ABSTRACT

Aim: This study evaluated the influence of light emitting diode (LED) illumination bleaching technique on color stability of various computer-aided design–computer-aided manufacturing (CAD–CAM) of ceramic materials.

Materials and methods: A total of 20 disk-shaped samples (width × length × thickness = 10 mm × 15 mm × 2 mm) were prepared from each of the ceramic materials for CAD–CAM including Lava™ Ultimate (L), Vita Enamic® (E), IPS e.max® CAD (M), InCoris® TZI (I), and Prettau® zirconia (P). The samples from each type of ceramic were randomly divided into two groups based upon different bleaching techniques using 35% hydrogen peroxide with and without LED illumination. Colorimetric evaluation was determined using spectrophotometer for color differences.

Results: The color difference (ΔE) upon bleaching, either without or with LED illumination [mean ± standard deviation (SD)], for each type of ceramic was 0.34 ± 0.21, 0.54 ± 0.41 for L groups; 5.59 ± 1.35, 5.81 ± 1.45 for E groups, 4.60 ± 1.80, 6.92 ± 1.42 for M groups, 3.43 ± 1.04, 4.14 ± 0.72 for I groups, and 6.61 ± 0.80, 7.03 ± 0.64 for P groups respectively.

The differences in color changes were indicated upon bleaching procedure in different degrees for the different ceramic materials. The analysis of variance (ANOVA) indicated significant differences of color change due to the effect of bleaching technique on the ceramic material (p < 0.05).

Conclusion: Bleaching can affect the color stability of dental ceramics for CAD–CAM. Using 35% hydrogen peroxide bleaching agent with LED illumination caused more color alteration of dental ceramics than without LED illumination.

Clinical significance: Bleaching affects color of ceramic materials. Clinicians should be careful in the protection of the existing restoration.

Keywords: Bleaching, Ceramic, Color stability, Illumination, Laboratory research, Light-emitting diode.

INTRODUCTION

Esthetic appearance in dentistry is primarily governed by the color of the teeth. The treatment amendments for unsatisfied color of teeth generally improve the self-confidence and personality of the patient. Esthetic dental treatment for discolored teeth can be performed with several approaches, for instance, enamel micro-abrasion, bleaching, veneering with porcelain or resin composite, and crown. Nevertheless, bleaching has become a prevailing treatment for recent years due to its conservative treatment approach to achieve an optimum esthetic appearance. Nowadays, bleaching agents are commercially provided in the form of hydrogen peroxide or as its compounds, such as carbamide peroxide. The mechanism of bleaching agents for the whitening of the tooth structure is through the decomposition of peroxide molecules into unstable free radicals. These free radicals can attack the large pigmented molecules that have precipitated on the dental tissues and convert them into smaller molecules, so that they can be easily diffusible.

Bleaching products are generally produced in the form of gels or paste containing 30 to 35% hydrogen peroxide to be used for in-office bleaching procedure, whereas 6 to 20% carbamide peroxide regimens are suggested for at-home bleaching procedure. The in-office bleaching technique usually uses a high concentration of bleaching agent that is applied to tooth structure for a short period of time because the product is capable of producing more peroxide radicals, hence accelerating the bleaching process. Furthermore, the use of light in association with the bleaching process has been increasing in popularity. Different light sources were introduced to be used in combination with bleaching agents, such as blue halogen lights, blue plasma arc lamp, LED, or light amplification by stimulated emission of radiation (lasers), such as diode laser, erbium-doped yttrium aluminum...
garnet laser, and CO₂ laser. The laser light was professsed for promoting the bleaching reaction by accelerating the release of hydroxyl free radicals and intensifying the efficacy of bleaching, even though tooth bleaching is considered to be comparatively safe in terms of potential risk and harmful to tooth structure. On the contrary, tooth sensitivity, resorption of tooth surface at cervical area, and microscopic alteration of tooth structure were reported, especially when bleaching agents were used at high concentrations.

Ceramics have shown the most accurate reproduction of the appearance, color, and texture of natural teeth. The technological advancements in CAD–CAM in dentistry have helped dental clinicians and researchers in developing new ceramic biomaterials to render high-quality restorative materials. Although traditional dental solutions, hence, it may be possible that free radicals released from bleaching agents are extremely reactive, unstable, and tend to induce acidic environment during bleaching, it may create adverse effects on structural changes of restorative materials and lead to material failure. The difference in bleaching materials and techniques exhibited different influence on various restorative materials. Although traditional dental ceramics are considered to be the most inert dental restorative material among others, their surfaces were reported to manifest surface deterioration on contact with some acidulated materials, such as fluoride gels and other solutions. Hence, it may be possible that free radicals produced from bleaching agents may cause dissolution in ceramic networks and leaching of alkali ion component of dental ceramic.

Some studies evaluated the effects of bleaching agents on the changes in surface morphology of teeth and restorative materials. Bleaching is effective at whitening certain types of discolored teeth, but its effect on restorative materials is not clearly understood. Controversial results with regard to the effect of bleaching on color of restorative materials were reported. Few studies have been conducted on the effects of bleaching agent on the color stability of various dental ceramic materials. In addition, there is no report in the literature regarding the influence of highly concentrated bleaching agents on dental ceramics, especially newly developed ceramic materials for CAD–CAM, and bleaching techniques with LED illumination. Therefore, this study aimed to determine the effects of 35% hydrogen peroxide in conjunction with LED illumination on quantitative color changes of different types of ceramic materials used for CAD–CAM. The null hypotheses were that bleaching either with or without LED illumination would not have meaningful effect to the color of different CAD–CAM ceramic materials.

**MATERIALS AND METHODS**

This study investigated the color stability on different bleaching techniques on different types of ceramic materials that were resin nanoceramic [Lava™ Ultimate (LV), 3M ESPE, St. Paul, Minnesota, USA], hybrid ceramic [Vita Enamic® (En), VITA North America Inc., Yoba Linda, California, USA], lithium disilicate glass ceramic [e.max® CAD (M.L.) Ivoclar Vivadent, Schaan, Lichtenstein], and zirconia ceramic including inCoris® TZI (IC; Sirona Dental Systems GmbH, Bensheim, Germany) and Prettau® zirconia (P.r.; Zirkonzahn Inc., Atlanta, Georgia, USA). Each type of ceramic was bleached with 35% hydrogen peroxide (Pola office, SDL, Bayswater, Victoria, Australia), using two techniques including bleaching with and without LED illumination (BT-cool, APOZA Enterprise, New Taipei, Taiwan).

**Sample Preparation**

A total of 20 disk samples from each type of ceramic material were prepared in a rectangular shape with the dimension of width, length, and thickness equal to 10 mm × 15 mm × 2 mm. The resin nanoceramic and hybrid ceramic samples were prepared from the Lava™ Ultimate and Vita Enamic® ceramics by cutting into disk shapes using a diamond-coated wheel (Isomet® 1000, Beuhler, Lake Bluff, Illinois, USA). The samples were then ground flat and polished with a silicon carbide abrasive paper at 800, 1000, and 2000 grit particles with a polishing machine (Ecomet®3 polisher, Beuhler, Lake Bluff, Illinois, USA) to achieve the required sample dimension.
The lithium disilicate ceramic samples were prepared from the presintered IPS e.max® CAD blank to achieve the required dimension, coated with a thin layer of IPS e.max® CAD crystal (Ivoclar Vivadent, Schaan, Lichtenstein), and then sintered to achieve crystallization and glazing surface in a porcelain furnace (Programmat® P100, Ivoclar Vivadent, Schaan, Lichtenstein) according to the firing schedule recommended by the manufacturer.

The zirconia samples were prepared from the presintered zirconia blanks of inCoris® TZI and Prettau® zirconia at the dimension approximately 20% larger than the final required dimension to compensate for volumetric shrinkage on sintering process in a sinter furnace (inFire®, Ivoclar Vivadent, Schaan, Lichtenstein) according to the manufacturer recommendations. All samples were stored in distilled water at 37°C for 24 hours before determination for the color parameters.

**Bleaching Techniques**

All samples from each type of ceramic materials were randomly divided into two groups on which the two bleaching techniques were performed according to the manufacturer instructions at room temperature (25°C). Newly mixed 35% hydrogen peroxide was applied to the top surfaces of the sample using a microbrush. Bleaching was performed according to the manufacturer recommendation for 8 min. The bleaching was performed in conjunction with LED illumination for the group that was power bleached. Thereafter, the bleaching agent was washed out with water jet spray for 60 seconds. Then, the bleaching agent was applied again as described previously for four times. On the conclusion of bleaching, all samples were stored in distilled water at 37°C for 24 hours before the determination of color parameters.

**Color Assessment**

The color was assessed using a reflectance spectrophotometer (ColorQuest XE, Hunter Inc., Reston, VA) at room temperature of 25°C. This instrument measures the spectral reflectance of color parameters based on D65 illuminant with 2° inclination of standard mirror observ- ers. The (Commission International de L’Eclairage L* a* b*) color system was used to determine for color parameters of each sample. The sample was subjected to the determination for color parameter at eight different locations both before and after the bleaching. Three measurements of color parameters were made, and the average reading was calculated for each location. The differences of color parameter value between pre- and postbleaching were calculated for the values of ΔL*, Δa*, and Δb*. The total variation of color was calculated using Equation 1.30

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

\[ (1) \]

where ΔL* = ΔL*Pr, Δa* = Δa*Pr, Δb* = Δb*Pr, Pr is the prebleaching value, Po is the postbleaching value.

The critical remarks of the color change (ΔE) were certified by the National Bureau of Standards (NBS), according to NBS units of color difference, which rates the way that a color change is evaluated by the human eye as described in Table 1. The formula for conversion from ΔE to NBS units shown in Equation 2 was used.10

\[
NBS = \Delta E \times 0.92
\]  

\[ (2) \]

**Statistical Analysis**

The data were statistically analyzed using Statistical Package for the Social Sciences statistical software (SPSS PC version 17, SPSS Inc., Chicago, Illinois, USA). The Kol- mogorov–Smirnov test was used to evaluate the normal distribution of data. An ANOVA was used to evaluate the effects of type of ceramic materials and bleaching techniques as well as their interaction effect on color change. Tukey multiple tests were determined for pairwise comparisons at 95% level of confidence.

**RESULTS**

The means and SD values of ΔE parameter for determination of color changes are presented in Table 2 and Graph 1. The average color change (ΔE) values for each type of ceramic materials on bleaching without and with implementing LED illumination were as follows: Lava™ Ultimate = 0.34, 0.55, Vita Enamic® = 5.59, 5.81, IPS e.max® CAD = 4.60, 6.92, inCoris® TZI = 3.43, 4.44, and Prettau® zirconia = 6.60, 7.03. Two-way ANOVA indicated significant differences in ΔE value of ceramic materials due to the effect from method of bleaching and type of ceramic materials tested (p < 0.05), as shown in Table 3. There was no significant difference in ΔE value as a result of interaction between ceramic materials and bleaching technique (p > 0.05). Post hoc Tukey multiple comparison tests indicated significant differences in ΔE value among groups of ceramic materials (p < 0.05) as shown in Table 4. The bleaching process using 35% hydrogen peroxide both without and with LED illumination results in significant color alteration of ceramics tested (p < 0.05). The bleaching technique with LED illumination resulted in

<table>
<thead>
<tr>
<th>Table 1: National bureau of standards system of expressing color differences</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>NBS units</strong></td>
</tr>
<tr>
<td>0.0 to &lt;0.5</td>
</tr>
<tr>
<td>0.5 to &lt;1.5</td>
</tr>
<tr>
<td>1.5 to &lt;3</td>
</tr>
<tr>
<td>3 to &lt;6</td>
</tr>
<tr>
<td>6 to &lt;12</td>
</tr>
<tr>
<td>≥12</td>
</tr>
</tbody>
</table>

NBS: National bureau of standard
significant increase in the change of ΔΕ value to ceramic materials compared with bleaching group without LED illumination (p < 0.05). The use of LED illumination indicated an increasing powerful bleaching efficacy for bleaching agent and resulted in more change in color of ceramic materials.

On visual perception in color change determination based on NBS unit color system, the results between the values of 0.0 and 0.5 are “trace,” the results between the values of 0.5 and 1.5 are “slight,” the results between the values of 1.5 and 3 are “noticeable,” the results between 3 and 6 are “appreciable,” and the results between 6 and 12 are “much color changes.” The resin nanoceramic (Lava™ Ultimate) exhibited trace color change on bleaching without LED implementation, but showed slight color change on bleaching with LED implementation. The hybrid ceramic (Vita Enamic®) exhibited a noticeable color change for both bleaching techniques. The lithium disilicate ceramic (IPS e.max® CAD) bleached without LED illumination exhibited appreciable color changes, but that with LED illumination showed much color change. The inCoris® TZI underwent appreciable

### Table 2: Mean, SD, and 95% CI of ΔΕ values for ceramic materials after different bleaching techniques

<table>
<thead>
<tr>
<th>Group</th>
<th>Ceramic</th>
<th>Bleaching method</th>
<th>n</th>
<th>Mean ± SD</th>
<th>LB</th>
<th>UB</th>
<th>NBS units</th>
</tr>
</thead>
<tbody>
<tr>
<td>LNL</td>
<td>Lava™ Ultimate</td>
<td>BNL</td>
<td>10</td>
<td>0.34 ± 0.21</td>
<td>0.18</td>
<td>0.47</td>
<td>0.31</td>
</tr>
<tr>
<td>LBL</td>
<td>Lava™ Ultimate</td>
<td>BL</td>
<td>10</td>
<td>0.55 ± 0.41</td>
<td>0.25</td>
<td>0.83</td>
<td>0.51</td>
</tr>
<tr>
<td>NLNL</td>
<td>Vita Enamic®</td>
<td>BNL</td>
<td>10</td>
<td>5.59 ± 1.35</td>
<td>4.61</td>
<td>6.55</td>
<td>5.14</td>
</tr>
<tr>
<td>NLBL</td>
<td>Vita Enamic®</td>
<td>BL</td>
<td>10</td>
<td>5.81 ± 1.45</td>
<td>4.76</td>
<td>6.85</td>
<td>5.34</td>
</tr>
<tr>
<td>NLNL</td>
<td>IPS e.max® CAD</td>
<td>BNL</td>
<td>10</td>
<td>4.60 ± 1.80</td>
<td>3.31</td>
<td>5.89</td>
<td>4.23</td>
</tr>
<tr>
<td>NLBL</td>
<td>IPS e.max® CAD</td>
<td>BL</td>
<td>10</td>
<td>6.92 ± 1.42</td>
<td>5.90</td>
<td>7.94</td>
<td>6.36</td>
</tr>
<tr>
<td>NLNL</td>
<td>inCoris® TZI</td>
<td>BNL</td>
<td>10</td>
<td>3.43 ± 1.04</td>
<td>2.69</td>
<td>4.18</td>
<td>3.15</td>
</tr>
<tr>
<td>NLBL</td>
<td>inCoris® TZI</td>
<td>BL</td>
<td>10</td>
<td>4.44 ± 0.72</td>
<td>3.62</td>
<td>4.66</td>
<td>4.08</td>
</tr>
<tr>
<td>NLNL</td>
<td>Prettau® zirconia</td>
<td>BNL</td>
<td>10</td>
<td>6.60 ± 0.80</td>
<td>6.03</td>
<td>7.18</td>
<td>6.07</td>
</tr>
<tr>
<td>NLBL</td>
<td>Prettau® zirconia</td>
<td>BL</td>
<td>10</td>
<td>7.03 ± 0.64</td>
<td>6.57</td>
<td>7.49</td>
<td>6.46</td>
</tr>
</tbody>
</table>

BNL: Bleaching without LED; BL: Bleaching with LED; LB: Lower bound; UB: Upper bound; CI: Confidence interval

### Table 3: An ANOVA of ΔΕ for different dental ceramic for CAD/CAM system on different bleaching techniques

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of square</th>
<th>df</th>
<th>Mean of square</th>
<th>F</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ceramic</td>
<td>564,866</td>
<td>4</td>
<td>141,217</td>
<td>146,256</td>
<td>0.000</td>
</tr>
<tr>
<td>Bleaching method</td>
<td>20,910</td>
<td>1</td>
<td>20,910</td>
<td>13,842</td>
<td>0.000</td>
</tr>
<tr>
<td>Ceramic × bleaching method</td>
<td>13,612</td>
<td>4</td>
<td>3,403</td>
<td>2,235</td>
<td>0.084</td>
</tr>
<tr>
<td>Error</td>
<td>54,801</td>
<td>36</td>
<td>1,522</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Table 4: Tukey post hoc multiple comparison of ΔΕ related to the effect of different ceramic materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Lava</th>
<th>Enamic</th>
<th>e.max</th>
<th>inCoris®</th>
<th>Prettau</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lava</td>
<td>1.000</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Enamic</td>
<td>0.000</td>
<td>1.000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>e.max</td>
<td>0.000</td>
<td>0.000</td>
<td>1.000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>inCoris®</td>
<td>0.000</td>
<td>0.000</td>
<td>0.002</td>
<td>1.000</td>
<td></td>
</tr>
<tr>
<td>Prettau</td>
<td>0.000</td>
<td>0.031</td>
<td>0.036</td>
<td>0.000</td>
<td>1.000</td>
</tr>
</tbody>
</table>

Graph 1: Color change (ΔΕ) of different ceramic materials for CAD–CAM before and after bleaching with different bleaching techniques. BNL: Bleaching without LED; BL: Bleaching with LED
DISCUSSION

The color stability is a crucial determining factor for the long-term esthetic success of dental restoration. It is necessary for restorative materials to maintain color stability over time in the oral environment. Minimal color alterations are difficult to distinguish by human eyes and need for precise instrument, such as spectrophotometer to provide reliable color comparison as the use in this study.29 The results revealed significant differences in means of color change among groups. Hence, the null hypothesis was rejected. The study indicated that the different bleaching techniques produce different effect on color alteration of ceramic materials. A significant change in color was found in every ceramic material after their exposures to bleaching. Prettau zirconia was affected the most (∆E = 7.03) while Lava™ Ultimate (∆E = 0.34) was affected the least on bleaching.

The color change following bleaching may relate to the type of ceramic materials. The amount of color change was not visually detectable for nano-resin ceramic, while it was slightly appreciable change for hybrid ceramic. Both materials consist of different filler particles that are bound with the resin matrix. The resin matrix may undergo surface degradation leading to color change.10 Hydrogen peroxide is a strong oxidizing agent that breaks down into free radicals causing oxidative cleavage to polymer chains and enhancing the hydrolytic degradation of resin matrix. The free radicals can degrade the pigments responsible for coloration of materials.20 The color change induced by hydrogen peroxide depends on not only matrix structure but also type, size, shape, and volume of fillers.21,22 The trace color change of nano-resin ceramic materials might be attributed to the presence of highly cross-linked polymer as well as lower filler content compared with hybrid ceramic. The hybrid ceramic possesses more intermolecular spaces that lead to easy hydrolysis of matrix and penetration of bleaching agent. The resin content of the ceramic may have undergone chemical disassociation and triggered the color alteration. Thus, the hybrid ceramic showed greater effect in color change than nano-resin ceramic after bleaching. This result is in agreement with those of other studies.10,13 The Vita Enamic® comprises 86% by weight of fillers, while Lava™ Ultimate possesses only 80% by weight. The greater the volume of filler particles in the composition of ceramic–polymer networks, the lower will be the degree of conversion. This results in larger number of double bonds and lower quality of bonds may be formed.9 Therefore, such composition will be more predisposed to the action of the bleaching agent, leading to plasticization action which induces greater color alteration. The hydrogen peroxide free radicals prefer to attack at the interface between inorganic filler and resin matrix; this can cause the fillers to disintegrate from the material surface.9 The cleavage effect of filler from resin matrix of these resin ceramic are also responsible for color change.

The IPS e.max CAD materials revealed appreciable changes in color on bleaching per se but much color change on bleaching with LED illumination. The reason may relate to the nature of crystal structures of lithium disilicate glass that tends to be dissolved easily by hydrogen peroxide bleaching agent. The microstructure degradation on bleaching of IPS e.max® CAD is the prime reason for color change.

The bleaching exhibited much color change to Prettau® zirconia and appreciable color change to inCoris® zirconia. The amount of color change in the zirconia ceramic may relate to the disintegration of the metal oxide content of the colorants that used to provide the ceramic color appearance. The disruption of metal oxides was initiated by peroxide and intensified by light irradiation that would likely cause the color alteration to zirconia as supported by other studies.11,13 The color alteration of zirconia ceramic also related to the spontaneous phase transformation from tetragonal (t) to monoclinic (m) phase that occurred over time, defined as low-temperature degradation (LTD).12 It is considered as a crucial factor affecting the durability and stability of metastable microstructure during the long-term performance of Y-TZP.14 This phenomenon was described to generate the loss of small zirconia grains at the surface causing surface irregularities that led to the changes in the optical and mechanical properties.12,17 The bleaching reacts at the surface of zirconia and induces degeneration of zirconia grains causing surface upraises, grain evaucation, and surface irregularity.22 The larger grains are less resistant to phase transformation but more favorable to mechanical properties.18 The grain size of the Prettau® zirconia is approximately 0.35 ± 0.03 μm, which is larger than that of inCoris® TZI that is approximately 0.21 ± 0.02 μm.16 Thus, the Prettau® zirconia tends to exhibit LTD phenomenon rather easier than inCoris® TZI and
results in exhibiting color alteration on bleaching more than InCoris® TZI.

This study indicated that the bleaching technique significantly affects color change of ceramic restorative materials. The LED illumination induced more bleaching effect to the ceramic material, and more color change was manifested. In general, the color difference value of ΔΕ >3.3 is recognized to be clinically significant and is necessary for restoration replacement. Considering the color difference values in this study, it can be concluded that bleaching causes color change clinically perceivable to every ceramic material tested except for Lava™ Ultimate.

CONCLUSION

The color stability is the particular property of each material to resist color change. The color stability of ceramic restorative materials depends on the nature of each ceramic material. Bleaching exhibited the alteration in color of dental ceramics for CAD–CAM. The technique of bleaching using 35% hydrogen peroxide with LED illumination results in more increase in color alteration of ceramic materials than bleaching without LED illumination. The bleaching indicated clinical significance on the color alteration of ceramic material.

CLINICAL SIGNIFICANCE

Dentists should be aware whether there is any ceramic restoration in the patient’s mouth before performing bleaching procedure for their patients. It is crucial to realize the type of ceramic material used for any restoration to avoid the undesirable effect on the existing restorations. Appropriate selection of bleaching technique as well as protection of the existing restoration should be considered. However, patients need to be informed of bleaching’s potential effects on the color of restoration.

ACKNOWLEDGMENT

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