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ORIGINAL ARTICLE

Frequency doubled neodymium:yttrium–aluminum–garnet and diode laser-activated power bleaching—pH, environmental scanning electron microscopy, and colorimetric in vitro evaluations

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Abstract Corrosiveness of enamel surfaces of Smartbleach®, Opus White®, Opalescense Xtra Boost® and a gel containing titanium dioxide (TiO_2) particles, activated either by a frequency doubled neodymium: yttrium–aluminum–garnet (Nd:YAG) laser (532 nm) or a diode laser (810 nm) was evaluated by environmental scanning electron microscopy (ESEM). Changes in teeth color shades and the pH were also evaluated. Each bleaching agent was laser activated for 30 s and removed after 1 min or 10 min. This procedure was repeated up to four times, the bleaching agent receiving a maximum application time of 40 min, with total irradiation times of 0.5 min to 2 min of laser activation. The results of the pH measurements showed that only Smartbleach® was in the alkaline pH range, whereas the other three were acidic. The surface effects were unrelated to the pH of the bleaching agents. With the exception of Opus White®, no severe alterations on the enamel surface were detected. Although short application times were chosen, improved changes in brightness of up to ten steps on the Vitapan® classical shade guide were detected.

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Keywords Bleaching agents · Laser power bleaching · Morphology changes · pH value · Whitening

Introduction

The use of laser energy is a relatively novel approach for teeth whitening and presents some advantages over most available over-the-counter, home, and in-office bleaching products. The procedure can be completed with a single in-office treatment and allows one to focus on a single tooth or even a selected part of a tooth. The choice of the wavelength is based on the light–target tissue relationship. The bleaching gel, on the one hand, should absorb the light, and the tooth structure, on the other hand, should be minimally affected. Therefore, photo-initiators or dyes are incorporated, which are adjusted to absorb the wavelength of the light source used [1]. This photo-thermal bleaching effect is used by diode (810 nm or 980 nm) and neodymium: yttrium–aluminum–garnet (Nd:YAG) (1,064 nm) lasers. The carbon dioxide (CO_2) laser (10,600 nm) radiation is readily absorbed within approximately 0.1 mm of water-based solutions independently of any absorber. This rapid absorption heats the bleaching agent more quickly than does a conventional heat source, so that the pulp is purportedly not affected [2]. High-intensity green laser light has, additionally, a photochemical effect, which relies upon specific absorption of a narrow spectral range of green light (510–540 nm) into chelate compounds formed between apatite, porphyrin, and tetracycline compounds [3]. The argon ion laser (514.5 nm) and the frequency doubled Nd:YAG laser (2ωNd:YAG laser at 532 nm) can be used for photochemical bleaching, since their wavelengths approximate the absorption maxima of these chelated compounds (525–530 nm) [4]. These green-

light emitting lasers can achieve a positive result in cases that are completely unresponsive to conventional photo-thermal power bleaching [5].

There are serious concerns about the safety of conventional hydrogen peroxide-containing bleaching products. Alterations of the surface texture of enamel, including shallow depressions, increased porosity, and slight erosion, have been reported, via the use of scanning electron microscopy [6, 7]. The prism layers to the depth of the enamel rods are exposed and possibly extend into the dentin [8].

The critical pH for enamel demineralization to occur is between 5.2 and 5.8 [9, 10]. Nevertheless, in a laboratory environment, a 6.4 pH solution of hydrogen peroxide is capable of removing mineral contents [11]. A wide variation has been found in the pH of different brands of bleaching gels [10]. In an effort to reduce the erosive effect of bleaching solutions, one should give preference to those with a pH close to neutral, because the solutions' pH may be responsible for the erosive effect [7].

We undertook this study to determine possible ultrastructural changes in four laser-activated power bleaching products at different impact times, using environmental scanning electron microscopy (ESEM). The pH of the tooth-whitening products was measured so that potential risks could be recognized. To confirm that lightening had taken place, we also evaluated tooth color shades.

Materials and methods

Surface effects of four different laser bleaching agents, activated either by 2ωNd:YAG laser or diode laser, on tooth structure and on color change were evaluated by environmental scanning electron microscopy and a digital colorimeter. A pH meter served to evaluate the pH of the products used.

Specimen preparation

Only carious-free incisors and single-rooted premolars with intact enamel surfaces were used that had been extracted for periodontal ($n=12$) or orthodontic ($n=12$) reasons. Further conditions were none to minimal fillings, hardly any plaque, and no root canal treatment. To avoid dehydration the teeth were stored in physiological saline solution immediately after extraction. The time between extraction and bleaching procedure was no longer than 21 days.

To achieve optimal surface conditions we tested different cleaning procedures under optical and electron microscopes. The most effective and protective method for the enamel surface was determined to be polishing with pumice, medium and fine fluoride-free polishing pastes (Clean Polish®, Super Polish®, KerrHawe, Bioggio, Switzerland) and a final ultrasonic bath for 30 min. Each step was followed by a 2 min application of air–water spray. After control under the optical microscope 28 teeth were chosen for the study.

The buccal enamel surface of each specimen was divided into two halves by a groove prepared with a diamond burr and filled with Beauty Pink® wax. One-half of each tooth remained untreated and served as a negative control. It was covered with a flowable light-cured resin material (Opal Dam Light Cured®).

Enamel specimens were numbered, measured for tooth color determination, and embedded into gypsum blocks up to approximately 2 mm below the enamel–cement border for easier handling. The 28 cleaned teeth were randomly divided into 14 groups and treated with different impact times (Table 1). Each group consisted of one tooth from a periodontal patient, because of the higher discoloration, and one from an orthodontic patient, due to the absolute sound surface. Bleaching occurred in a humidity chamber at 37°C.

Products and devices

The four bleaching gels were each mixed from a powder and a 35% to 55% hydrogen peroxide solution. These products may be used only under dental supervision (in the office).

The Smartbleach® (High Tech Laser, Milton QLD, Australia) power bleaching gel can be activated with argon ion laser (514.5 nm) or with the 2ωNd:YAG laser as used in this study. Powder and 55% hydrogen peroxide are merged, whereby the concentration decreases to 25%. Manufacturer's instruction recommend a holding time of 5 min to allow the carbonate buffer system within the gel to elevate the pH to approximately 9.5.

The 37% hydrogen peroxide gel Opus White® (Opus Dent, London, UK) was activated by diode laser; pH (manufacturer's information) 5.5–6.5.

An improved 35% hydrogen peroxide bleaching agent developed at the Institute of Solid State Physics (Technical University, Vienna, Austria) was irradiated with the diode laser. This gel contains, among others, fine TiO_2 particles with a diameter of 3–30 nm (VWR Merck, Darmstadt, Germany), which can give very good scatter but also absorb radiation of approximately 800 nm to 1100 nm, to a certain amount, with the effect that the laser energy remains within the gel and is minimally transmitted. Therefore, it can be activated with diode (810 nm and 980 nm) and Nd:YAG (1064 nm) lasers; pH (manufacturer's information) 7.0.

Opalescence Xtra Boost 38%® (Ultradent, South Jordan, Utah, USA) uses carotene as a light-absorbing, heat-producing agent, but it does not need a source of light for activation. Laser or light irradiation nevertheless accelerates the procedure. In this study activation occurred with a diode laser; pH 7.0. The high absorption of the 810 nm

Table 1 Treatment parameters and results of surface ultrastructural evaluations (*cw* continuous wave, TiO_2 titanium dioxide)

Group	Product	Laser	Setting	Radiation time (s)	Impact Time (min)	ESEM[a]	Efficacy[b]
1.1	Smartbleach®	2ωNd:YAG	cw, 0.7 W	1×30	1×1	++	2
1.2	Smartbleach®	2ωNd:YAG	cw, 0.7 W	2×30	2×1	++	3
1.3	Smartbleach®	2ωNd:YAG	cw, 0.7 W	3×30	3×1	+	4
1.4	Smartbleach®	2ωNd:YAG	cw, 0.7 W	4×30	4×1	+	4
1.5	Smartbleach®	2ωNd:YAG	cw, 0.7 W	2×30	2×10	~	4
1.6	Smartbleach®	2ωNd:YAG	cw, 0.7 W	4×30	4×10	~	10
2.1	Opus White®	Diode	cw, 0.8 W	2×30	2×1	~	1
2.2	Opus White®	Diode	cw, 0.8 W	4×30	4×1	−	6
3.1	TiO_2 gel	Diode	cw, 0.8 W	2×30	2×1	++	7
3.2	TiO_2 gel	Diode	cw, 0.8 W	4×30	4×1	++	7
3.3	TiO_2 gel	Diode	cw, 0.8 W	2×30	2×10	++	7
3.4	TiO_2 gel	Diode	cw, 0.8 W	4×30	4×10	++	10
4.1	Opalescense Xtra B®	Diode	cw, 0.8 W	2×30	2×10	++	6
4.2	Opalescense Xtra B®	Diode	cw, 0.8 W	4×30	4×10	+	7

[a] ESEM evaluation:
++ no surface alteration
+ minimal damage limited to preexisting erosions
~ slight alterations
− marked surface damage
[b] Improvement in brightness steps on the basis of the Vita® brightness scale (Table 2) in comparison with the baseline (mean of ten measurements).

wavelength in the red bleaching gel was determined in preliminary tests. The attenuation of the laser beam increases exponentially with the transmission depth and is dependent on the absorption coefficient of the gel. The absorption coefficient itself depends on the wavelength to be absorbed. There is a correlation between the absorption coefficient of the bleaching gel and the course of temperature increase in the pulp chamber. Intrapulpal temperature measurements revealed no temperature increase in the pulp chamber when Opalescence Xtra Boost 38%® was irradiated with 810 nm, 1 W, for 60 s. The control without gel application showed a 5°C increase. The values for 2 W and 60 s were 2.8°C and 9.6°C, respectively.

Irradiation occurred with a Smart Lite® 2ωNd:YAG [potassium-titanyl-phosphate (KTP)] laser (DEKA Dental Laser Systems, Florence, Italy) at 532 nm and an output power of 1 W in continuous wave (cw) mode (effective output power measured with a watt meter: 0.7 W) and an LD 15® diode laser at 810 nm (Dentec Laser Systems, Bremen, Germany), 1.5 W, cw (effective output power 0.8 W). The distance of the bleaching handpieces was adjusted to achieve a fixed beam diameter of 6 mm.

Bleaching procedure

The same treatment protocol was used for all products: The bleaching gel was attached on the right enamel surface of each sample, and the laser power was applied stationary with a spot size of 6 mm in the non-contact mode for each irradiation, respectively, for both laser systems, resulting in irradiances of 2.5 W/cm^2 and 2.8 W/cm^2, respectively. Preliminary investigations revealed that intermittent irradiation of six times for 5 s, with 5 s breaks, with the appropriate laser resulted in a temperature increase of less than 3°C in the pulp chamber for both laser systems. This treatment protocol excluded thermal damage to the pulp and discomfort to the patient when applied in vivo. After an impact time of 1 min and/or 10 min, the gel was removed by suction and rinsed off for 2 min with H_2O. This procedure was repeated up to four times (Table 1), so that a maximum impact time of 40 min with 2 min of maximum irradiation time could be obtained. These impact times were chosen on the basis of the manufacturer's recommendation and preliminary in vitro and in vivo investigations. After removal of the light-cured resin, the teeth were rinsed off

Table 2 Value-oriented Vita® brightness scale ranging from the visibly lightest (B1) to darkest (C4) shade guide

B1	A1	B2	D2	A2	C1	C2	D4	A3	D3	B3	A3,5	B4	C3	A4	C4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16

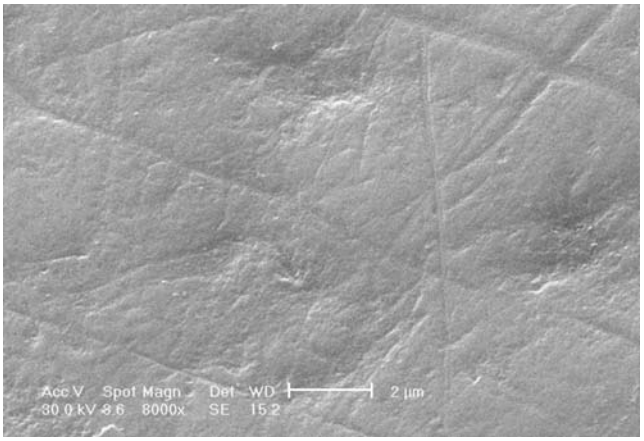

Fig. 1 ESEM of a control specimen shows scratch lines from residual polish (group 1.4) ×8,000

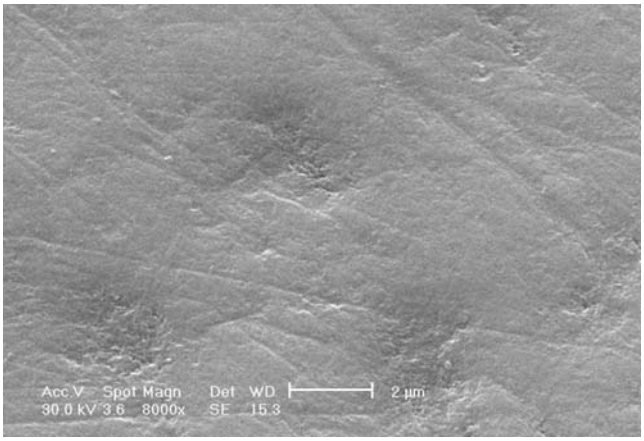

Fig. 2 ESEM of Smartbleach® and 2ωNd:YAG laser irradiation at 0.7 W, 4×30 s, 4 min impact time reveals minimal changes at previously damaged areas (group 1.4) ×8,000

once again and placed in artificial saliva containing 1.5 mM Ca^{2+} and 0.9 mM PO_4^{3-} (pH 7.0, 37°C), for potential remineralization, in light-protected containers for 48 h.

Evaluation of treatment effects

PH testing A WTW® pH meter pH 323/325 (Wellheim, Germany) was recalibrated with buffered pH solutions before each experiment. For each bleaching product, five readings were performed, 5 min after the gel had been mixed, and averaged. The products were in contact with the pH electrode for 5 min at room temperature to allow the pH value to stabilize.

Color measurement Color determination served solely to confirm that whitening had taken place. Enamel specimens were measured for tooth color before and 2 days after the bleaching procedure with the Shofu intra-oral contact colorimeter Shade Eye-Ex® Chroma Meter (Kyoto, Japan). In the meantime, the samples were stored in artificial saliva in light-protected containers, since rebound of color change often occurs due to remineralization and rehydration at the end of bleaching treatment [12]. Tooth dehydration is a probable cause of immediate tooth lightening [13, 14], and it presumably is greater with increased tooth heating [15].

Based on the shade, value, and hue results, the colorimeter selects the nearest Vitapan® classical shade guide equivalent and prints it as the guide number. For each sample, five measurements were recorded from the middle one-third of the tooth. Effectiveness was determined after a numerical shade score (Vita®, Bad Saeckingen, Germany) had been assigned, ranging from 1–16 and based on the sequence recommended by the manufacturer (Table 2).

Environmental scanning electron microscopy

Forty-eight hours after treatment, the ultrastructural effects of the bleaches on enamel were determined with a Philips XL30 ESEM® (Amsterdam, The Netherlands). For each sample, the middle third of the treated and untreated buccal surfaces were examined and assessed on the basis of the degree of surface damage (Table 1). The related sides were evaluated primarily in real-time to ensure the interpretation of the complete treated side. Only sample pairs of the same tooth were compared and recorded. A total of 112 images with a magnification of ×500 and ×8,000 were taken. Attention was focused on the degree of surface alteration over a large area rather than to specific sites. The use of ESEM has been established for providing a useful means for non-destructive microscopic histomorphography of surface areas of naturally wet oral hard tissues, without the need for a complex preparation and drying process. Another advantage is the avoidance of preparation artifacts. No statistical analysis was performed, because the observation by ESEM was designed to be qualitative.

Results

The pH values deviated from the manufacturers' specifications. Smartbleach® was the only product that was determined to have an alkaline pH. The remaining products were all in the acid range.

The mean pH [± standard deviation (SD)] of the bleaching products ranged from 6.1±0.4 for the gel containing TiO_2 particles (acidic) to 7.2±0.1 for Smartbleach® (neutral). A pH of 6.5±0.5 for Opus White® and 6.8±0.4 for Opalescense Xtra Boost® were in the acid range.

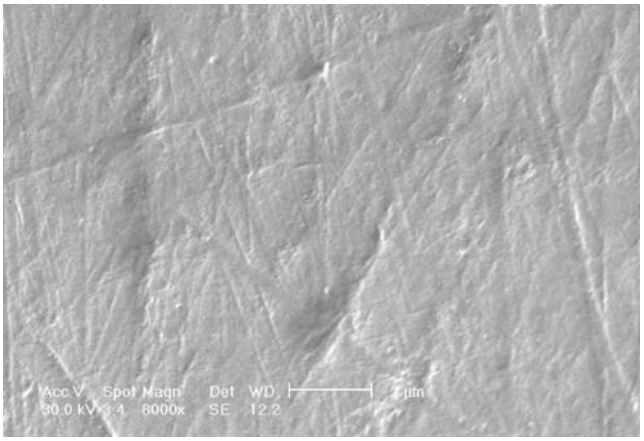

Fig. 3 ESEM of a control side shows remaining polishing scratches (group 1.6) ×8,000

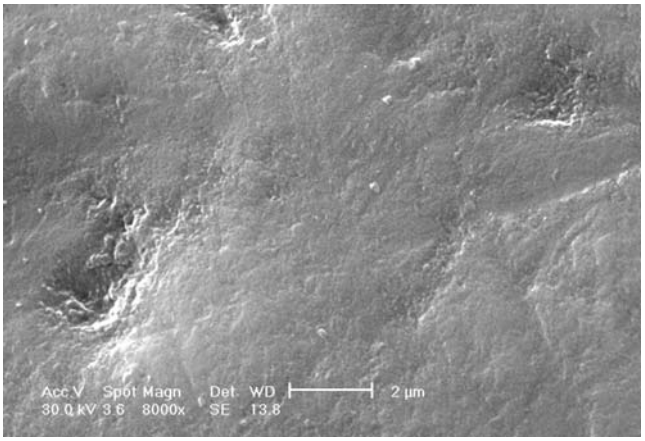

Fig. 5 ESEM of untreated control shows pits (group 2.2) ×8,000

Color changes associated with laser bleaching are shown in Table 1. Bleaching was highly successful; the power bleaching procedures showed significant whitening effects on the Vita shade tab, with decreases ranging from 1 unit to 10 units on the numerical Vita® shade guide brightness scale. Treatments with the 2ωNd:YAG laser-activated Smartbleach® gel and the TiO_2 gel activated by diode laser showed the strongest bleaching reactivity. The effective bleaching shown for specimens under these conditions verified their applicability for assessments of effects on surface ultrastructure. Owing to in vitro conditions of this study, color measurements served solely to confirm that whitening had taken place in any case.

Table 1 shows the results of surface ultrastructural evaluations of the treatment groups. With the exception of Opus White®, which generated marked damage, superficial dissolution and erosions, laser bleaching did not decisively alter enamel surfaces (Figs. 1 and 2). The prolonged use of Smartbleach® caused some areas of mild erosion (Figs. 3 and 4); a short impact time, however, showed minimal changes only at previous damaged areas (Figs. 5 and 6). Moderate superficial alteration, especially at pre-existing damaged areas, due to the extended impact time was also noticed for Opalescense Xtra Boost® (Figs. 7 and 8). No adverse effects on surface morphology were seen following laser bleaching with the gel containing TiO_2, even after 40 min of application (Figs. 9 and 10).

Discussion

External bleaching therapy with activation by light or laser may be accompanied by a temperature increase at the tooth surface, as well as in the pulpal chamber. However, the bleaching gel usually applied may act as an isolator, reducing intrapulpal temperature increase in comparison with that with laser irradiation only [16]. This means that laser activation (830 nm diode laser, 30 s, 3W) without the

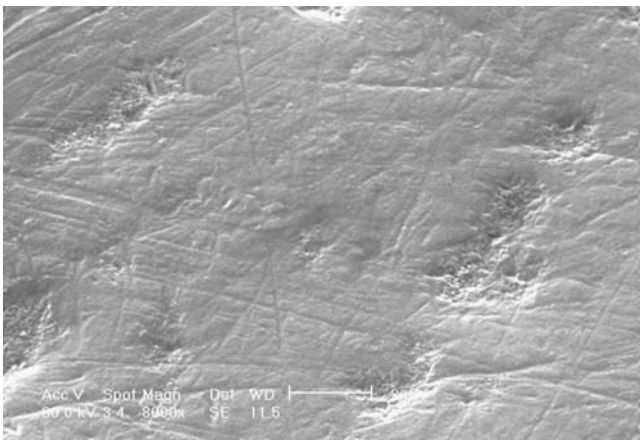

Fig. 4 ESEM of Smartbleach® and 2ωNd:YAG laser irradiation at 0.7 W, 4×30 s, 40 min impact time, reveals moderate superficial alteration, expanded at pre-existing damaged areas due to the long impact time (group 1.6) ×8,000

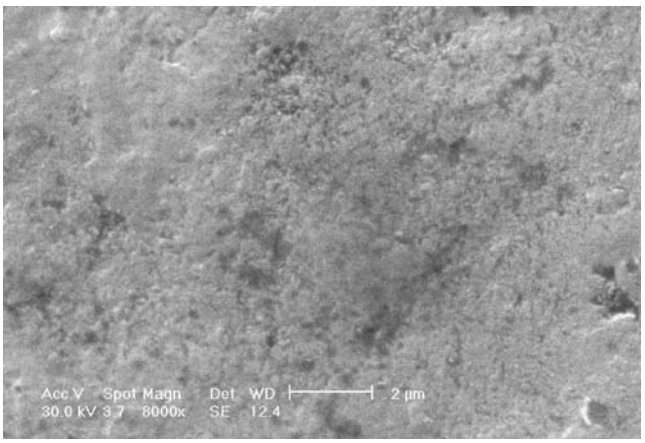

Fig. 6 ESEM of Opus White® and diode laser irradiation at 0.8 W, 4×30 s, 4 min impact time, reveals marked damage, superficial dissolution and erosion (group 2.2) ×8,000

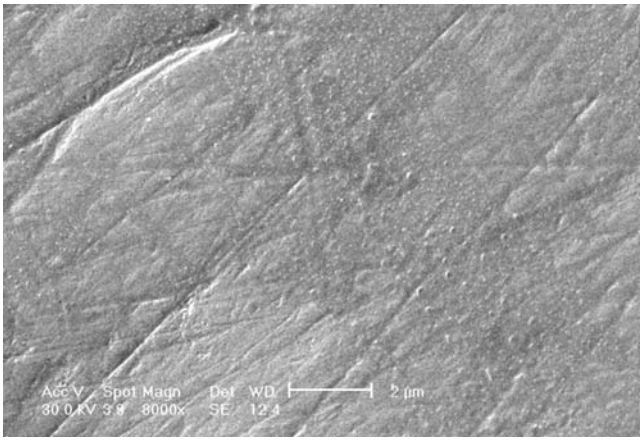

Fig. 7 ESEM of control after removal of the flowable composite material ×8,000

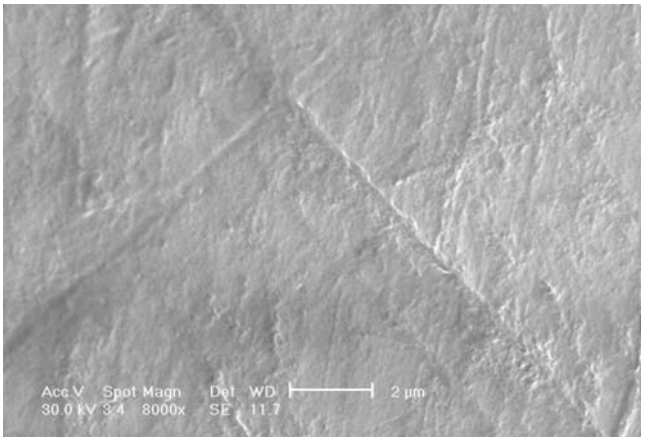

Fig. 9 ESEM of the untreated reference shows enamel surface with some scratches (group 4.2) ×8,000

use of bleaching gel results in an intrapulpal temperature increase of approximately 16°C, whereas only an 8.7°C temperature increase was recorded when a gel was applied during activation [17]. The increase in the pulp chamber temperature with a diode laser used at 1–2 W is below the critical temperature increase of 5.5°C that is nowadays regarded as the threshold value, which should not be exceeded, to prevent irreversible pulp damage [18]. A temperature increase of 2–8°C and 4–12°C was observed when a 960 nm diode laser was used to activate Opalescence Extra and Opus White for 60 s (0.9 W) and 30 s (2W) [19]. For a hydrogen peroxide bleaching agent, the mean maximum pulpal temperature rise was 2.95°C for a light-emitting diode (LED), 3.76°C for a 2ωNd:YAG laser, and 7.72°C for a diode laser [20]. With an output power of 1 W of a 810 nm diode laser, pulpal temperature increase was shown to be approximately 3°C with the Opus White® gel, whereas a TiO_2 emulsion showed almost no temperature changes in the pulp [21].

Our treatment protocol: intermittent irradiation of six times for 5 s, with 5 s breaks in between, at a power setting of less than 1W, excludes thermal damage to the pulp and discomfort to the patient in vivo. Irradiation times and corresponding breaks were determined in preliminary tests with thermocouples so that a temperature increase of less than 2°C in the pulp chamber could be guaranteed.

Controlling the color of teeth and dental restorations is difficult and is affected by many factors, such as individual differences in understanding and perceiving color, experience of the observer, lightening, and surrounding gingival color [22–24]. Shade guides made by the same manufacturer may differ slightly, and shades often lack the volume of color space required to represent the natural dentition [25, 26]. Colorimeter measurements have been compared with spectrophotometer readings and seemed reliable and accurate for color difference measurements [27]. In this study, whitening was determined with the intra-oral contact

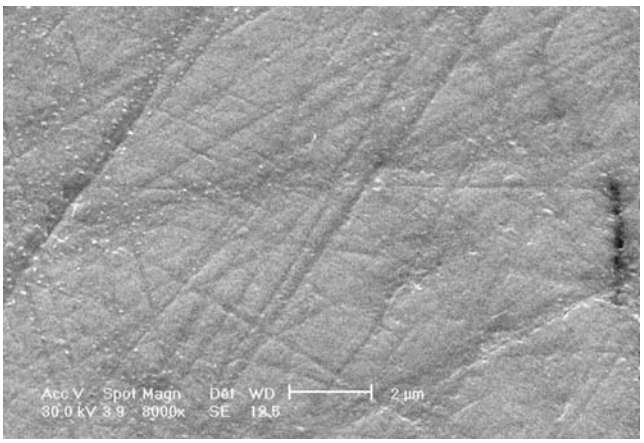

Fig. 8 ESEM of TiO_2 gel and diode laser irradiation at 0.8 W, 4× 30 s, 4 min impact time. No alteration of the enamel's surface can be observed (group 3.1) ×8,000

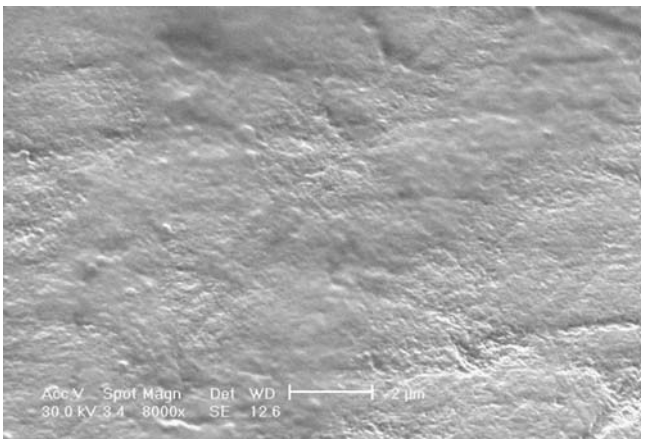

Fig. 10 ESEM of Opalescense Xtra Boost® and diode laser irradiation at 0.8 W, 4×30 s, 40 min impact time, reveals minimal damage, limited to pre-existing erosions (group 4.2) ×8,000

colorimeter Shade Eye-Ex® Dental Chroma Meter (Shofu). Based on the shade, value and hue results, the colorimeter selects the nearest Vitapan® classical shade tab equivalent and prints it as the guide number. Colorimetric measurement procedures can introduce variation as well. The pressure with which the contact tip is applied and the angle at which it is held are both important factors.

Finally, we want to discuss the tooth surface characteristics which can influence the measurements. There are a limited number of teeth with a flat surface large enough to accommodate the 4 mm measurement tip [27]. The spectral reflectance of a contoured surface can hardly be duplicated. It is therefore proper to expect some variation in the results [28].

Importantly, the color analysis of treated sides in this study confirmed that bleaching had, indeed, occurred, rendering later measurements of the effect of bleach on enamel properties more relevant. Owing to in vitro conditions of this study, color measurements served solely to confirm that whitening had taken place in either case. Highest bleaching ability was detected for the Smartbleach® gel, in combination with 2ωNd:YAG laser, and the TiO_2 gel, activated with 810 nm diode laser. Poorest results were determined for the combination of the Opus White® gel and diode laser, which is in accordance with in vivo DOTCAM findings of Walsh et al. [12]. Frequency doubled Nd:YAG laser-induced bleaching gives a significantly higher delta L than does diode laser treatment [20]. Activation with a 960 nm diode laser also shows that the mean brightness increase is always higher for Opalescense Xtra® than for Opus White® [29].

The pH of bleaching agents determines the rate of reaction of the bleaching process. The more free radicals are produced, the higher the pH [30]. Optimal ionization occurs when hydrogen peroxide is buffered in a range of pH 9.5–10.8 [31]. In this range, the bleaching effect could be 50% better than in an acidic environment. However, most commercial bleaching products are acidic, as this results in longer shelf life [32]. Thus, most products are optimized for shelf life rather than for bleaching action [30]. Under alkaline conditions, the perhydroxyl radical is produced from hydrogen peroxide [33]. This radical is more reactive than superoxide and other radicals. In addition etching of the tooth surface does not occur [12].

The results of the pH measurements showed that only the Smartbleach® gel was slightly alkaline, whereas the other three products were acidic. The mean pH of the bleaching products ranged from 6.1±0.4 to 7.2±0.1.

Bleaching agents cause superficial structural changes to dentin [34] and enamel [35, 36], and the acid pH probably produces an acid etch effect on dentin, increasing its permeability [37]. Concentrated 30% solutions of hydrogen peroxide can also reduce the microhardness of enamel and dentin. This reduction can be noted with exposure times as short as 5 min for dentin and 15 min for enamel [38]. Superficial destruction was documented after 6 weeks, with the appearance of patterning similar to that of acid etching and the presence of some crystalline areas emerging from the body of the prisms [33]. Studies by scanning electron microscopy have shown that even a concentration of 10% carbamide peroxide, breaking down in the presence of saliva into 7% urea and 3% hydrogen peroxide [39], alters enamel, causing surface dissolution and exposing a porous surface [40–43]. There was also a trend for the microhardness of enamel surfaces initially to decrease when the enamel was exposed to bleaching agents [7]. However, slight alterations of the enamel surface did not become more severe in vivo within 6 months [44].

Three agents in this study showed no severe alterations to the enamel surface. No adverse effects on surface morphology were seen following laser bleaching with Opalescense Xtra Boost® or the new gel containing TiO_2 particles, or with the Smartbleach® gel. However, the prolonged use of Smartbleach® caused some areas of mild erosion. Opus White® generated marked damage, superficial dissolution, and erosions. Moderate superficial alteration, especially at pre-existing damaged areas, due to the long impact time was noticed for most applications.

These results demonstrate, with the exception of the Opus White® gel, no significant ultrastructural effects within enamel associated with laser bleaching of various intensities. Nevertheless, excessive treatment cannot be recommended.

Conclusions

Laser-activated bleaching offers an improvement in terms of effectiveness and enamel surface protection. Prerequisites are a perfect match of the chosen laser wavelength and the bleaching gel, a short impact time, and the absence of pre-damaged areas. A neutral or alkaline pH of the gel is also advantageous.

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