Effect of light units on tooth bleaching with visible-light activating titanium dioxide photocatalyst

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This study evaluated the influence of different light sources on the efficiency of an office bleaching agent containing visible-light activating titanium dioxide photocatalyst (VL-TiO2) using an artificial discoloration tooth model. Extracted bovine teeth were stained by black tea. The CIE L*a*b* values were measured before and after nine consecutive treatments by the VL-TiO2-containing bleaching agent (TION in Office, GC, Tokyo, Japan). A halogen light unit (CB; CoBee, GC) or an LED unit (G-light, GC) with two modes (blue and violet: GL-BV, blue: GL-B) were used to activate the bleaching agent in three groups (n=8). Brightness (ΔL) and color difference (ΔE) increased as bleaching repeated in all groups. Two-way ANOVA showed that both number of treatments and light source influenced the efficiency of the tooth bleaching with VL-TiO2.

Keywords: Bleaching, Titanium dioxide photocatalyst, Light source

INTRODUCTION

Tooth appearance is an important factor defining beauty and attractiveness, and aesthetic dental treatments are highly demanded by patients nowadays. Tooth bleaching is one of the most conservative dental treatments to improve or enhance a person’s smile9). Bleaching has been faster growing oral care sectors.

Three bleaching approaches are available namely, home bleaching, office bleaching and walking bleaching. The former two techniques are advocated for improving the color of discolored vital teeth, and the latter applies to non-vital, endodontically treated teeth. Tooth bleaching was first described in 1848 using chloride of lime2). The use of hydrogen peroxide (H2O2) to bleach the teeth was later introduced in 18843). In 1918, Abbot5) described the chairside bleaching method, as it is known today, using 35% H2O2 together with heat and light to boost the oxidation reaction. On the other hand, home bleaching which allowed the patients to perform the bleaching procedures by 10% carbamide peroxide and custom trays at home appeared for the first time in 19896). Current home bleaching products typically contain a relatively low level of the bleaching agent, which is applied to the teeth via a custom fabricated mouth guard and worn every night for at least 2 weeks6).

Compared to the home bleaching agents, available office bleaching products generally contain relatively high levels of the bleaching agents, for example 35% H2O2, and are applied for shorter time periods6). Factors contributing to the efficacy of the office bleaching are temperature, H2O2 concentration, application time, light or energy sources, and the presence of some catalysts. A combination of these factors can accelerate the office bleaching process and result in a higher short-time efficacy8). In this regard, office bleaching treatment can result in significant bleaching after several treatment visits6).

In order to provide faster and more effective treatment, office bleaching agents have been used in association with an energy source. Absorbance of the energy in the bleaching gel will produce heat that accelerates the oxidation reaction in the office bleaching agent. In the earlier systems, the use of a heated spatula or a heat lamp was recommended to accelerate the chemical reaction. On the effect of heat on office bleaching, it was reported that a 10°C increase could double the chemical reaction rate10). However, the temperature achieved by these instruments was very high, and could damage pulp tissue of the vital tooth. Other methods to deliver the energy required for accelerating the chemical reaction have been required, such as light units and/or catalysts. Various devices at different wavelength spectra and radiation energies, such as halogen, light emitted diodes (LEDs), diode lasers, argon lasers and plasma arc lamps have been used in this regard. However, there are questions in subsequent scientific dialogue about the benefits brought by these light units on the office bleaching results11).

Apart from the effect of heat on the office bleaching, some bleaching agents contain photosensitive agents and components designed to absorb additional energy from the light source. The absorbed energy may speed up the oxidation reaction of H2O2, and consequently
enhance effectiveness and speed of bleaching\textsuperscript{12}. A new office bleaching material containing visible-light activating titanium dioxide photocatalyst (VL-TiO\textsubscript{2}) and H\textsubscript{2}O\textsubscript{2} has been recently developed. Titanium dioxide is a very low-toxic and inexpensive substance that has been used for various cleaning applications. It has been also known as the most important semiconductor photocatalyst reacting to ultraviolet light\textsuperscript{13}. VL-TiO\textsubscript{2} was modified from original titanium dioxide photocatalyst and the application of visible light on a bleaching agent containing H\textsubscript{2}O\textsubscript{2} and VL-TiO\textsubscript{2} could increase the bleaching efficacy\textsuperscript{12,14}. The VL-TiO\textsubscript{2} works as a photocatalyst reacting to visible light especially at low wavelength\textsuperscript{12}. However, few studies to date have investigated various aspect of the newly developed photo-accelerated bleaching agent. While there are various light units available in the practices, mainly used for photopolymerization of light activated resin-based restorative materials, it is unclear how different light sources and wavelengths affect office bleaching by an agent containing VL-TiO\textsubscript{2}. Based on this background, we evaluated the influence of different light sources on the efficacy of an office bleaching agent which contains VL-TiO\textsubscript{2} using an artificial discolored bovine tooth model. The null hypothesis was that there were no significant differences in efficacy of the bleaching agent between the light sources.

**MATERIALS AND METHODS**

*Preparation of discolored teeth*

Thirty extracted bovine incisors were used in this study. The teeth were stored frozen after extraction and were thawed by running tap water before the experiment. The crowns were cleaned by removing soft tissue remnants using a scalpel, and polished with a brush with a contra-angle micromotor handpiece and non-fluoridated polishing paste (Pressage, Shofu, Kyoto, Japan). The roots were cut using a diamond disc (Summadisk, Shofu, Kyoto, Japan) with straight-type micromotor handpiece and the pulpal tissue was removed by a reamer. The pulp chamber was irrigated with 5 mL of 5% sodium hypochlorite (Wako Pure Chemical, Osaka, Japan) to remove any tissue remnants followed by thorough washing, drying, and finally etching with 30% phosphoric acid gel (K-etchant, Kuraray Medical, Tokyo, Japan) for 10 s, to expose the tubule system and thereby encourage stain uptake into the dentin\textsuperscript{16}. Labial surfaces were ground to leave enamel of 1 mm in thickness, and polished to create a smooth and flat enamel surface with ascending-grit silicon carbide papers (Sankyorikagaku, Saitama, Japan) starting from #280 up to #1,500 under running water.

A stain solution was prepared by immersion of one black tea bag (2 g) (Nittoh-tea, Mitsui Norin, Tokyo, Japan) in 100 mL of boiled water for five minutes. Specimens were immersed in the solution and stored inside the incubator for 7 days at 37°C. The solution was stirred once every day to avoid decantation of the solution and changed after every three days.

*Color measurement*

After removing from the solution, the specimens were rinsed and dried. Labial surfaces were covered with a masking tape with a 5 mm-diameter hole to fit the probe of a dental chroma meter (ShadeEye NCC, Shofu, Kyoto, Japan). This procedure ensured measuring the same area at each step. The CIE $L^*a^*b^*$ values of tooth surfaces were recorded prior bleaching for the first time as a baseline data, using the dental chroma meter and then the photograph of each experimental surface was taken by a digital camera. In order to decrease the variation among the specimens, only the specimens which showed an $L^*$ value between 45 and 65 were included in the design. In this manner, a total of 24 teeth were selected for following experiment.

<table>
<thead>
<tr>
<th>Table 1 Composition of the bleaching agent used in this study</th>
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<tbody>
<tr>
<td><strong>TiON in office (GC, Tokyo, Japan)</strong></td>
</tr>
<tr>
<td>Reactor ethanol, titanium dioxide, thickener, pH conditioner, water</td>
</tr>
<tr>
<td>syringe A 35% hydrogen peroxide</td>
</tr>
<tr>
<td>syringe B 30% carbamide peroxide, glycol solvent, thickener, pH conditioner</td>
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<table>
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<tr>
<th>Table 2 Visible light units employed in this study</th>
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</thead>
<tbody>
<tr>
<td><strong>Light units and (code)</strong></td>
</tr>
<tr>
<td><strong>Light source irradiation mode and light intensity</strong></td>
</tr>
<tr>
<td><strong>Manufacturer</strong></td>
</tr>
<tr>
<td>CoBee (CB) Halogen 900 mW/cm\textsuperscript{2} GC, Tokyo, Japan</td>
</tr>
<tr>
<td>G-light (GL-BV) 7 blue and 1 violet LED units 650 mW/cm\textsuperscript{2} GC, Tokyo, Japan</td>
</tr>
<tr>
<td>(GL-B) 7 blue LED units 600 mW/cm\textsuperscript{2}</td>
</tr>
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Tooth bleaching

The selected specimens were treated with an office bleaching agent (TiON in office, GC, Tokyo, Japan) according to the manufacturer’s instruction. The composition of the bleaching agent used in this study was shown in Table 1.

Before application of the bleaching agent, the specimens were randomly assigned into three groups (n=8). The reactor was applied on the experimental surface to be whitened using a disposable brush, and excess reactor was removed by gentle air confirming evaporation of solvent of reactor. A and B syringes were connected and mixed. The mixed gel was applied on the experimental surface.

The specimens in each group were photo-irradiated for one minute using one of the light units: a conventional halogen light unit (CB; CoBee, GC, Tokyo, Japan) or LED light unit (G-Light, GC, Tokyo, Japan). G-Light contains seven blue LED units and one violet LED units. G-Light was utilized in two modes; blue and violet LED (GL-BV) or only blue LED (GL-B) irradiation modes in this experiment. Specifications of the light units according to the manufacturer were shown in Table 2. Following photo irradiation for 1 min, the bleaching agent was left on the specimens for 5 min. The bleaching gel was then wiped off from the experimental surface using a piece of damp gauze. The specimen was washed thoroughly under tap water, dried gently and the color values of $L^*a^*b^*$ were measured using the chroma meter. Bleaching treatments and measurements were subsequently repeated for 9 times on each specimen. The difference of $L^*$, $a^*$ and $b^*$ between the baseline and each period of the bleaching were expressed as $\Delta L$, $\Delta a$, and $\Delta b$ respectively. The color difference ($\Delta E$) was calculated from the values obtained at the baseline and after each treatment, according to the following equation:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

The wavelength spectra of each unit were also measured using a spectroradiometer (USR-45V/D, Ushio Electric, Tokyo, Japan).

Statistical analysis

$\Delta E$ data of each group was subjected to statistical analysis with the two-way ANOVA test, with the light irradiation method and treatment time as factors. Tukey's HSD test was used as post hoc test for multiple comparisons. The statistical significance was defined as $p<0.05$.

RESULTS

Figure 1a–d showed photographs of a representative

![Fig. 1 Representative images of tooth bleaching in CB group. (a) before bleaching of experimental surface (b) after first time bleaching (c) after sixth time bleaching (d) after ninth time bleaching (e) Crosscut surface of the stained bovine tooth before bleaching. (f) Crosscut surface after ninth bleaching. The arrows showed bleached area.](image-url)
specimen from CB group at the baseline and after 3, 6 and 9 times of bleaching. A gradual but remarkable bleaching effect was observed during the nine times of the treatment in all groups. The crosscut surfaces of specimens at the baseline and after the 9th treatment time were also presented (Fig. 1e, f). In the crosscut surface, the bleaching effect was confirmed not only on the bleached surface but also within the underlying enamel and dentin.

$\Delta L$, $\Delta a$, $\Delta b$ and $\Delta E$ values of each bleaching time in each group were shown in Figs. 2–4. After bleaching, $\Delta L$ and $\Delta E$ values gradually increased in all groups, while $\Delta a$ and $\Delta b$ showed a gradual decrease. Figure 5 summarized $\Delta E$ change in each group. Results of the two-way ANOVA test were summarized in Table 3. Both factors of light irradiation method and treatment time had a significant effect on $\Delta E$ ($p<0.05$), but their interaction was not significant ($p=0.05$). Multiple comparisons test showed that there was a significant difference between GL-BV and GL-B groups ($p<0.05$), and there were no significant differences among other pairs ($p>0.05$). The ascending order of $\Delta E$ was shown in

![Fig. 2](image2.png) The mean $\Delta L$, $\Delta a$, $\Delta b$, and $\Delta E$ values of CB group.

![Fig. 4](image4.png) The mean $\Delta L$, $\Delta a$, $\Delta b$, and $\Delta E$ values of GL-B group.

![Fig. 3](image3.png) The mean $\Delta L$, $\Delta a$, $\Delta b$, and $\Delta E$ values of GL-BV group.

![Fig. 5](image5.png) $\Delta E$ of each experimental group.

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Results of statistical analysis by two way ANOVA for color difference ($\Delta E$)</th>
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<tbody>
<tr>
<td></td>
<td>Sum of Squares</td>
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<tr>
<td>Main factors</td>
<td>Light</td>
</tr>
<tr>
<td></td>
<td>Period</td>
</tr>
<tr>
<td>2-way interaction</td>
<td>Light*Period</td>
</tr>
<tr>
<td>Residual</td>
<td>3356.491</td>
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<tr>
<td>Total</td>
<td>43534.600</td>
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Table 4. Color difference (ΔE) at each application time

<table>
<thead>
<tr>
<th>times</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
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<tbody>
<tr>
<td>CB</td>
<td>0.0</td>
<td>1.6</td>
<td>4.6</td>
<td>6.9</td>
<td>9.3</td>
<td>11.3</td>
<td>13.3</td>
<td>14.5</td>
<td>16.0</td>
<td>17.3</td>
</tr>
<tr>
<td></td>
<td>(0.0)</td>
<td>(1.2)</td>
<td>(2.0)</td>
<td>(2.6)</td>
<td>(2.8)</td>
<td>(2.9)</td>
<td>(3.3)</td>
<td>(4.0)</td>
<td>(4.0)</td>
<td>(4.2)</td>
</tr>
<tr>
<td>GL-BV</td>
<td>0.0</td>
<td>4.4</td>
<td>6.4</td>
<td>8.7</td>
<td>10.7</td>
<td>12.5</td>
<td>14.6</td>
<td>16.4</td>
<td>18.0</td>
<td>19.5</td>
</tr>
<tr>
<td></td>
<td>(0.0)</td>
<td>(3.9)</td>
<td>(4.9)</td>
<td>(5.4)</td>
<td>(5.9)</td>
<td>(6.0)</td>
<td>(5.8)</td>
<td>(5.8)</td>
<td>(6.1)</td>
<td>(5.9)</td>
</tr>
<tr>
<td>GL-B</td>
<td>0.0</td>
<td>3.1</td>
<td>5.2</td>
<td>7.5</td>
<td>8.9</td>
<td>10.3</td>
<td>11.5</td>
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<td>14.0</td>
<td>15.4</td>
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<tr>
<td></td>
<td>(0.0)</td>
<td>(1.3)</td>
<td>(2.5)</td>
<td>(2.9)</td>
<td>(3.0)</td>
<td>(3.9)</td>
<td>(4.4)</td>
<td>(4.5)</td>
<td>(4.7)</td>
<td>(5.7)</td>
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Number in parentheses shows standard deviation. Horizontal bar means no statistical difference.

DISCUSSION

This study was conducted to evaluate the influence of different light sources on the efficacy of an office bleaching agent containing VL-TiO₂ using an artificial discolored bovine tooth model. To date, numerous in vitro models have been used to evaluate the efficacy of tooth bleaching products and methods⁹,¹⁴,¹⁵. The majority of these models used whole or cut human or bovine tooth specimens at their pre-existing colors. However, some in vitro models increased the levels of intrinsic tooth color by pre-staining with black tea or blood components. In this in vitro study, bovine teeth were used as the substrate, as it has been reported that chemical and physical properties of bovine tooth such as composition, heat capacity, hardness, dentin tubule density and permeability are similar to human teeth¹⁶,¹⁷. The discolored bovine tooth model used in this study seemed to be suitable for the evaluation of the bleaching materials and procedures.

In this study, the tea extract was used for staining the teeth, based on the discoloration model reported by Sulieman et al.¹⁵. The tea was used for staining the teeth in many previous studies concerning tooth bleaching⁷,⁸,¹⁵,¹⁸,¹⁹, as well-known procedure. The type of tea was chosen according to a number of previous experiments, where the stain solution could develop an intrinsic stain, which was consistent with tooth discoloration observed clinically⁷,¹⁵,¹⁸. Moreover, the discoloration was reproducible, as confirmed by the chroma meter at the baseline.

The bleaching procedures were repeated 9 times in the present study. According to the manufacturer's instruction of the bleaching agent, the procedure can be performed up to 3 times in a visit in order to reach the optimum results. This study was designed simulating 3 times.
visits, which make a total of 9 treatments.

There are many methods for evaluation of tooth shade changes after bleaching, and these methods can be classified as subjective, such as visual analysis by comparison with a standard tooth shade guide, and objective, such as use of spectrophotometers, chroma meter, and image analysis techniques with software. Although the evaluation of tooth shade by color guide matching is a simple method to use, it is not very reliable, because it is influenced by the observer’s experience, eye fatigue, and variation of the ambient light, among other factors. In this study, a chroma meter (ShadeEye NCC) and photography were used for the evaluation of the color change. The chroma meters can supply parametric data, which could be easily applied for statistical analysis. ShadeEye NCC is an intraoral contacting colorimeter. It is designed to measure the shades of natural teeth (tooth mode) and metal-ceramic restorations (porcelain mode) through a disposable contact tip of 2.5 mm in diameter. The manufacturer explained that it could accurately determine the tooth shade. It has been reported that the instrumental shade selecting by ShadeEye NCC could make better results than visual method within the uncomplicated cases.

The efficiency of the bleaching agent depends on the release of free radicals such as oxygen (O·) and hydroxyl (OH·). The free radicals act upon chromophore molecules and degrade them to smaller and more soluble molecules, thus reducing the chroma concentration and removing the chromatogens from the tooth. This reaction depends on the conditions, such as H2O2 concentration and the presence of some catalyst ions. Bleaching effect is also affected by heat. Although some researches showed that light irradiation accelerated bleaching efficacy, other could not find any efficacy of the light irradiation for the bleaching materials without photocatalyst. The bleaching effect of H2O2 generally depends on the extent to which hydroxyl radicals are generated. H2O2 has a tendency to absorb UV, which then induces the molecular vibration and degradation of H2O2, subsequently leading to an increase in temperature. However, clinical use of UV light for dental bleaching may have undesirable side-effects. A prolonged exposure to UV irradiation can result in toxic irritation that leads to cellular damage and immune suppression, skin cancer, and photoaging.

The material tested in this study contained 35% H2O2 in syringe A, and 30% carbamide peroxide in syringe B respectively. The mixture of both syringes will result in a gel containing approximately 23% H2O2, which was applied on the surface of the tooth treated by reactor which contained VL-TiO2. VL-TiO2 of TiON was the substitutional doping of nitrogen (personal communication with the manufacturer). The photocatalytic activity of VL-TiO2 doped with nitrogen is superior to that of the TiO2 catalyst in the visible range of irradiation. In a previous study, it was shown that addition of VL-TiO2 to the H2O2-based bleaching agent could enhance the bleaching efficacy of the agent when exposed to the visible light from a dental light unit. These lights were less effective for hydrogen peroxide alone without VL-TiO2. It was suggested that bleaching was enhanced by hydroxyl radical generation through the photocatalytic action of VL-TiO2. However, the effect of light sources on the bleaching effect had not been well-known.

In a quest to understand the effect of different light sources on the bleaching effect of the agent in the present study, a halogen light unit and an LED unit with two different modes were used. The use of GL-B on this bleaching agent resulted in a significantly lower bleaching efficacy compared to the GL-BV. The spectrum of both GL-BV and GL-B, showed a significant peak at 470 nm of blue LED, while that of GL-BV showed another small peak at 405 nm of violet LED. It was previously reported that while the photocatalyst exhibited the highest absorbance in the UV region; a strong absorbance of visible light in the wavelength region close to the UV region (around 405 nm) was also observed. It was thought that VL-TiO2 well reacted with the visible light around 405 nm and this reaction induced a better bleaching result in GL-BV group than in GL-B group. On the other hand, no statistically significant difference was found between CB and GL-BV, and between CB and GL-B groups. While it is difficult to interpret these results, at least it was found that the halogen light unit was effective for bleaching with VL-TiO2. Apparently, in addition to the wavelength, other attributes of the light might influence the efficacy of the new bleaching agent.

LEDs have a longer lifetime and undergo little degradation of output over time when compared to halogen light units. Taking the advantage into account together with similar bleaching efficacy yet lower output intensity of GL-BV compared to CB, the use of the LED light unit can be recommended instead of the halogen unit. Moreover, based on the results obtained, it could be suggested that the light spectrum of the violet LED was likely to increase the activity of the VL-TiO2.

Nevertheless, the bleaching efficacy with all the light sources in this study gradually increased with repeated treatment times. It should be noted that a ΔE more than 3.3 was considered to be the clinical change of color; in this study, all light units achieved 3.3 within 2 treatment times, indicating that a short bleaching time with the material can achieve remarkable effects. The VL-TiO2 containing gel can accelerate the bleaching process decreasing the clinical time required and potentially lowering the adverse effects of bleaching agents with longer application times on enamel. Future study is warranted on other aspects of the new bleaching agent, including the effects on mechanical properties of enamel.

**CONCLUSION**

The null hypothesis was rejected; it was concluded that the blue and violet LED light curing was more effective than blue LED light curing and conventional halogen lamp for tooth bleaching with VL-TiO2 photocatalyst. This new material was affected by wavelength of the
irradiating light.

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