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**ORIGINAL ARTICLE**

# Color change of direct resin-based composites after bleaching: An *in vitro* study

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**Abstract** This *in vitro* study was designed to quantitatively evaluate and compare color changes in different types of direct resin-based composites including a silorane-based low-shrink resin composite, after bleaching with 10% carbamide peroxide.

Fifty disc-shaped specimens (10/material) from five resin-based composites (Z250, Z350, Valux Plus, P90, and Dyract Extra) were fabricated in a cylindrical rubber mold (10 mm diameter × 2 mm depth) according to the respective manufacturers' instruction. A spectrophotometer (Color Eye 7000A) against a white background using CIE ( $L^*a^*b^*$ ) relative to a standard illuminate D65 was used to measure the color of specimens before bleaching procedure as baseline records and after bleaching procedure using (Opalescence PF-10% carbamide peroxide) 8-h daily for 14 consecutive days. The magnitude of color difference (between baseline and after bleaching measurements) is represented by  $\Delta E$ . Data were subjected to a one-way ANOVA and Bonferroni multiple comparison test at 95% confidence level.

The mean color change values ( $\Delta E$ ) were less than 1 and visually non-perceptible to the naked eye for silorane-based low-shrink composite (P90) as well as the other resin-based composites used in this study (nanofilled Z350, hybrid Valux Plus, and microhybrid Z250). Whereas, the mean color change value ( $\Delta E$ ) was higher than 1 and noticeable to the naked eye for polyacid-modified resin-based composite (Dyract Extra), yet considered clinically not significant.

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## 1. Introduction

The increasing demand by patients for a noninvasive approach of esthetic dentistry in the last decade resulted in the introduction of teeth whitening procedures. Currently, several bleaching systems are available in the dental market to be used for improving tooth color which include in-office bleaching, at-home bleaching, and over-the counter bleaching products (Haymann, 2005; White et al., 2002; Gurgun and Yalcin, 2007).

All teeth whitening procedures use either hydrogen peroxide or carbamide peroxide. Currently available home bleaching agents often contain up to 10% hydrogen peroxide or 22% carbamide peroxide as active ingredients and they are applied to the teeth via a ready-made or custom-fabricated tray (Li, 1998; Canay and Cehreli, 2003). The most common of at-home bleaching agents is 10% carbamide peroxide because of its favorable clinical results, effectiveness and safety (White et al., 2002; Li, 1998).

During at-home bleaching procedure; the bleaching agent contacts both teeth and existing restorations in the patient's mouth for extended periods of time. Several studies were conducted to investigate the effect of at-home bleaching agents on surface microhardness, roughness, and color stability of tooth-colored restorative materials (Canay and Cehreli, 2003; Cehreli et al., 2003; Yu et al., 2008; Polydorou et al., 2007; Bailey and Swift, 1992). Color changes of such existing restorations, recently placed or old, may compromise esthetics.

Some patients with recently placed direct composite restorations would seek teeth whitening soon after having such restorations. A question has arisen as to whether the whitening degree of such restorations achieved by such safe and effective type of bleaching agent (10% carbamide peroxide) would create an acceptable color match similar to the surrounding bleached teeth, with no need for composite replacement or veneering.

Understanding the effect of at-home bleaching agents on recently placed direct resin-based composite restorations is of paramount importance. Reviewing the literature reveals no information on such effect on silorane-based low-shrink composites.

The purpose of this *in vitro* study was to quantitatively, evaluate and compare color changes in different types of direct resin-based composites; simulating recently placed restorations; after bleaching with 10% carbamide peroxide.

## 2. Materials and methods

### 2.1. Specimens fabrication

Five different types of resin-based composite materials were used in this study (Table 1). These materials included silorane-based low-shrink, hybrid and microhybrid, as well as nano-filled and polyacid-modified resin-based composites. Fifty disc-shaped specimens, 10 from each material, were fabricated using shade A3 in a cylindrical rubber mold (10 mm diameter  $\times$  2 mm depth) according to the respective manufacturer's instructions. Specimens were fabricated each by filling the rubber mold with composite resin, covered with Mylar strips and pressed between two glass plates. Light polymerization was performed for 40 seconds at the top and bottom surfaces using a halogen light (Elipar™ 2500, 3M ESPE) at 500 mW/cm<sup>2</sup>-verified with a Model 100 Curing Radiometer. The distance between the light tip and the specimen surfaces was standardized by touching the glass slide. The specimens were polished using medium, fine, and ultrafine polishing discs (Sof-Lex, 3M ESPE) with a slow-speed handpiece rotating in one direction. The final thickness of polished specimens was  $2 \pm 0.1$  mm, which was verified by a micrometer (Ultra-Cal Mark III, Fowler Tools and Instruments, Sylvac, Swiss made).

### 2.2. Color testing

A spectrophotometer (Color Eye 7000A, Gretag Macbeth LLC, New Windsor, NY, USA) against a white background using CIE ( $L^*a^*b^*$ ) relative to a standard illuminate D65 was used to measure the color of specimens. The color was measured before bleaching procedure as a baseline and also after bleaching for 14 consecutive days. In this scheme, the color was measured in three coordinate dimensions of  $L^*$  (lightness),  $a^*$  green-red ( $-a^*$  = green;  $+a^*$  = red) and  $b^*$  blue-yellow ( $-b^*$  = blue;  $+b^*$  = yellow).

**Table 1** Materials used in this study.

| Material       | Composition   | Material                          | Manufacturer       |
|----------------|---|-----------------------------------|--------------------|
| Filtek™ Z250   | Matrix: Bis-GMA, UDMA, and Bis-EMA<br>Filler: zirconia/silica (0.01–3.5 $\mu$ m)<br>Filler volume: 60%  | Microhybrid resin composite       | 3M ESPE            |
| Filtek™ Z350   | Matrix: Bis-GMA, UDMA, TEGDMA, and Bis-EMA<br>Filler: combination of aggregated zirconia/silica cluster filler (0.6–1.4 $\mu$ m) and non-aggregated 20 nm silica filler<br>Filler volume: 59.5% | Nanofilled resin composite        | 3M ESPE            |
| Filtek™ P90    | Matrix: new ring-opening Silorane<br>Filler: inorganic filler (0.1–2 $\mu$ m)<br>Filler volume: 55%   | Low-shrink resin composite        | 3M ESPE            |
| Valux Plus™    | Matrix: Bis-GMA and TEGDMA<br>Filler: single filler 100% zirconia/silica (0.01–3.5 $\mu$ m)<br>Filler volume: 66%   | Hybrid resin composite            | 3M ESPE            |
| Dyract Extra   | Matrix: UDMA, carboxylic acid modified dimethacrylate resin, and TEGDMA<br>Filler: strontium fluorosilicate glass (0.8 $\mu$ m)<br>Filler volume: 50%   | Polyacid-modified resin composite | Dentsply Caulk     |
| Opalescence PF | 10% carbamide peroxide  | At-Home Bleaching                 | Ultradent products |

Bis-GMA: bisphenol-glycidyl methacrylate; UDMA: urethane dimethacrylate; Bis-EMA: bisphenol-A polyethylene glycol dietherdimethacrylate; TEGDMA: triethylene glycol dimethacrylate.

### 2.3. Baseline measurements

Before baseline color measurement, specimens were rinsed with distilled water for 1 min and blotted dry with gauze. Three measurements at the top surface were taken for each specimen at three separate non-overlapping areas to ensure a representative assessment, and the mean was calculated.

### 2.4. Bleaching procedure and color measurements

The specimens were treated with bleaching gels (Opalescence PF, 10% Carbamide Peroxide, Ultradent Products) for 8-h daily, for 14 consecutive days at 37 °C humidity to simulate at-home bleaching (Leonard et al., 1998). The bleaching gel was applied on the top surface of each specimen where the thickness of the gel was 1 mm. After the 8-h bleaching procedure, the specimens were fully cleaned under running water, and then stored in distilled water at 37 °C humidity for the remaining time (16 h). At the end of 14-day bleaching procedure, all the specimens were removed from the bleaching gel, cleaned under running water and then stored in distilled water for 24 h at 37 °C humidity. Prior to color measurement, the specimens were removed and blotted dry with gauze, then three measurements at the top surface of the specimen were taken as previously described for the baseline measurement.

The magnitude of total color difference (between baseline and after bleaching measurements) is represented by a single number  $\Delta E$  (Commission Internationale de L'Eclairage, 1979), where

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

A limit of  $\Delta E$  value  $\leq 3.3$  is considered clinically insignificant (Ruyter et al., 1987). Color changes ( $\Delta E$ ) have been qualified by the National Bureau of Standards (NBS), according to NBS units of color difference (Table 2) (Shotwell et al., 1992). NBS units are expressed by the following equation:

$$\text{NBS unit} = \Delta E \times 0.92$$

The NBS units were calculated to reveal whether the color changes of the specimens were clinically detectable.

### 2.5. Statistical analysis

All statistical analyses were carried out using SPSS statistical software. Data were subjected to one-way ANOVA and Bonferroni multiple comparison test at 95% confidence level.

**Table 2** National Bureau of Standards (NBS) system of expressing color differences.

| Critical remarks of color difference | $\Delta E$ NBS units |
|--------------------------------------|----------------------|
| Trace                                | From 0.0 to < 0.5    |
| Slight                               | From 0.5 to < 1.5    |
| Noticeable                           | From 1.5 to < 3.0    |
| Appreciable                          | From 3.0 to < 6.0    |
| Much                                 | From 6.0 to < 12.0   |
| Very much                            | $\geq 12.0$          |

**Table 3** Means, standard deviations and NBS units of color change of tested direct resin-based restorative materials after 14 days of bleaching.

| Materials    | Bleaching            |                      |
|--------------|----------------------|----------------------|
|              | Mean $\Delta E$ (SD) | $\Delta E$ NBS units |
| Filtek™ Z250 | 0.523 (0.238)        | 0.481 (trace)        |
| Filtek™ Z350 | 0.882 (0.539)        | 0.811 (slight)       |
| Filtek™ P90  | 0.722 (0.330)        | 0.664 (slight)       |
| Valux Plus™  | 0.733 (0.291)        | 0.674 (slight)       |
| Dyract Extra | 1.936 (0.588)        | 1.781 (noticeable)   |

$\Delta E$ : Total color change after 14 days of bleaching.

SD: Standard deviation.

NBS units: color change according to National Bureau of Standards system.

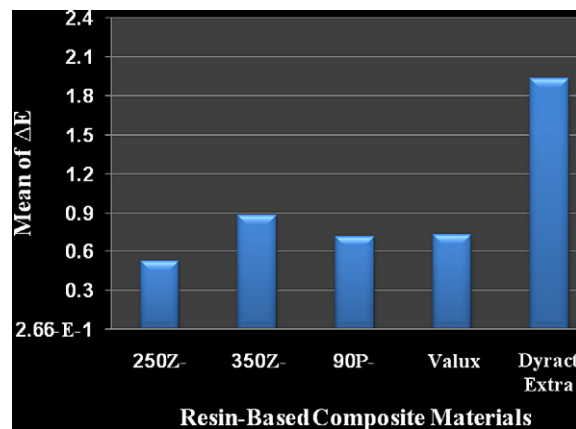
## 3. Results

The means of color change ( $\Delta E$ ) along with standard deviations (SD), and (NBS) units of the five different types of resin-based composites after 14 days of bleaching are presented in Table 3 and Fig. 1.

One-way ANOVA revealed that the color differences were statistically significant among all bleached groups ( $p = 0.0001$ ).

Bonferroni multiple comparison test at 95% confidence level showed that the mean color change value ( $\Delta E$ ) for Dyract Extra was significantly higher than those values for Filtek™ Z250, Filtek™ Z350, Filtek™ P90, and Valux Plus™ and where the  $p$  values were 0.0001, 0.008, 0.002, and 0.002, respectively. There was no significant difference in color change values among Filtek™ Z250, Filtek™ Z350, Filtek™ P90, and Valux Plus™ where ( $p = 1.000$ ).

After conversion of the mean  $\Delta E$  values to NBS units (Table 3), it was found that Dyract Extra (polyacid-modified resin-based composite) had “noticeable” color change and was the highest among the tested materials after bleaching, followed by Filtek™ Z350 (nanofilled resin-based composite), Valux Plus™ (hybrid resin-based composite), Filtek™ P90 (low-shrink silorane-based composite). While, Filtek™ Z250



**Figure 1** Mean  $\Delta E$  of different types of resin-based composites after 14 days of bleaching.

(microhybrid resin-based composite) had “trace” color change and was the lowest among the tested materials after bleaching.

#### 4. Discussion

Color changes can be evaluated using a visual method and color measurement devices. Most of the color measurement devices utilized in dentistry use the  $\Delta E$  from the Commission International de l'Éclairage CIE ( $L^*a^*b^*$ ) color system to determine the color differences or changes. In this scheme, color is measured in three coordinate dimensions of  $L^*$  which represents lightness (from white to black; similar to value),  $a^*$  corresponds to the green-red axis (negative value indicates green; positive value indicates red), and  $b^*$  corresponds to the blue-yellow axis (negative value indicates blue; positive value indicates yellow) (Chu et al., 2004).

The total color ( $E$ ) is computed for all three spectral values, and obtained by the following equation:  $E = (L^* + a^{*2} + b^{*2})^{1/2}$ . The total color change is described by  $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$  (Commission Internationale de l'Éclairage, 1979; Villalta et al., 2006). Several studies have shown that color differences greater than one unit ( $\Delta E > 1$ ) can be perceptible visually by 50% of human observers (Canay and Cehreli, 2003; Seghi et al., 1990, 1989), and the general population can distinguish color differences of value ( $\Delta E > 3.3$ ) and are considered clinically significant (Ruyter et al., 1987).

Since the introduction of bleaching by Haywood and Heymann (1989), the use of bleaching agents has become increasingly popular for whitening stained teeth. These products contact tooth structures for extended periods of time especially during at-home bleaching treatment; however, it is unavoidable to prevent restorations from exposure to bleaching agents (Celik et al., 2009).

In this present study, 10% carbamide peroxide bleaching agent was applied for 14 days on different types of resin-based composite materials. Results showed that the amount of color change was not visually detectable for microhybrid, nanofilled, hybrid, and silorane-based low-shrink resin-based composites used in this study, while it was slightly noticeable for polyacid-modified resin-based composite. This finding was in agreement with that of a previous study (Canay and Cehreli, 2003), whereby 10% carbamide peroxide was used to bleach macrofilled, hybrid, and polyacid-modified resin-based composites.

The result of this study indicated that color change induced by 10% carbamide peroxide agent might be dependent upon matrix structure, filler volume, as well as the filler type of different types of resin-based composites tested. This finding is in agreement with that of Hubbezoğlu et al. (2008).

The highest and noticeable color change of polyacid-modified resin-based composite (Dyract Extra) compared to other resin-based materials used in this study might be attributed to the presence of carboxylic acid modified dimethacrylate resin in matrix, reactive strontium fluorosilicate glass and lower filler content. This result is in agreement with those of other studies (Canay and Cehreli, 2003; Rosentritt et al., 2005).

The mean of color changes ( $\Delta E$ ) of the other tested resin-based composite materials ranked from the highest to the lowest (nanofilled, hybrid, silorane-based low-shrink and microhybrid) was less than one unit and undetectable by the naked eye. The color change of nanofilled (Z350) and hybrid (Valux Plus) resin-based composites was higher than that of

microhybrid type (Z250). This might be related to the presence of Bis-GMA and/or UDMA resin matrix polymers in nanofilled (Z350), hybrid (Valux Plus), and microhybrid (Z250) resin-based composites (Canay and Cehreli, 2003; Hubbezoğlu et al., 2008), and the absence of a diluent monomer (TEGDMA) in microhybrid (Z250).

The color change of silorane-based low-shrink (P 90) resin-based composite was less than nanofilled (Z350) and hybrid (Valux Plus) types. This might be attributed to the resistance of inorganic filler and/or new ring-opening silorane resin matrix to bleaching. On the other hand, lower volumetric filler content of low-shrink (P90) resin-based composite compared to microhybrid type (Z250) could be the reason for higher degree of oxidation and hydrolytic degradation of silorane-based low-shrink resin matrix by hydrogen peroxide (Park et al., 2004); which is the active ingredient in carbamide peroxide home bleaching agents; leading to higher color change of low shrink compared to microhybrid resin-based composites.

Another study showed that bleaching agents can successfully remove the exterior staining from composite resins; therefore, they should be used cautiously to remove the exterior stain on the surface of composite resin restorations (Gerlach et al., 2000).

Based on the results obtained in this study, it could be pointed out that the low concentrate 10% carbamide peroxide home bleaching gels had a noticeable color change effect on polyacid-modified resin-based composite and slight to trace color change on the other types of resin-based composites. This finding was in agreement with that of a previous study (Rao et al., 2009), which showed that a low concentration of bleaching agents had an influence on the color of tooth-colored restorative materials. This color change may be clinically perceptible for some materials such as compomer or glass ionomer cement, and produce a better color match with natural teeth after bleaching, whereas it may not be clinically perceptible for other tooth-colored restorative materials such as resin composites (Yu et al., 2009).

#### 5. Conclusions

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. The mean color change values ( $\Delta E$ ) were less than 1 and non-perceptible visually to the naked eye for the silorane-based low-shrink composite (P90) as well as the other resin-based types of composite used in this study (nanofilled Z350, hybrid Valux Plus, and microhybrid Z250).
2. The mean color change value ( $\Delta E$ ) of polyacid-modified resin-based composite (Dyract Extra) was higher than 1 and noticeable to the naked eye yet considered clinically insignificant.
3. Low concentrate 10% carbamide peroxide home bleaching agents may change the color of the existing direct tooth-colored restorations; whether recently placed or old, to match that of home bleached teeth.

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