Effects of Two Different Bleaching Agents on Surface Roughness and Microhardness of Different Novel Nano-Restorative Materials

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Abstract

Objectives: The aim of this study was to evaluate the effects of 15% hydrogen peroxide (Illuminé Office) and 16% carbamide peroxide (VivaStyle) on the surface roughness and hardness of nano-restoratives. Materials and Methods: A total of 30 specimens of each material were fabricated using Clearfil Majesty Esthetic (Group1), Tetric EvoFlow (Group2) and Ketac N100 (Group3). Each group were divided into 3 subgroups and treated as follows: Group 1a, 2a, 3a was treated with Illuminé Office, Group 1b, 2b, 3b was treated with VivaStyle, Group 1c, 2c, 3c was stored in distilled water at 37°C for two weeks (control). Surface roughness and microhardness tests were performed. Results: There were no significant differences in terms of roughness and microhardness among Clearfil Majesty Esthetic and Tetric EvoFlow groups, separately ($P > 0.05$). Illuminé Office increased the roughness and decreased the microhardness of Ketac N100 ($P < 0.05$). Conclusion: Bleaching may affect the roughness and microhardness of nano-restoratives depending on material and bleaching system.

Keywords: Bleaching, microhardness, nano restorative, surface roughness

INTRODUCTION

External bleaching is a noninvasive, effective, and safe treatment commonly used for dental esthetic clinical procedures. The application of hydrogen peroxide or carbamide peroxide is a popular method of vital teeth bleaching.[1] Procedures may be performed at the dental office (in-office bleaching) or by applying the agent in a gel form within the confines of a custom tray by the patient (home bleaching).2-4 Bleaching is one of the most conservative treatments for discolored teeth compared with other treatments, such as veneers, crowns, or composite bonding.5,6

The mechanism of bleaching is still not clear, but it consists of an oxidation reaction of 10% carbamide peroxide that in contact with saliva and the oral fluids is dissociated into 7% urea and 3% hydrogen peroxide.7,8 The hydrogen peroxide diffuses and releases free radicals that can break the pigmented carbon rings of high molecular weight into smaller molecules which are lighter in color.7-9 The prognosis and longevity of a restoration not only depends on mechanical properties, but also on the physical and biological properties of the materials.10,11 Surface roughness has been a major concern for researchers and clinicians as it is associated with plaque retention, which may lead to gingival inflammation and caries formation.10-12

Hardness may be explained as the resistance of solid structures to permanent indentation or penetration.13 Alterations in hardness may reflect the state of the setting reaction of a material and the presence of an ongoing reaction or maturity of the restorative material.14 Besides, hardness shows depth of cure and effective polymerization degree.15 Materials that have lower surface hardness are more susceptible to scratching than composites with higher hardness values,15,16 which can compromise fatigue strength and cause premature failure of restoration.13

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86
The effects of bleaching agents on oral tissues have been evaluated by several studies, but there have been a limited number of studies evaluating their effects on dental materials. Studies on oral tissues demonstrated that bleaching has no clinically significant results on enamel. Nevertheless, some clinicians still express concerns about the effects of bleaching systems on restorative materials. As various restorative materials have come to be used for on increasingly wide range of applications, esthetic factors are now receiving a greater attention.

Therefore, the purpose of this in vitro study was to evaluate the effects of 15% hydrogen peroxide (Illuminé Office/Dentsply) and 16% carbamide peroxide (VivaStyle/Ivoclar-Vivadent) on the surface roughness and microhardness of three different tooth-colored nano-restoratives; a nano-composite (Clearfil Majesty Esthetic/Kuraray), a flowable nano-composite (Tetric EvoFlow/Ivoclar-Vivadent), and a light-curing nano-ionomer restorative (Ketac N100/3M ESPE). The null hypothesis was application of two types of bleaching materials which would not affect the microhardness and surface roughness values of the three different restorative materials in this study.

**Materials and Methods**

A nano-composite (Clearfil Majesty Esthetic, Kuraray, Düsseldorf, Germany), a flowable nano-composite (Tetric EvoFlow, Ivoclar-Vivadent, Schaan, Liechtenstein), and a light-curing nano-ionomer restorative (Ketac N100, 3M ESPE, St. Paul MN, USA) were utilized in this study. An office bleaching agent (Illuminé Office, Dentsply/Caulk, Milford, DE, USA) and a home bleaching agent (VivaStyle, Ivoclar-Vivadent, Schaan, Liechtenstein) were used. The properties of the materials tested are presented in Table 1. Ninety disc-shaped specimens were prepared using a Plexiglass mold (10 mm in diameter and 2 mm in thickness). Specimens were divided into three subgroups, 30 discs per restorative material. After placing the restorative materials to the mold, they were covered with a Mylar strip. A glass slide, 1–2 mm thick, was placed over the strip before polymerization with a light-emitting diode (LED) light-curing unit (Elipar FreeLight 2, 3M ESPE, St. Paul MN, USA) to flatten the surfaces and inhibit oxygen inhibition layer. The specimens were then cured for 20 s through the Mylar strip and glass slide. The polymerization was performed on both sides of the specimen for 20 s after removing the strips and glasses. The curing light was placed perpendicular to the specimen’s surface at or less than a distance of 1.0 mm. Curing light intensity was measured at 620 mW/cm² and was monitored with a light meter. To reduce variability, all specimen preparations and treatment procedures were performed by the same operator. Specimens were examined for obvious voids, labeled on the bottom.

Samples were ground wet with a 1200 grit silicon carbide paper on a metallurgical finishing wheel to provide a baseline before polishing systems. Sof-Lex Pop-On Discs at medium, fine, and super-fine grits were used for 30 s (15,000 rpm with a slow speed handpiece), each on the composite samples. After each step of polishing, all specimens were thoroughly rinsed with water and air-dried before the next step until final polishing. Each disc was discarded after use.

Thirty discs in each restorative group were randomly subdivided into three groups (n = 10) for different bleaching systems and were treated as follows:

- **Group 1a:** Clearfil Majesty Esthetic treated with Illuminé Office for 2 h

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**Table 1: Manufacturers and composition of the materials utilized in the study**

<table>
<thead>
<tr>
<th>Products</th>
<th>Type</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Illuminé Office</td>
<td>Office bleaching agent</td>
<td>30% hydrogen peroxide, poly (methyl vinyl ether/maleic anhydride) mixed calcium/sodium salts, titanium dioxide</td>
<td>Dentsply/Caulk, Milford, DE, USA</td>
</tr>
<tr>
<td>VivaStyle</td>
<td>Home bleaching agent</td>
<td>16% carbamide peroxide</td>
<td>Ivoclar-Vivadent, Schaan, Liechtenstein</td>
</tr>
<tr>
<td>Clearfil Majesty Esthetic</td>
<td>Nanofilled composite</td>
<td>Matrix: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, di-Camphorquinone, initiators, accelerators, pigments, Filler: Silanated barium glass filler, prepolymerized organic filler</td>
<td>Kuraray Europe Gmbh, Düsseldorf, Germany</td>
</tr>
<tr>
<td>Ketac N100</td>
<td>Light-curing nano-ionomer restorative</td>
<td>De-ionized water, HEMA, vitrebond copolymer (a methacrylate-modified polyalkenoic acid), fluoroaluminosilicate glass, nanomers, and nanoclusters</td>
<td>3M ESPE, St. Paul MN, USA</td>
</tr>
</tbody>
</table>

Bis-GMA – Bis-phenol A diglycidylmethacrylate, UDMA – Urethane dimethacrylate, HEMA – 2-hydroxyethyl methacrylate
Bicer, et al.: Effects of different bleaching agents on surface roughness and microhardness

Results

The average surface roughness values and standard deviation of three restorative materials are presented in Table 2. Surface roughness values before bleaching procedures showed no significant differences for the three evaluated restorative materials (control groups) \((P > 0.05)\). There were no significant differences in terms of surface roughness after bleaching among Clearfil Majesty Esthetic groups and Tetric EvoFlow groups, separately \((P > 0.05)\). However, Illuminé Office significantly increased the roughness of Ketac N100 \([P < 0.05, \text{Figure 1}].\)

For Illumine Office (15% hydrogen peroxide) bleaching agent, the ranking for surface roughness values was as follows: Clearfil Majesty Esthetic = Tetric EvoFlow < Ketac N100 \((P < 0.05)\).

For Vivastyle (16% carbamide peroxide) bleaching agent, surface roughness values from least were as follows: Tetric EvoFlow = Ketac N100 = Clearfil Majesty Esthetic \((P > 0.05)\).

For control groups (stored in distilled water at 37°C for 2 weeks), the ranking for surface roughness was as follows: Tetric EvoFlow = Clearfil Majesty Esthetic = Ketac N100 \((P > 0.05)\).

The average microhardness values and standard deviations of three restorative materials are shown in Table 3. The statistical analysis showed that both bleaching regimens did not have a significant effect in terms of microhardness in Clearfil Majesty Esthetic groups and Tetric EvoFlow groups, separately \((P > 0.05)\). However, Illuminé Office significantly increased the microhardness of Ketac N100 \((P < 0.05)\).

### Results

#### Surface roughness test

Surface roughness tests were performed with a contact-type profilometer (Perthometer M1 Mahr, Göttingen, Germany). Three successive measurements in different directions were recorded for all specimens in each group, and average surface roughness (Ra) values were noted. Sampling length for each surface roughness measurement was 1.25 mm with a cutoff value of 0.25 mm.

### Microhardness test

Vickers hardness number was determined using microhardness test machine (Buehler Lake Bluff, Illinois, USA). Indentations were made as diagonals of the square-based pyramid impressions with a 500 g load applied for 15 s. Three indentations were recorded at different points on each specimen, and the microhardness value was obtained as the average of these findings.

### Statistical analysis

Statistical analysis was calculated using SPSS software version 21.0 (IBM Corp, Armonk, NY, USA). Data were tabulated and evaluated for normality by Kolmogorov–Smirnov test. Comparison between baseline and posttreatment measurements was performed using one-way ANOVA. For multiple comparisons, Tukey’s honest significant difference test was used \((P < 0.05)\). A significance level of 5% was assumed in all tests.

### Table 2: Mean values and standard deviations of groups for surface roughness

<table>
<thead>
<tr>
<th>Groups</th>
<th>Mean±SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil Majesty Esthetic (1)</td>
<td>0.092±0.004</td>
</tr>
<tr>
<td>Tetric EvoFlow (2)</td>
<td>0.073±0.006</td>
</tr>
<tr>
<td>Ketac N100 (3)</td>
<td>0.096±0.020</td>
</tr>
</tbody>
</table>

Values with the same superscripts are not significantly different. SD – Standard deviation
decreased the microhardness of Ketac N100 compared to control group (Group 3c) \[ P < 0.05, \text{Figure 2}. \]

For Illumine Office (15% hydrogen peroxide) bleaching agent, the ranking for microhardness values from least to most was as follows: Tetric EvoFlow < Ketac N100 = Clearfil Majesty Esthetic \[ P < 0.05. \]

For Vivastyle (16% carbamide peroxide) bleaching agent, the ranking for microhardness values from least to most was as follows: Tetric EvoFlow < Ketac N100 = Clearfil Majesty Esthetic \[ P < 0.05. \]

For control groups (stored in distilled water at 37°C for 2 weeks), the ranking for microhardness values from least to most was as follows: Tetric EvoFlow < Ketac N100 = Clearfil Majesty Esthetic \[ P < 0.05. \]

In dentistry, surface roughness measurements are usually carried out with the help of a profilometer.\[11,13\] In the current study, profilometer is used to determine the surface roughness. Arithmetical average roughness values are the most commonly used parameter in the assessment of surface roughness.\[32,33\]

Surface roughness of a restoration is important as it plays a major role in the formation of biofilms and bacterial adhesion that may lead to gingival inflammation and caries.\[34\] Several studies have investigated the effects of bleaching agents on the surface characteristics of restorative materials.\[25,35-38\]

Even though the usual duration of home bleaching is between 2 and 4 weeks, darker, discolored teeth, especially tetracycline-stained teeth, may require longer bleaching time.\[39,40\]

Bleaching has gained popularity over the past decade due to its efficiency, ease of use, and minimal side effects. Thus, the effect of bleaching agents on the properties of restorative materials is important.\[12\] Investigations on the surface roughness of restoratives after bleaching treatment have shown contradictory results. Some studies showed that the surface finish of composite restorations was not affected by home bleaching agents,\[41,42\] while another one reported surface changes after bleaching treatment.\[43\] The opposing results may be attributed to the diverse bleaching protocols and materials tested. According to our results, only Illuminé Office application significantly increased the surface roughness of Ketac N100.

A study bleached the specimens only once\[42\] while others have bleached specimens for 2–4 weeks.\[41,43\] In the present study, bleaching treatment was carried out for 2 weeks based on a clinically relevant protocol.

Microhardness changes are related to a loss or gain of mineral (demineralization or remineralization) of the dental structure. The first defense mechanism capable of neutralizing acids of the bleaching agents is the saliva-buffering capacity. Bicarbonates in the saliva provide dilution and neutralization of the acids.\[9\]

It has been mentioned that the degree of polymerization of composite resins affects the hardness of the resin matrix. Hardness values increase when conversion rate of carbon double bonds rises.\[13\] In this study, to obtain adequate polymerization, all samples were polymerized according to manufacturers’ instructions using a LED-curing light unit.

A few studies have been performed on the effects of bleaching agent on the microhardness of various aesthetic restorative dental materials. According to the literature, the microhardness of resin-based composites exposed to bleaching products has been reported to increase,\[30,44,47\] decrease,\[43\] and remain unchanged.\[42,48\] In the present study, both the bleaching regimens did not have a significant effect on the microhardness of the nano-composite restorative materials tested, while Illuminé Office significantly decreased the microhardness of the nano-ionomer Ketac N100.

The peroxide concentrations used in the current study may facilitate cumulative softening effects of the in-office whitening

### Table 3: Mean values and standard deviations of groups for microhardness (Vickers hardness number)

<table>
<thead>
<tr>
<th>Groups</th>
<th>Mean±SD</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Clearfil Majesty Esthetic (1)</td>
</tr>
<tr>
<td>Illuminé Office (a)</td>
<td>48.00±1.61\textsuperscript{v}</td>
</tr>
<tr>
<td>VivaStyle (b)</td>
<td>48.57±0.76\textsuperscript{v}</td>
</tr>
<tr>
<td>Control (c)</td>
<td>48.75±1.53\textsuperscript{v}</td>
</tr>
</tbody>
</table>

Values with the same superscripts are not significantly different. SD – Standard deviation

**Figure 2**: Mean microhardness values for the nano-restorative materials tested.
agents. Some studies have shown the importance of peroxide concentration and pH of bleaching agents in having the adverse effects on restorations.\cite{3},\cite{4} The softening effect of the carbamide peroxide restricted to the surface layer of composite restorations was supported by a study. The authors suggested re-polishing of the softened composite surface after bleaching with 16% carbamide peroxide.\cite{5} Mor et al. recommended to polish composite restoration after bleaching to eliminate the roughened outer surface, preventing the adherence of the microorganisms.\cite{6} However, a study by Hannig et al. demonstrated a decreased microhardness of deep layers in the resin restorations treated by different bleaching techniques. They concluded that polishing may not be able to re-establish the surface hardness of the filling after the bleaching.\cite{7} On the other hand, water or saliva may dilute or buffer the bleaching agents and influence the clinical results. A salivary layer might modify or decrease the effects of bleaching agents on the restoration in the intraoral situation.\cite{9} However, the absorption of salivary proteins by the composite surface decreases in time so that the impact of bleaching procedures can be affected by complex circumstances.\cite{10}

Fukazawa et al. demonstrated the increase of the surface roughness of the light-curing nano-ionomer restorative after using Illuminé Office bleaching agent and proposed that H+ ions diffused into the cement replaced with metal cations that cross-linked the polycarboxylic and acidic molecules in the cement matrix.\cite{11}

In the present study, the influence of 15% hydrogen peroxide (office bleaching agent) and 16% carbamide peroxide (home bleaching agent) on the surface roughness and microhardness of three different tooth-colored nano-restoratives was investigated. As the bleaching treatment has been becoming increasingly a popular treatment in esthetic dentistry, further studies are needed to determine the influence of home and office bleaching agents to the different restorative materials.

**Conclusions**

Within the limitations of this *in vitro* study, it may be concluded that:

1. Bleaching treatment may affect the roughness and microhardness of nano-restorative materials depending on the resin material and bleaching system
2. There was a negative correlation between surface roughness and microhardness as office bleaching agent decreased microhardness and increased surface roughness of the nano-ionomer restorative material.

**Clinical relevance**

Nano-ionomer restorative materials’ microhardness values decrease and surface roughness values increase with the use of office bleaching agents.

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Nil.

**Conflicts of interest**

There are no conflicts of interest.
Bicer, et al.: Effects of different bleaching agents on surface roughness and microhardness


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