Effects of bleaching agent on physical and aesthetic properties of restorative materials

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To investigate the effects of bleaching agent on microhardness, color and light transmission of different restorative materials. Specimens (n=20) of Tetric EvoCeram (TEC), Tetric EvoCeram Bulk Fill (TECBF) and Equia Fill (EQUIA) were treated with either 40% hydrogen peroxide Opalescence Boost or destilled water for 45 min. Specimens were stained in tea solution or stored in deionized water for one and two weeks. Color, microhardness and light transmission were monitored at the baseline, after the bleaching and after the tea immersion or storage in deionized water. After the bleaching a significant reduction in surface microhardness (p<0.001) was recorded for all materials. Clinically visible color change (ΔE>3) was observed after the bleaching and after treatment in tea solution, but only in EQUIA samples. The absorption coefficient was the largest for the samples stained in tea solution. Bleaching can affect the microhardness and color of fillings; therefore, they should be sometimes replaced.

Keywords: Bleaching agents, Composite material, Glassionomer cement, Light transmission, Microhardness

INTRODUCTION

In the last decades, interest in tooth whitening has been growing as it is considered the easiest and most cost-effective procedure for treating tooth discoloration. Latest research shows that the outcome of whitening the vital and non-vital teeth is successful if carried out in controlled conditions⁴. Teeth whitening procedures have proved to be safe in terms of hard dental tissues, and so far macroscopic and clinically visible changes caused by whitening procedure have not been reported²⁸–³⁰. Studies have shown microscopic damage of hard dental tissue⁹, change in histology and chemical composition with the reduction in surface microhardness⁷,⁸ caused by the use of hydrogen or carbamide peroxide for tooth whitening. However, these changes were minimal and not clinically significant⁹,¹⁰.

There is also an interest and potential concern about the effects of whitening on the physical, mechanical and chemical properties of restorative materials¹¹. Amalgam fillings release mercury when being exposed to carbamide peroxide for a long time. The amount of released mercury varies depending on the type of amalgam and bleaching agents. Ceramics and gold generally do not show any changes during bleaching²⁹.

The components of composite materials such as monomers or acids in the glass ionomer cements may be susceptible to bleaching components so changes in their physical, mechanical and aesthetic properties can occur. The most frequently reported is the change in their color after the bleaching²⁸, as well as softening of the surface and the increase of surface roughness with pits and fissures formation⁴. Due to the presence of organic resins in its composition, composite materials have shown a significant change in chemical structure after the bleaching²⁰. In addition to the changes in chemical composition and mechanical properties, hydrogen peroxide can also lead to the discoloration of restorative materials¹⁶,¹⁷. The ideal composite material should remain unchanged after the tooth whitening⁹. Some studies demonstrated a significant increase in surface roughness and porosity of the composite material⁹, while others showed no damage⁶,²¹. Changes in the surface microhardness of restorative materials depend on the application of bleaching agents as well as on the type and properties of the material¹³. SEM microscopy showed that the use of 10–16% carbamide peroxide cause slight, but statistically significant increase in the surface roughness and porosity²².

Applications of hydrogen and carbamide peroxide could lead to significant changes in color of composite materials, especially microfilled and nanofilled composites²³, and these changes can be attributed to the volume of the organic matrix and filler type²⁴. Hydrogen peroxide contributes to increased brightening of composite materials compared to carbamide peroxide of the same concentration²⁰. Also, the changes in color and light transmission of restorative materials after the bleaching may be attributable to the surface amine oxidation, also responsible for the change in the color of composite due to aging, or to the difference in the amount of organic matrix as well as to the degree of conversion. Therefore, the greater change in color was observed in the chemically cured composites²⁵. One more concern is that bleached glass ionomer and composite restorations are more susceptible to different staining solutions, and
get more discolored than unbleached ones.

The aim of this in vitro study was to evaluate the effects of bleaching agent for professional use on surface microhardness, color and light transmission of three restorative materials: conventional high viscosity composite material, bulk fill composite and glassionomer cement, and to assess their color stability after later tea exposure.

**MATERIALS AND METHODS**

**Specimen preparation**

Table 1 describes the materials used in this study. A total of 120 disk-shaped specimens were prepared with a high-viscosity bulk-fill resin composite [Tetric EvoCeram Bulk Fill —TECBF, IVA shade (Ivoclar Vivadent, Liechtenstein)], conventional nano-hybrid composite material [Tetric EvoCeram —TEC, A2 shade (Ivoclar Vivadent)] and high-viscosity glass ionomer cement [Equia Fil —EQUIA, A2 shade (GC, Japan)] with low-viscosity nanofilled surface coating resin Equia Coat (GC, Japan). Each material was inserted into circular teflon molds (9 mm diameter×2 mm thick and 9 mm×1 mm thick) using a metal spatula. A mylar strip (3M ESPE, USA) was placed on top and pressed flat with a microscope slide plus a 500 g weight for 30 s to expel excess material and create a smooth surface. Composites were then irradiated for 20 s with a polywave LED curing unit (Bluephase Style, Ivoclar Vivadent). The output light intensity of the curing unit (1,100 mW/cm²) was measured using calibrated Bluephase Meter (Ivoclar Vivadent), and verified periodically during the experiments. The discs were removed from the molds and stored in an incubator (Culture, Ivoclar Vivadent) filled with deionized water for 24 h while maintaining the temperature of 37°C to allow thorough leaching of any non-polymerized resins and to establish equilibrium in water uptake. Subsequently, the specimens upper surface was polished using a water cooled carborundum disc (Water Proof Silicon Carbide Paper, 4000 grit, Buehler, Dusseldorf, Germany) and 1.0, 0.3 and 0.05 μm microparticle powder (Buehler). The polished discs were then stored in deionized water for 24 h before being used.

Sixty samples of 2 mm thickness were prepared and randomly assigned to different groups (TEC, TECBF or EQUIA). The color change and microhardness was measured before, after the bleaching and after one- and two-week storage in tea solution or denionized water. Also, sixty samples of 1 mm thickness were prepared and randomly assigned to different groups (TEC, TECBF or EQUIA) to measure the change in light transmission before and after the bleaching, and after one- and two-week storage in tea solution or denionized water.

**Bleaching procedure**

Samples of each material (n=20) were randomly divided into two groups (n=10) before the bleaching process. First group was treated with 40% hydrogen peroxide Opalescence BOOST (Ultradent, South Jordan, USA)

| Restorative material (code) | Manufacturer Shade/LOT | Resin composition | Acid composition | Filler amount (wt%/vol%), composition and size | Manufacturers' recommended composite layer thickness (mm) | Manufacturers' recommended curing time |
|---------------------------|------------------------|------------------|------------------|-----------------------------------------------|-----------------------------------------------------|-------------------------------------|
| Tetric EvoCeram (TEC)     | Ivoclar Vivadent, Schaan, Liechtenstein | A2/P80726 | Bis-GMA, Bis-EMA, UDMA | — | 76/55 Barium glass, YbF3, mixed oxide, PPF (0.04–3 μm) | ≥1,000 mW/cm²/10 s; ≥500 mW/cm²/20 s |
| Tetric EvoCeram Bulk Fill (TECBF) | Ivoclar Vivadent | IVA/R04686 | Bis-GMA, Bis-EMA, UDMA | — | 81/61 Barium glass, YbF3, mixed oxide, PPF (0.04–3 μm) | ≥1,000 mW/cm²/10 s; ≥500 mW/cm²/20 s |
| Equia (EQUIA) GC, Japan | A2/1405211 | 40% aqueous polyacrylic acid | — | 95% strontium fluoroaluminosilicate glass, 5% polyacrylic acid | 4 | — |
| Equia Coat GC | —/1504171 | Methyl methacrylate | — | 0.09% camphorquinone | — | 20 s |

Bis-GMA: bisphenol-A-glycidyldimethacrylate, Bis-EMA: ethoxylated bisphenol-A-dimethacrylate, PPF: prepolymerized fillers, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate, YbF3: Ytterbiumtrifluoride, NP filler composition and size not provided.
gel applied with Heideman spatule in 2 mm thick layer during 3×15 min time period. After the first 15 min, bleaching gel was removed with Heideman spatule and cotton pallet and the surface was cleaned with deionized water, dried with compressed air and cotton tissues and another layer of bleaching gel was put on the surface. During the bleaching treatment, the specimen was placed on the cotton pallet soaked with deionized water. Second group (n=10) formed a control, in which the specimens were kept in deionized water for 45 min. The same bleaching process was used in samples for microhardness measurements as well as for the color and light transmission measurements.

**Immersion in staining solutions**

After performing the bleaching treatment, specimens in each group (n=10) were allocated into two subgroups (n=5). The first subgroup was stained in tea solution made from 2 g of green tea (Cedevita, Croatia) boiled in 100 mL of deionized water for 5 min\(^2\)\(^2\). Specimens were immersed in tea solution for 3 h/day, at room temperature, over the 14 days. After every 3-h period of immersion time, the specimens were rinsed thoroughly and stored in deionized water at 37°C. The second subgroup served as negative control and the specimens were kept in deionized water during the observation period.

**Microhardness measurements**

Microhardness was measured by a Vickers diamond (Leitz Miniload 2 Microhardness Tester, Leitz, Germany) at a load of 100 g applied for 10 s. For each specimen, three indentations were performed with the distance of 100 μm between them, and averaged. Indentations were performed before, at the end of bleaching treatment and after two-week storage in tea solution or deionized water.

**Color and light transmission measurement**

Spectrophotometers and colorimeters are usually used to determine the color of restorative materials as the standardized and reliable tools based on the CIELAB system. In clinical terms, the CIE-Zahnfabrik, Bad Säckingen, Germany) according to using a spectrophotometer (VITA Easyshade, VITA Säcken, TECBF and EQUIA) and type of treatment (no treatment, bleaching, tea and deionized water) were defined as factors in the ANOVA model. The Anderson-Darling test (p>0.25) indicated that the residuals of microhardness model were approximately normally distributed, while Brown-Forsythe's test did not reject the assumption of variance homogeneity (p=0.368). The opposite results were obtained for measurements of color change. Box-Cox test suggested that those measurements should be log-transformed prior to analysis. Therefore, analysis of color change measurements was performed on logged values which satisfied the assumptions of normality and variance homogeneity (Anderson-Darling test p=0.217; Brown-Forsythe’s test p=0.985). Before reporting, the values of color change were transformed back to the original scale by using exponential transformation.

The significance level was set at 0.05. p-Values were adjusted for multiple comparisons according to the Benjamini-Hochberg method, controlling the false discovery rate. Analysis was performed using SAS System, version 8.2 (SAS Institute, NC, USA).

**RESULTS**

**Microhardness measurements**

Average microhardness measurements and associated 95% confidence intervals for tested materials —TEC, TECBF and EQUIA recorded after different treatments— baseline (initial measurements), bleaching (with 40% Opalescence BOOST for 45 min), tea (two weeks of staining in tea) and water treatment (two weeks in deionized water) are presented in Fig. 1. Baseline microhardness measurements for EQUIA (HV=6.5) were on average considerably lower than for TECBF (HV=7.3; p<0.001) and TEC (HV=7.4; p<0.001)

The measurements were repeated three times. All measurements were conducted on a white paper by the same investigator, and under the same laboratory conditions at +20°C.

The absorption coefficient of the material was experimentally measured using a white LED light source (9W, LED Engin, San Jose, USA), sample holder, optical fiber and spectrometer. Disk shaped sample (9×1 mm thick) was set between the white LED and optical fiber and the light was guided to the spectrometer HR4000, resolution 0.25 nm (OceanOptics, Dunedin, USA). The light transmission through the samples was measured before, at the end of bleaching treatment and after one and two weeks of storage in tea solution or deionized water. Based on the intensity of light detected in the spectrometer I(λ), and the initial intensity I₀(λ), which actually represents the unab sorbed spectrum of white LED, the absorption coefficient was calculated from the Beer-Lambert law as follows:

\[
I(\lambda) = I_0(\lambda) \cdot e^{-k(\lambda) \cdot \chi}
\]

where \(\chi\) is the length of light through the sample (its thickness), and \(k(\lambda)\) is the absorption coefficient.

**Statistical analysis**

Two-way analysis of variance (ANOVA) model was used to assess the changes in microhardness and color. The type of material (TEC, TECBF and EQUIA) and type of treatment (no treatment, bleaching, tea and deionized water) were defined as factors in the ANOVA model. The Anderson-Darling test (p>0.25) indicated that the residuals of microhardness model were approximately normally distributed, while Brown-Forsythe’s test did not reject the assumption of variance homogeneity (p=0.368). The opposite results were obtained for measurements of color change. Box-Cox test suggested that those measurements should be log-transformed prior to analysis. Therefore, analysis of color change measurements was performed on logged values which satisfied the assumptions of normality and variance homogeneity (Anderson-Darling test p=0.217; Brown-Forsythe’s test p=0.985). Before reporting, the values of color change were transformed back to the original scale by using exponential transformation.

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A significant reduction in microhardness was observed after the bleaching treatment which was on average the largest for TECBF (ΔHV=−1.6; p<0.001) and of equal, but lower, intensity when using EQUIA and TEC (ΔHV=−1.3; p<0.001). After two-week treatment in tea the average microhardness was significantly reduced compared to its baseline values—the largest difference was observed for TEC (ΔHV=−1.1; p<0.001), followed by TECBF (ΔHV=−0.6; p<0.001) and EQUIA (ΔHV=−0.5; p=0.043). On the other hand, after two-week treatment in deionized water a significant decrease in the average microhardness values compared to baseline measurements was observed for EQUIA (ΔHV=−0.5; p=0.035) only.

Color measurements

Color change was tested for the following treatments: bleach (bleaching with 40% Opalescence BOOST for 45 min), water (45 min in deionized water), tea7 (seven days in tea), bleach_water7 (bleaching for 45 min and seven days in tea), bleach_teat7 (bleaching for 45 min and seven days in tea), bleach_water14 (bleaching for 45 min and 14 days in deionized water), tea14 (14 days in tea), water14 (14 days in deionized water), bleach_teat14 (bleaching for 45 min and 14 days in tea) and bleach_water14 (bleaching for 45 min and 14 days in deionized water).

Table 2  Comparison of microhardness among different materials and different treatments

| Material | Baseline measurements | Bleaching | Tea | Deionized water |
|----------|-----------------------|-----------|-----|-----------------|
|          | Mean | SD 1 | Mean | SD 1 | Mean | SD 1 | Mean | SD 1 |
| TECBF    | 7.26 | A  0.39 | 5.68 | B  0.41 | 6.65 | C  0.65 | 7.00 | A,C 0.32 |
| EQUIA    | 6.48 | D  0.59 | 5.15 | E  0.66 | 6.02 | F  0.51 | 6.00 | F  0.51 |
| TEC      | 7.39 | A  0.50 | 6.06 | F  0.40 | 6.26 | F  0.57 | 7.20 | A  0.53 |

Note: The same letters in a row indicate the absence of statistically significant differences between treatments, while the same letters in column indicate the absence of statistically significant differences between the various materials.

1 SD=Standard Deviation.
ΔL*=0.72, Δa*=0.10, Δb*=1.42). The recorded values of ΔE were on average 1.9 times higher for tea treatment (tea14) compared to bleaching only (bleach; p=0.004), 1.7 times higher compared to bleaching with two-week tea treatment (bleach_tea14; p=0.049) and 1.9 times higher compared to two-week treatment in deionized water only (water14; p=0.013). Comparison of other treatments did not reveal significant differences in color change of TEC material. On the other hand, different treatments had more diverse effects on the color change of EQUIA. Only comparisons of two-week tea treatment after the bleaching (bleach_tea14) with similar seven-day treatment (bleach_tea7) and two-week treatment in tea only (tea14) did not show significant differences in change of EQUIA color. The largest difference in change of EQUIA color was observed for seven-day tea treatment after the bleaching (bleach_tea7; ΔL*=−6.66, Δa*=0.08, Δb*=10.08) in comparison to the similar water treatment (bleach_water7; ΔL*=0.68, Δa*=−1.06, Δb*=-2.08). The tea treatment resulted in 4.7 times higher average ΔE values (p<0.001).

Comparison of TECBF and TEC did not reveal statistically significant differences in color change for different types of treatment. However, when using EQUIA significantly larger color differences (ΔE) were detected compared to TECBF and TEC after all treatments except the treatment in deionized water (water). The largest difference in color change between EQUIA and TECBF was observed after the bleaching and seven-day tea treatment (bleach_tea7; ΔL*=−6.66, Δa*=0.08, Δb*=10.08 for EQUIA and ΔL*=−0.02, Δa*=−0.22, Δb*=1.56 for TECBF) with an average 5.0 times higher ΔE values recorded for EQUIA (p<0.001). On the other hand, the largest difference in color change between EQUIA and TEC was observed after the bleaching and two-week tea treatment (bleach_tea14; ΔL*=−9.40, Δa*=−0.98, Δb*=10.44 for EQUIA and ΔL*=−1.18, Δa*=−0.92, Δb*=1.08 for TEC) with recorded ΔE values on average 5.5 times higher for EQUIA (p<0.001).

Transmission measurements
The samples of TECBF and TEC measured after polymerization in the visible spectrum (400–800 nm) showed the lower absorption coefficient, i.e. the better light transmission, than EQUIA sample (Figs. 3–5). Generally, bleached and unbleached samples that were kept in deionized water showed similar absorption coefficients. The largest absorption coefficient produced the samples stained in tea solution. There was not much difference in the absorption coefficient between bleached and unbleached samples stained in tea. EQUIA samples showed a greater difference in absorption coefficient among different treatments used than TECBF and TEC samples.
DISCUSSION

Restorative material should remain unchanged during the bleaching process, but changes in surface microhardness or color still remains controversial. Microhardness of the material depends on its type, composition, and propensity to degrade over time. Contradictory findings regarding the effects of bleaching on surface microhardness of restorative materials may be attributed to the composition, concentration, and pH of the active agent as well as to the application time. Bleaching agents are unstable and can release free radicals, which stimulate the attenuation of polymer chains and cracking of double bonds. Also, hydrogen peroxide has the capability of diffusion while the free radicals can affect the filler and the resin compound thereby resulting in microcracks. Apart from the type of bleaching agent, changes in the composition of the composite material depend on the differences in the composition of the organic matrix and the size and quantity of filler particles. TECBF and TEC have the same resin in its composition (Bis-GMA, Bis-EMA, UDMA), but the difference in filler contain in TECBF which is 81/61 (wt%/vol%) compared to 76/55 (wt%/vol%) in TEC can affect the increased absorption of water and potentially lead to cracking of bonds between fillers and resins thereby causing microhardness reduction. Our study reported a significant reduction in average surface microhardness after 45 min of bleaching with high concentrate of hydrogen peroxide, with the largest being recorded for nanohybrid composite TECBF. The microhardness reduction for high-viscosity glass ionomer EQUIA and nanohybrid composite TEC was slightly lower. Similar was found by Kamangar et al. who recorded a significant reduction in the microhardness of the surface of nanohybrid and microhybrid composite material after using 15% carbamide peroxide for two weeks and 40% hydrogen peroxide for 20 min. Al-Qahtani recorded greater microhardness reduction in microhybrid siloranes and nanohybrid composites than in microhybrid composites after application of 10% carbamide peroxide. Baroudi et al. and Li et al. have demonstrated that both higher and lower hydrogen or carbamide peroxide concentrations cause time-dependent degradation of the conventional resin-modified glass ionomer cements. High viscosity glass ionomer cement (EQUIA) used in our study demonstrated significant microhardness reduction after the bleaching which could be attributed to the existence of air bubbles within the material during the mixing process. Mair and Joiner noted that there is no change in the material due to the rupture of primary bonds or that these changes are reversible due to softening of the surface because filler particles are surrounded by a hydrogel matrix within the glass ionomer cement. In addition, the hydrogel matrix is permeable and allows the penetration of bleaching agent. Taher concluded that bleaching agents generally lead to softening and reduction of surface microhardness. The same was found in our study where the application of 40% hydrogen peroxide for 45 min led to a reduction in surface microhardness of all tested restorative materials. Contrary to our study, it had been shown that bleaching with low and high concentrations of hydrogen and carbamide peroxide does not lead to surface microhardness reduction. Polydorou et al. also concluded that bleaching treatment does not lead to the significant reduction in microhardness of composite materials and therefore there is no need for replacing a composite restoration after bleaching process. Yu et al. also did not report a significant reduction in microhardness of nanohybrid composite after application of 15% carbamide peroxide, which can be attributed to the dilution effect of saliva which can minimize the effect of the bleaching agent.

In addition to the bleaching agents, various solutions such as coffee (pH=5.5) and wine (pH=4.2) can cause staining of restorations, which can result in the reduction of surface microhardness and can be attributed to acidity. Similar was found in our study where staining in tea for 14 days led to the significant reduction in surface microhardness of TEC, TECBF and EQUIA, but with lower intensity than during the bleaching. The green tea has pH of 7, which should not contribute to the reduction in surface microhardness; the observed effect may be attributed to a long and everyday exposure to tea and potential hydrolytic degradation of the composite resins and fillers.

Aesthetic properties of restorative materials allow them to fully replace the natural tooth, which is associated with the matching color of material and tooth. Change in color of material is the result of a number of intrinsic and extrinsic factors. Intrinsic factors, such as types and properties of the organic matrix and incomplete polymerization have a very large effect on the final color change. This change is associated with the degradation of the resin-filler bonds, as well as degradation and increased solubility of resin alone. Extrinsic factors such as water absorption, eating habits, smoking, and poor oral hygiene can also contribute to color change. Li et al. recorded change in the color of nanohybrid composite after the bleaching with 15% carbamide peroxide, and similar changes were observed when using 35% hydrogen peroxide in microfilled composites. These changes can be attributed to the amount of resin and particle size. Kamangar et al. concluded that bleaching with 15% carbamide peroxide and 40% hydrogen peroxide does not lead to the significant changes in color of nanohybrid and microhybrid composite materials which was similar to the results obtained in our study. There are still controversies upon which ΔE color change becomes clinically significant, i.e. visible. Some researchers believe that the changes must be greater than ΔE=2, or ΔE=3 or even ΔE=3.7. Nanofilled composite materials possess greater color stability due to the larger total surface area and the greater filler particles. De Andrade et al. showed that 35% hydrogen peroxide affects the color of nanohybrid composite as it had become brighter. In our study, according to ΔE,*
After two-week tea treatment without bleaching in darkening effects were recorded for TEC material further two-week treatment in tea compared to the brighter (ΔE and Δa*) and more yellow after the bleaching and particularly tea treatments had significantly larger effects on color of EQUIA material compared to TECBF and TEC. Staining in tea for one and two weeks produced statistically significant and clinically visible differences in color (ΔE>3), but the corresponding values of ΔE were not that higher from the critical threshold (ΔE~3). However, bleaching, and particularly tea treatments had significantly larger effects on color of EQUIA material compared to TECBF and TEC. Staining in tea for one and two weeks produced statistically significant and clinically visible differences in color (ΔE>7). The largest difference in change of EQUIA color was observed for seven-day tea treatment after the bleaching in comparison to the similar water treatment, showing that the color of material is becoming much darker (less brighter) and much more yellow.

Li et al.13 also recorded discoloration in glass ionomer conventional cements after the bleaching with 15% carbamide peroxide for four weeks. According to this, it is evident that the bleaching process leads to discoloration of restorative materials, but the changes were not clinically visible as opposed to the changes after subsequent staining in tea throughout one and two weeks.

The optical properties of restorative materials are essential during photopolymerization, because they can affect the light passing through the material and potentially lead to insufficient conversion of monomers in the polymer which can weaken the mechanical properties. Since no studies on light transmission of bleached restorative materials were found in the literature, our results cannot be compared to those of other authors.

This in vitro study has some limitations. Under clinical conditions, the tooth surface as well as the restoration is protected by the saliva and enamel pellicle which can dilute the bleaching agent and therefore minimize the effect of the bleaching agent. Further studies with different concentrations of bleaching agent and stimulation of oral conditions are necessary to characterize the precise change in physical and aesthetic properties of restorative materials.

CONCLUSION

Under the conditions of the present in vitro study, it can be concluded that bleaching agents can affect the color of glass ionomer cements and microhardness of both composite materials and glass ionomer cements.

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