.30 (3.69) Cab	27.19 (3.92) Bb	30.56(4.69) Aab
		1%	9.30 (10.02) Da	23.29 (5.44) Ca	26.01 (5.39) Bb	27.04(6.48) Aab
		5%	10.59 (8.37) Da	25.04 (3.29) Ca	29.53 (3.40) Ba	31.67 (3.05) Aa
		10%	8.06 (9.64) Da	19.31 (8.02) Cb	24.23 (7.43) Bb	26.13 (8.85) Ab
<i>p</i> -value		p(TiO ₂)=0.6382; p(Light)=0.5413; p(TiO ₂ × Light)=0.9405; p(Time) < 0.0001; p(TiO ₂ × Time)=0.0383; p(Light × Time)=0.8492; p(TiO ₂ × Light × Time)=0.4811				

Different letters (capital letters) in the horizontal line show comparisons between times and lowercase letters in the vertical column show comparisons between concentrations of TiO₂, within each category of light, and indicate statistically significant differences ($p \leq 0.05$)

TiO₂) after the second clinical bleaching session. From this aspect, considering that the WI_D parameter was developed with the aim of establishing a strong correlation with the perception of tooth bleaching [29], in addition to demonstrating superior performance when assessing the degree of perceptible bleaching in comparison with other indices [29], the suggestion could be that among all the treatments evaluated, a higher bleaching index could be obtained in the second session when using the bleaching agent with 5% TiO₂ incorporated into it. This could be an advantage in cases when the patient does not require or does not wish to perform a third tooth bleaching session. These results could suggest that 5% TiO₂ might have potentiated the bleaching effect of the hydrogen peroxide used, which may have increased the degradation reaction of the bleaching agent and contributed to the breakdown of chromophores present in the tooth structure [4, 7, 8]. However, with the need for a 3rd clinical session, no significant difference was observed between the treatments evaluated, irrespective of whether or not they contained TiO₂ in the composition.

Despite the bleaching agent into which a concentration of 5% TiO₂ was incorporated having a pH similar to the bleaching agent without any incorporation, the former caused a significant increase in the average size of particles present in the material. This also occurred with the incorporation of a concentration of 10%. The incorporation of different concentrations of TiO₂ into the bleaching agents produced gels with good viscosity and handling characteristics, but a change in the original reddish color of the gel was observed when using the concentration of 10% TiO₂, as well as more thickening of the gel and significant presence of bubbles in it. The increase in the mean particle sizes in the bleaching

agent may be related to the rheological characteristics of the material, considering that despite the use of nanometric particles in the tubular conformation of TiO₂, their incorporation led to a change in the characteristics of polydispersity and colloidal stability of the bleaching agent. The particles used had sizes in the range from 0.1 to 100 nm. The purpose was to make the product lighter and improve its properties [36] with the use of anatase particles, especially due to their photochemical properties [18], thereby promoting an increase in surface area and quantum effects.

The polydispersity index is defined by the distribution of particles in the material, with lower values indicating uniformity of dispersion [37]. From this aspect, all TiO₂ concentrations improved the polydispersity characteristics, with a lower probability of agglomeration forming in the bleaching gel. However, it was only at the 5% concentration that the zeta potential values of the bleaching agent reached “good colloidal stability” (−44.53 mV), unlike the 10% concentration, which obtained “incipient stability” (−18.87 mV). The zeta potential is an important indicator of the stability of colloidal dispersions. For molecules and particles that are small enough, a high zeta potential will confer stability, i.e., the solution or dispersion will resist aggregation. Therefore, solutions or colloids with high zeta potential (negative or positive) are electrically stabilized — leading to greater material stability — while colloids with low zeta potential tend to coagulate or flocculate [20, 38].

The association of the use of a light source during the bleaching treatment has been recognized as a factor that leads to an increase in temperature, which can accelerate the decomposition reaction of hydrogen peroxide [39] and could impact the reduction in bleaching treatment time. However,

several systematic reviews have shown that activating the bleaching gel in in-office treatment with different types of lights and protocols does not seem to lead to greater effectiveness in color change [39–43], with inconclusive answers regarding tooth sensitivity, and some reviews reporting a decrease [42], while others reported that sensitivity was not affected by the use of lights [39, 43]. These differences may occur due to the sensitivity assessment criteria, protocol, and type of light used (halogen, LED or laser) [39, 41, 42]. Studies with the use of polywave LED light associated with bleaching treatment have not yet been conducted, and the present study demonstrated that the activation of the bleaching agent with this type of light promoted greater luminosity, irrespective of the presence and concentration of TiO₂ in the

composition of the bleaching agent, leading to the rejection of the third null hypothesis of the present study.

Although the safety of the pulp temperature that can be reached in the tooth when using the light-application protocol has not yet been recognized, the suggestion could be that the increase in the temperature of the bleaching gel may have led to greater degradation of hydrogen peroxide, causing a higher level of bleaching effectiveness, which impacted the color change values obtained by ΔE_{ab} after the second and third tooth bleaching sessions. In fact, Rotstein et al. [44] showed that when increasing the temperature from 24 to 37 °C, this doubles the amount of hydrogen peroxide free radicals that reach the pulp, thereby even further increasing the penetration into the dental substrate when a temperature

Table 3 Mean (standard deviation) of the color change, in comparison with baseline, for the variables ΔE_{ab} , ΔE_{00} e ΔWI_D according to concentration of TiO₂, activation with LED light, and time

Variable	Activation with light	TiO ₂	Time		
			After 1st bleaching session	After 2nd bleaching session	After 3rd bleaching session
ΔE_{ab}	Absent	Absent	7.63 (3.36) a	8.92 (3.94) a	10.61 (4.21) a
		1%	7.24 (3.10) a	8.62 (2.61) a	10.46 (3.46) a
		5%	8.02 (4.24) a	10.14 (5.00) a	10.36 (5.05) a
		10%	7.25 (2.92) a	8.46 (2.71) a	9.33 (3.21) a
	Present	Absent	10.4 (5.78) a	* 11.62 (6.06) a	* 14.44 (5.99) a
		1%	9.06 (3.12) a	* 10.58 (3.37) a	* 11.18 (2.33) a
		5%	9.17 (4.76) a	* 11.80 (5.08) a	* 13.16 (5.10) a
		10%	7.26 (2.70) a	* 9.97 (2.18) a	* 11.37 (3.78) a
<i>p</i> -value		p(TiO ₂)=0.5920; p(Light)=0.1327; p(TiO ₂ ×Light)=0.8130	p(TiO ₂)=0.5277; p(Light)=0.0280; p(TiO ₂ ×Light)=0.9652	p(TiO ₂)=0.526; p(Light)=0.0272; p(TiO ₂ ×Light)=0.8610	
ΔE_{00}	Absent	Absent	4.42 (1.84) a	4.98 (1.94) a	6.12 (2.25) a
		1%	4.45 (2.02) a	5.33 (1.71) a	6.49 (2.28) a
		5%	4.78 (3.17) a	6.07 (3.50) a	6.32 (3.57) a
		10%	4.38 (2.13) a	4.82 (1.35) a	5.40 (1.77) a
	Present	Absent	5.58 (2.39) a	6.21 (2.24) a	8.06 (2.41) a
		1%	5.19 (1.62) a	6.17 (1.95) a	6.52 (1.41) a
		5%	5.24 (2.60) a	6.78 (2.92) a	7.56 (2.92) a
		10%	4.20 (1.40) a	5.79 (1.07) a	6.66 (2.26) a
<i>p</i> -value		p(TiO ₂)=0.7429; p(Light)=0.3337; p(TiO ₂ ×Light)=0.8504	p(TiO ₂)=0.4175; p(Light)=0.0528; p(TiO ₂ ×Light)=0.9504	p(TiO ₂)=0.615; p(Light)=0.0666; p(TiO ₂ ×Light)=0.7554	
ΔWI_D	Absent	Absent	10.71 (5.82) a	13.93 (6.27) a	16.98 (6.33) a
		1%	10.52 (5.69) a	12.61 (6.12) a	16.29 (5.88) a
		5%	12.33 (5.25) a	15.64 (7.11) a	16.56 (7.29) a
		10%	8.27 (6.48) a	12.96 (8.07) a	13.37 (9.42) a
	Present	Absent	16.09 (8.17) a	18.99 (8.02) a	22.36 (8.47) a
		1%	13.98 (5.98) a	16.70 (6.33) a	17.74 (5.08) a
		5%	14.45 (8.90) a	18.94 (8.94) a	21.08 (9.01) a
		10%	11.25 (6.01) a	16.17 (4.67) a	18.06 (6.95) a
<i>p</i> -value		p(TiO ₂)=0.6621; p(Light)=0.0892; p(TiO ₂ ×Light)=0.9187	p(TiO ₂)=0.8555; p(Light)=0.0656; p(TiO ₂ ×Light)=0.9023	p(TiO ₂)=0.855; p(Light)=0.0684; p(TiO ₂ ×Light)=0.9387	

*Differs from group without activation with LED light in the same conditions of concentration of TiO₂ ($p \leq 0.05$). Different letters in the vertical column compare the concentrations of TiO₂ in the same conditions of light and indicate statistically significant differences ($p \leq 0.05$)

of 47 °C is reached. It is possible that the protocol used did not lead to reaching this high temperature, even more so considering the fact that 15 s of activation were applied at 5-min intervals, which is shorter than the time used to clinically light-activate an increment of composite resin [24, 28]. Other protocols use continuous application for 1 min of blue/violet LED light at 650 mW/cm² [13] or halogen light at 700 mW/cm² [24], violet LED at 140.2 mW/cm² [45] or up to 3 min with green LED at 180 mW/cm² [25]. However, the use of polywave LED light was shown to be safe in vivo relative to intrapulpal temperature during the light-activation of composite resin in class V cavities for the time of 60 min since the temperature obtained was close to 5.5 °C (with power of 1244 mW/cm²) [46]. Nevertheless, studies are being conducted for better evaluation, since light can potentiate the heating of the gel, which can perpetuate the increase in the temperature of the dental substrate due to its permanence on the tooth, which could increase the temperature of the pulp and, clinically, result in higher degrees of sensitivity.

However, the significantly greater effectiveness of color change promoted by activation with LED light after the second and third session in the ΔE_{ab} parameter had no impact when the threshold values of acceptability and perceptibility were observed. Even after the first tooth bleaching session, the values obtained for the parameters of color change for ΔE_{ab} [26, 27], ΔE_{00} [27], and ΔWI_D [30] were found to be much higher than the limits of perceptibility and acceptability, denoting that, irrespective of the association with light, presence and concentration of TiO₂ in the composition, the bleaching obtained was clinically perceptible and acceptable.

According to Joiner & Leo [47] and Lilaj et al. [48], the WI_D parameter showed better correlation with visual perception data than all other parameters tested, both in studies under laboratory and clinical conditions, allowing changes to be observed in the initial and final time intervals of bleaching, a fact also observed in the present study, which may demonstrate that the WI_D value was significantly higher when using a bleaching agent containing TiO₂, especially at a concentration of 5% in the 2nd bleaching session, than in the other groups studied. Therefore, 5% TiO₂ incorporated into the bleaching agent was capable of enhancing tooth bleaching even without light application, when using the WI_D parameter.

The present study allowed the conclusion that the pH and zeta potential of the bleaching agent were not modified when TiO₂ at different concentrations was incorporated into the bleaching agent, but it reduced the polydispersity of the gel. There was an increase in the particle size of the bleaching agent when concentrations of 5 and 10% TiO₂ were incorporated into the bleaching agent. The association of a concentration of 5% TiO₂ into a bleaching agent containing 40% hydrogen peroxide could lead to a greater tooth bleaching

response in the second clinical session, which could be an advantage when the patient does not require or does not wish to perform a third dental bleaching session. The association of polywave LED light potentiated the color change only for the ΔE_{ab} parameter, irrespective of the presence of TiO₂ in the bleaching gel. However, the color changes were noticeable and acceptable for all evaluation parameters, irrespective of activation with polywave LED light, presence and concentration of TiO₂ in the bleaching agent composition.

Author contribution Edina Veloso Gonçalves Antunes and Rosanna Tarkany Basting were responsible for developing the experiment, discussion of the results, and writing the paper. Flávia Lucisano Botelho do Amaral, Fabiana Mantovani Gomes França, Kamila Rosamilia Kantovitz, and Cecilia Pedroso Turssi interpreted the results and wrote the paper. Erika Soares Bronze-Uhle and Paulo Noronha Lisboa Filho developed and provided the TiO₂. Roberta Tarkany Basting developed the experimental design, wrote the paper, and helped with the discussion.

Data Availability Authors can confirm that all relevant data are included in the article and other supplementary information are available from the corresponding author upon reasonable request.

Declarations

Competing interests The authors declare no competing interests.

Ethical approval All procedures performed in studies involving human participants were in accordance with the ethical standards of the institutional and national research committee and with the 1964 Helsinki Declaration and its later amendments or comparable ethical standards.

Informed consent Informed consent was obtained from all individual participants included in the study.

Conflict of interest The authors declare no competing interests.

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