Abstract
The purpose of this in vitro study was to compare the effect of in-office bleaching application on the color, microhardness, and surface roughness of five different tooth-colored restorative materials. Thirty specimens were fabricated from each restorative materials nano-hybrid composite (Grandio/Voco), micro-hybrid composite (Filtek Z250/3M), flow able composite (Filtek P60/3M), compomer (Dyract AP/DentsplyDeTrey) and glass-ionomer cement (Ketac Molar Easymix/3M] and divided into 3 subgroups. Specimens in group 1 were stored in distilled water at 37°C (control) during the hiatus period. Specimens in group 2 and 3 were treated with 15% hydrogen peroxide (HP) and 25% hydrogen peroxide, respectively. The data were analyzed with ANOVA and T-test. 15% HP and 25% HP groups showed an apparent color change (∆E*) than control group. In particular, Dyract AP and Ketac Molar Easymix showed a noticeable color change and statistically significant differences (p<0.05). HP groups showed a reduction in microhardness. Filtek Z250 and Filtek P60 does not have a statistically significant difference (p>0.05), Dyract AP and Ketac Molar Easy mix showed a statistically significant difference (p<0.05). Surface roughness was increased in 25% HP group after bleaching. In-office bleaching agents may affect the surface of existing restorations. Bleaching agents should not be used indiscriminately when these restorations are present.

Keywords: Microhardness, Surfaceroughness, Tooth Bleaching, Tooth Color

1. Introduction
The modern era has seen the rapid development of esthetic and restorative dentistry, driven by the importance modern people attach to facial appearance as an influential factor in making a good impression on others in their interpersonal relationships and in their social life and by the desire to enhance the esthetic value of tooth shape and color as a factor greatly influencing their facial appearance. There are a number of causes affecting the color of teeth, with the main causes being enamel discoloration or damage, degeneration of pulp tissue, heavy bleeding after pulp extirpation, trauma, infection, drugs, and fillers. One of the treatment methods to enhance esthetic appeal by controlling tooth discoloration is tooth bleaching, which occupies an important position in esthetic dentistry. Another method is restorative treatment using esthetic restorative materials.

Currently available peroxide-containing tooth-bleaching materials include professionally dispensed and supervised products for use by patients at home, professional-use in-office products, and over-the-counter products for sale directly to consumers. Contemporary in-office bleaching systems use an HP formulation of varying concentration (15~38%) directly on the tooth surface.

Over the past few years, in-office bleaching systems employing the use of strong oxidising agents have been re-introduced. Advantages are that it is totally under the dentist’s control, the soft-tissue is generally protected from the process and it has the potential for bleaching
quickly in situations in which it is effective. Hydrogen peroxide (HP) acts as a strong oxidising agent through the formation of free radicals, reactive oxygen molecules and HP anions. These reduce or cleave pigment molecule double bonds to either break down pigments to small enough molecules that diffuse out of the tooth or to those that absorb less light and hence appear lighter. The most effective agents release oxidising compounds, including HP, Carbamide Peroxide (CP), sodium percarbonate, or other compounds. HP is a primary component of tooth bleaching. HP may be applied directly, or produced in a chemical reaction from CP.

Given the fact over 40% of the population has at least one dental restorative materials, the effect of different bleaching treatments on dental materials have received much attention. Esthetic restorative materials, such as composite resin, Polyacid-Modified Composite (compomer), and Glass-Ionomer Cement (GIC), have become an integral and important part of modern dentistry.

The success of these methods depends on two aspects: physical properties and esthetic factors. Strength and wear resistance are two important physical properties, and esthetic factors conducive to satisfactory treatment results are color stability, radiopacity equivalent to that of enamel, durability of the restorative treatment in the oral cavity, and absence of side effects when applying other treatments. The bleaching agent may have a varying influence on the color behavior of composites and teeth, or even deteriorate restorative materials. Different bleaching efficiency on restorative materials or enamel may require the replacement of existing restorations for esthetic reasons.

Despite the popularity of in-office bleaching applications and esthetic restorative treatments, there are few studies on the health effects of esthetic restorative materials such as composites, GIC, and compomers in the intrarodentary space after the application of in-office tooth bleaching. Studies that evaluated the interaction between bleaching and the color, microhardness and surface roughness of esthetic restorative materials have reported contradictory results.

Therefore, the purpose of this study is to evaluate the effect of in-office bleaching gels, containing 15% hydrogen peroxide and 25% hydrogen peroxide, consistently applied over 14 days, on the color, microhardness, and surface roughness of five different esthetic restorative materials.

## 2. Methodology

### 2.1 Materials

The tooth-colored restorative materials selected for this study included a nano-hybrid composite (Grandio/ Voco), micro-hybrid composite (Filtek Z250/3M), compomer (Dyract AP/DentsplyDeTrey) and GIC (Ketac Molar EasyMix/3M). Hydrogen peroxide (15% and 25%, Discus Dental, Culver City, CA, USA) were the bleaching agent, which is the most common water at material used for in-office bleaching.

### 2.2 Methods

30 disc-shaped specimens from each material (150 specimens in total), 5 mm in diameter and 4 mm in height, were prepared in acrylic molds. And covered with mylar strips. A glass slide was then placed over extrude is and pressure applied to excess material. The composite resin, compomer, and Glass-ionomer cement specimens were then light polymerised (Elipar Highlight, 3M ESPE, Seefeld Germany) for 40 seconds to ensure adequate polymerisation. The glass ionomer cement was mixed as per the manufacturer’s instructions and packed into the molds. All restorative materials were shade A3 (of the Vita Shade guide). A total of 150 specimens (30 of each materials) were fabricated and stored in distilled water for 24 h at 37°C to ensure complete polymerisation. All specimens were polished and cleaned in distilled water in an ultrasonic cleaner 2 minutes to remove any surface debris. All specimens were then placed in 37°C distilled water for 7 days and randomly divided into three groups. Table 1 lists the materials tested. Specimens in Group 1 were stored in DW at 37°C for 2 weeks (control). Specimens in group 2 and 3 were treated with 15% HP and 25 % HP, respectively. Group 2 and 3 specimens were bleached in one session for three times (3 × 15 min session / week for two weeks). 15% HP and 25% HP groups were treated with bleaching gels according to manufactures’ instructions. Each day after the active treatment period, the specimens were rinsed for a standardised time of 1 minute with distilled water to remove the bleaching agents.

Color measurements were carried out before and after bleaching with a spectrophotometer (MINOLTA CM-3500d, Japan) in the L*a*b* mode described by the Commission Internationale de l’Eclairage (International Commission on Illumination, CIELab). Microhardness
was measured on a hardness testing machine (Micro-hardness tester, Japan) with a load of 50 g for 15 s intervals. Average surface roughness (Ra) was measured using a surface roughness tester (Profiler Brooks contour GT, USA).

2.3 Method of Data Analysis
The analysis of all the data was performed using SPSS 16.0 (SPSS, Chicago, IL, USA) that is the software for statistical analysis. All statistical analyses were carried out at significance level of $p<0.05$. ANOVA and Kruskal-Wallis analysis was used to determine any significant change in color, microhardness and surface roughness, and the difference between before and after bleaching was measured by paired $t$-test.

3. Results

3.1 Color Measurement
25% HP groups showed a color change ($\Delta E^*$) than control group (Table 2). In particular, Dyract AP and Ketac Molar Easymix showed a noticeable color change and statistically significant differences ($p<0.05$).

3.2 Microhardness Measurement
25% HP group showed a reduction in microhardness. Grandio, Filtek Z250 and Filtek P60 does not have a statistically significant difference ($p>0.05$), Dyract AP and Ketac Molar Easymix showed a statistically significant difference ($p<0.05$). For all test groups, Grandio, Filtek Z250 and Filtek P60 showed the highest values, whereas Dyract AP and Ketac Molar Easymix presented the lowest ($p<0.05$).

3.3 Surface Roughness Measurement
Ra of the tooth-colored restorative materials varied between 0.04 and 0.35 $\mu$m. Dyract AP and Ketac Molar Easymix showed significantly higher Ra values than all other tooth-colored restorative materials ($p<0.05$). 25% HP group increased the Ra values of materials more than did 15% HP group.

4. Discussion

The growing desire and demand for esthetic restoration and the emergence of new dental esthetic restorative materials have played a pivotal role in the development of...
innovative materials and techniques to replace the metallic restorative materials traditionally applied in clinical settings. Along with this trend, the growing interest in tooth bleaching to make natural teeth artificially clean and white has led to increasing application of bleaching procedures. In-office tooth bleaching, which is one such procedure, has the advantage of immediate effect without the need for repeated clinic visits. However, given that tooth bleaching involves contact of the oral tissue with bleaching agents and requires a certain length of treatment time, it is considered to have certain effects on intraoral restorative materials. Although previous studies investigated the characteristics of the tooth associated with tooth-bleaching agents, the current study focused on the effects of tooth-bleaching agents on dental esthetic restorative materials in terms of their color, hardness, and roughness. Hubbezoglu\textsuperscript{13} claimed that restorative materials should be replaced after bleaching based on their finding that 35\% HP bleaching induced substantial discoloration of composite resins such as Admira and Durafill VS. According to the findings of a study conducted by Monaghan\textsuperscript{14}, bleaching with 30\% HP resulted in composite color changes of $\Delta E^* \geq 3$. In a study by Yalcin\textsuperscript{15}, DY showed the greatest color changes when exposed to 10\% CP and 6.5\% HP, and there was a low degree of color change in composite resin, whereby 6.5\% HP yielded a higher degree of color change than 10\% CP. In this study, the exposure to composite resin did not lead to any obvious color changes, and the greatest color changes were shown in compomers and GIC.

In this study, considering the possible variations arising from different colors among materials, polymerisation methods, resin thicknesses, gauges, and dipping solutions, care was taken to keep the colors of the restorative materials and the measuring methods constant and to store all restorative materials in distilled water.

Ruyter reported that 3.3 is the threshold $\Delta E^*$ value for color change perceivable for the human eye\textsuperscript{16}. The measurements in this study yielded post-bleaching $\Delta E^*$ values of 4.5 and 4.9 in the DY group and KM group, respectively, i.e., values that are perceivable by the human eye. According to the analyses of esthetical restorative material surfaces performed by Plotino\textsuperscript{17} using SEM, surfaces of esthetical restorative materials undergo dissolution by bleaching agents, whereby microholes are generated on a dissolved surface and infiltrated by water and saliva, which is reflected on the surface and thus increases the $L^*$ value, and that the degree of surface dissolution by bleaching agents depends on the degree of the infiltration of bleaching agents into the restorative materials. Thus, such color changes vary according to the type of restorative material and length of contact time. In the case of composite resin composed of crosslinked polymers, its rapid bleaching process does not require much time, i.e., enough for the bleaching agent to induce its surface dissolution, thus resulting in less perceivable discoloration. Yap\textsuperscript{18} reported

\begin{table}[h]
\centering
\caption{Colormicrohardness, and surface roughness values of the restorative materials after tooth bleaching (mean±sd)}
\begin{tabular}{|c|c|c|c|c|}
\hline
Material & Group & $\Delta E^*$ & Microhardness & Surface roughness \\
\hline
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Grandio & Control & 1.5±1.4 & 95.2±0.2 & 0.05±0.8 \\
& 15\% HP & 1.5±0.8 & 94.1±0.2 & 0.09±1.3 \\
& 25\% HP & 1.6±0.6 & 93.7±0.2 & 0.11±0.6 \\
\hline
Fitek Z250 & Control & 1.3±0.9 & 89.0±0.1 & 0.04±0.9 \\
& 15\% HP & 1.3±1.2 & 88.0±1.1 & 0.05±0.2 \\
& 25\% HP & 1.4±0.4 & 87.0±0.7 & 0.06±0.5 \\
\hline
Fitek P60 & Control & 1.0±1.6 & 87.4±0.1 & 0.05±1.4 \\
& 15\% HP & 1.3±0.7 & 87.3±1.2 & 0.08±0.3 \\
& 25\% HP & 1.8±0.3 & 87.1±0.7 & 0.12±0.7 \\
\hline
Dyract AP & Control & 1.7±0.9 & 54.6±1.3 & 0.10±0.4 \\
& 15\% HP & 3.1±1.3† & 49.6±0.4 & 0.17±1.2 \\
& 25\% HP & 4.5±0.7† & 45.6±0.8† & 0.29±1.1† \\
\hline
Ketac Molar Easymix & Control & 2.0±0.4 & 58.8±0.9 & 0.11±1.0 \\
& 15\% HP & 3.7±0.8† & 54.2±0.3 & 0.18±0.5 \\
& 25\% HP & 4.9±0.9† & 43.8±0.7† & 0.35±0.3† \\
\hline
\end{tabular}
\end{table}
that compomers absorb water and tend to expand, leading to inter-filler ruptures and an increase in surface reflection, with a consequent increase in brightness. This mechanism is assumed to work on GIC, thus yielding results similar to those yielded by compomers. Satou19 reported that the water absorption rate plays an important role in the discoloration of composite resins. From this, it can be inferred that Z350 and P60 demonstrate higher brightness stability as compared to other restorative materials owing to their hydrophobic bis-EMA-induced low water absorption and solubility characteristics, thus resulting in less infiltration of the bleaching agent into the surface of the restorative materials.

The comparison of post-bleaching hardness among the esthetic restorative materials yielded the findings that although almost no change occurred to the composite resin, DY and KM showed statistically significant decreases in hardness (p < 0.05). In the studies carried out by Hannig20 and Rosentritt21, both composite resins and compomers showed a decrease in strength after bleaching with 10% CP and 38% HP, thereby affecting not only the surfaces of the restorative materials but also their interiors down to a depth of 2 mm. Much research has also been conducted regarding the effects of low-concentration tooth-bleaching agents on the microhardness of composite resins; composite resins are reported to be affected little in contrast to compomers, which showed a significant decrease in hardness. According to the results of Taher’s study, GI lost its hardness after 15-day bleaching with 15% CP. The decrease in hardness of the restorative materials was triggered by the polymer degradation induced by free radicals released as a result of decomposition, which attacked and decomposed the non-reactive double bond of the most vulnerable part, thus resulting in a reduction of surface microhardness due to reduced mass21.

The surface roughness of esthetic restorative materials is a clinically important physical property that has great impact on esthetics and oral health. A rough surface is more prone to the deposition of bacterial plaque and bacteria, triggering inflammation by gingival damage, and the patient is likely to feel discomfort after restorative treatment24. Yap25 reported that the average surface roughness corresponding to a threshold value for bacterial adhesion in the oral cavity is 0.2 µm. With a surface roughness <0.2 µm, bacterial accumulation stops, and at ≥0.2 µm, plaque accumulation increases in proportion to the surface roughness. It was further claimed that if the composite resin surface is rough, color stability decreases due to pigmentary deposit and subsequent discoloration and color change, which makes it necessary to replace the affected restorative materials. Several studies have demonstrated that bleaching with 10–16% CP led to an increase in surface roughness, surface porosity, and SEM-observed cracks26,27. In this study, all restorative materials tested showed a post-bleaching increase in surface roughness, whereby Z350 and P60 did not show statistically significant differences, with <0.2 µm roughness, whereas DY and KM showed significant pre- and post-bleaching differences, with roughness values of 0.29 and 0.35 µm, respectively. DY has a property of expansion by absorbing water, thereby inducing stress corrosion, leading to the total or partial detachment and split of the filler and subsequent increase in surface roughness28. HP is known to have high capacities of oxidation and reduction and may generate free radical species. Free radicals disrupt the arrangement of fillers and matrix, and the split fillers and matrix increase the surface roughness29,30.

It should be noted that this study has certain limitations. The in vitro test conditions used are imperfect representations of the oral cavity. Further research, comparing the effects of different concentrations of bleaching agents on the physical properties of dental restorative materials, should be performed in controlled clinical studied to corroborate the in vitro results in the present study. The problems likely to be encountered in relation to the oral cavity because of the increased surface roughness will have to be prevented by polishing DY and KM surfaces or replacing the restorative materials.

4. Conclusion

The effect of in-office bleaching agents on color, microhardness, and surface roughness were material dependent. No significant color, microhardness, and surface roughness changes were found after application of 15% and 25% HP for composite resin. Tooth bleaching agents (15% and 25% HP) affected the on color, microhardness, and surface roughness of DY and KM. 25% HP surface roughness of materials (DY and KM) more than did 15% HP. It is necessary to consider the type of the material before starting the treatment.

5. Acknowledgement

This work was supported by the SunMoon University Research Grant of 2014.
7. References


