The effect of in office bleaching on surface roughness and micro-hardness of newly developed composite materials (In vitro study)

Noor S. Nadhum, B.D.S., H.D.D. (1)

ABSTRACT
Background: Alterations in the microhardness and roughness are commonly used to analyze the possible negative effects of bleaching products on restorative materials. This in vitro study evaluated the effect of in-office bleaching (SDI pola office +) on the surface roughness and micro-hardness of four newly developed composite materials (Z350XT-nano-filled, Z250XT-nano-hybrid, Z250-micro-hybrid and Silorane-silorane based).

Materials and methods: Eighty circular samples with A3 shading were prepared by using Teflon mold 2mm thickness and 10mm in diameter. 20 samples for each material, 10 samples for base line measurement (surface roughness by using portable profilometer, and micro-hardness by using Digital Micro Vickers Hardness Tester), and 10 samples for after bleaching measurement. The appropriate bleaching procedure was performed on the top surface of test groups for 90 minutes total bleaching period. Then surface roughness and hardness were tested at the end of the duration. Statistical analysis was carried out using ANOVA, LSD and t-test.

Results: There was a highly significant increase in surface roughness of all tested groups after bleaching. There is a highly significant increase in micro-hardness for Z250, there is decrease in Micro-hardness for silorane and Z250xt and there is a non-significant increase in micro-hardness of Z350xt.

Conclusion: bleaching has a negative effect on surface roughness of all the tested materials, as surface roughness increased after bleaching. Micro-hardness is a material dependent, there is different reaction to bleaching depending on the resin, load and size of the fillers used in the materials. Nano-filled composite is the material that has better performance than the other tested materials, as it is the material that has the least affection by bleaching.

Key words: surface roughness, micro-hardness, in-office bleaching, nano-filled, nano-hybrid, silorane.

INTRODUCTION
Dental bleaching is one of the most commonly used dental esthetic clinical procedures. This treatment offers higher self-esteem to patients with minor consequences to teeth and gingival tissues when it is well indicated and performed (1). The aesthetic appearance of anterior teeth has become a major concern for patients. Discolored vital anterior teeth have long been treated with different approaches, including crowns, direct and indirect veneers, composite resin restorations, and, most conservatively, bleaching. Both take-home and in-office bleaching techniques have proven effective in whitening teeth, with the latter having the advantage of producing immediate results (2). The typical in-office bleaching regimen involves application of a high-percentage hydrogen peroxide formulation to the teeth surfaces, which is activated either chemically or by a light source. The theoretical advantage of using lights is their ability to heat hydrogen peroxide, thereby enhancing the rate of oxygen decomposition. The increased amount of oxygen-free radicals produced thus enhances the release of stain-containing molecules and, therefore, results in enhanced whitening (2).

Tooth-colored restorative materials, especially composite resin, have become an important part of modern dentistry. Use of this material has recently increased because of consumer demands for esthetic restorations (3). Newly developed composites with different matrix types, such as siloranes and filler types such as nano composites are used in clinical practice more often than hybrids (3).

The consequences of bleaching of resin-based materials can vary according to resin and bleaching gel compositions, frequency and duration of exposure (1). Alterations in microhardness and roughness are commonly used to analyze the possible negative effects of bleaching products (4). An increase in superficial roughness is clinically relevant, and irrespective of etiological factor, increase in roughness results in accumulation of food residues and formation of biofilms, leading to periodontal tissue disease (1).

MATERIALS AND METHODS
Four different composite resins that differ in their filler and resin content were selected: Filtek p90,3M ESPE, a silorane based composite, FiltekZ250,3M ESPE, microhybrid composite, Z350XT, 3M ESPE, nanofilled (nanoclusters) composite and Z250XT, 3M ESPE, nanohybrid composite.
Eighty circular samples were prepared, 20 samples for each of the four materials, by using Teflon molds with a circular hole, 2 mm in thickness and 10 mm in diameter, were fabricated \(^{5,6}\). The color corresponding to shade A3 was used for each material \(^{3}\). The Teflon mold was positioned on a glass slide. After inserting the materials into the Teflon mold, a transparent plastic matrix strip was put over them and a glass slide was secured as seen in (figure1) in order to flatten the surface and to prevent the formation of oxygen-inhibited layer on the surface of the samples \(^{7}\). A (200 gm) pressure has been applied for 1min. to expel excess material from the mold and to reduce voids \(^{7}\). The resin composites were cured by using a light-curing unit (QD,UK) at an intensity of 450 mW/cm\(^2\) which was verified before polymerization by using a radiometer \(^{8}\). Every sample was light cured for 80s in 2 steps (40 for each side) \(^{5}\).

**Figure1: Securing the mold with glass slide**

The samples were polished with medium, fine, and superfine disks (Soflex, 3M ESPE, St. Paul, MN, USA) on a slow hand piece, in accordance with the manufacturer’s instructions \(^{3, 6}\). After polishing, the samples were cleaned with distilled water and then the samples were put in ultrasonic cleaner for 2 minutes to remove any surface debris \(^{3}\). A mark was made on the side that will be untreated (unbleached) of each sample to identify the surface type \(^{1}\). All samples were stored in distilled water at room temperature for 24h before the initiation of any procedure. All samples were then divided into 8 test groups (n=10). Ten samples of each of the 4 different resin composite samples were selected for baseline surface roughness measurements (with the portable surface profilometer) and surface micro hardness tests (using digital Vickers Instrument) as control groups \(^{3}\). And 10 samples of each of the 4 materials were subjected to superficial treatment (bleaching using SDI pola office + 37.5%hydrogen peroxide) and then doing the surface roughness and micro hardness tests.

**Bleaching procedure**

The appropriate bleaching procedures were performed on the top of the unmarked surfaces of the samples of the test groups \(^{3}\). The bleaching agent was applied over the surface of each specimen or sample, the entire surface must be covered with adequate amount of bleach and that is 0.2cc for every sample (every 1cc is enough for 5 samples).

**Figure 2: Putting the bleaching on the sample.**

The groups were treated with bleaching agent (37.5% hydrogen peroxide SDI pola office +), and with the use of beyond halogen emitting light (beyond, USA). For 45minutes each time (every 15 minute the old material was removed with the use of distilled water and we put a new bleaching coat) \(^{6}\), at intervals of one week 1st and 7th day for totally 90 minutes for all the treatment period \(^{3}\). At the end of every bleaching procedure, the treated specimens were washed, under flowing distilled water and then the samples put in ultrasonic cleaner for 2 minutes \(^{3}\) to remove any remnant of the bleaching material. Then they were placed in fresh distilled water until the next application. The distilled water was replaced every day \(^{3}\).

**Figure 3: Enhancing the bleaching with beyond device.**

**Surface roughness measurements**

For surface roughness measurements, the specimens were examined by Portable Roughness Tester device (TR220 Portable RoughnessTester (Beijing TIME High Technology Ltd.). For each sample of all the groups, three randomized readings were performed on the challenged
The effect of in surfaces after and before bleaching protocol. Margins and visible irregularities were avoided (1) After the three readings, the mean surface roughness values were obtained for each sample (9).

**Microhardness measurements**

For micro hardness measurement, the control and the bleached groups (after finishing of all and complete bleaching procedure), tested by the use the digital VHT device (Digital Micro Vickers Hardness Tester TH714 (Beijing TIME High Technology Ltd.). The specimens were blotted dry using clean gauze and positioned beneath the indenter of a microhardness tester (10). Surface hardness of the specimens was measured with microhardness tester using a 100 g load and 15 s dwell time at room temperature (3). The diagonal length impressions were measured and the hardness number (H) was calculated immediately.

Statistical analysis of data by using ANOVA test for materials before bleaching showed that there is a highly significant differences between the surface roughness (Ra) of the four composite materials (p=0.000).

**RESULTS**

**Surface roughness**

The pre-bleaching surface roughness of the four composite materials results showed that group1 (A) has the highest surface roughness (Ra), followed by group4 (A) and then group3 (A) and then group 2 (A) which has the lowest (Ra) mean value so the lowest surface roughness before bleaching.

Statistical analysis of data by using ANOVA test for post bleaching surface roughness (Ra) values for all types of tested composites in this study revealed that there is no significant differences(P >0.05) in surface roughness.

**Table1: Means, standard deviation, standard error of surface roughness (Ra) values in µm, and comparison of all tested materials before and after bleaching.**

<table>
<thead>
<tr>
<th>Mats</th>
<th>Descriptive Statistics</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>S.D.</td>
</tr>
<tr>
<td>G1</td>
<td>(A)</td>
<td>1.09</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>1.58</td>
</tr>
<tr>
<td>G2</td>
<td>(A)</td>
<td>0.61</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>1.74</td>
</tr>
<tr>
<td>G3</td>
<td>(A)</td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>1.77</td>
</tr>
<tr>
<td>G4</td>
<td>(A)</td>
<td>1.02</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>1.75</td>
</tr>
</tbody>
</table>
There is a highly significant increase in the surface roughness (Ra) of all the tested materials after bleaching, that mean all the tested materials have an increase in their surface roughness after bleaching as seen (in table 1).

**Microhardness**

The pre bleaching Vickers hardness number (VHN) results showed that group 3(A) revealed the highest (VHN) mean value, followed by group2(A), then group 1(A), and finally group4(A) with the lowest (VHN) mean value, the least micro hardness. Statistical analysis of data by using ANOVA test before bleaching showed that there is a highly significant differences (p<0.001) in micro hardness of the tested four composite materials. The data revealed from ANOVA test were analyzed by LSD test for all types of tested composites before bleaching. LSD test revealed that there was a highly significant difference (p<0.001) in VHN between all types of tested composite used in this study.

From that table, we can see that there is no significant difference in micro hardness value between group1(A) and group1(B), that mean bleaching have no or very little effect on microhardness of this material. For group2, there is a highly significant increase in microhardness after bleaching. For group3 and group4, there is a highly significant decrease in micro hardness after bleaching.

**Table 2: Means, standard deviation, standard error of microhardness (VHN) values, and comparison of all tested materials before and after bleaching**

<table>
<thead>
<tr>
<th>Mat.</th>
<th>Descriptive Statistics</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>S.D.</td>
</tr>
<tr>
<td>G1</td>
<td>(A)</td>
<td>68.71</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>69.19</td>
</tr>
<tr>
<td>G2</td>
<td>(A)</td>
<td>91</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>96.28</td>
</tr>
<tr>
<td>G3</td>
<td>(A)</td>
<td>96.71</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>90.25</td>
</tr>
<tr>
<td>G4</td>
<td>(A)</td>
<td>45.81</td>
</tr>
<tr>
<td></td>
<td>(B)</td>
<td>43.19</td>
</tr>
</tbody>
</table>

**Figure 5: Bar chart shows the differences between the means microhardness (VHN) of the four composite materials before and after bleaching.**
DISCUSSION

Surface roughness

All the four tested materials showed an increase in surface roughness with a non-significant differences between them and that may be due too the oxidation process that occur in the organic matrix which can facilitate water absorption and lead to loss of particles so roughness is more affected by bleaching than hardness this finding agree with (1) who said that roughness seems to be more affected than microhardness.

The results showed that post bleaching surface roughness (Ra) for Z250 has the highest increase in surface roughness value followed by Z250XT then followed by Silorane and then finally followed by Z350XT which has the lowest increase in mean surface roughness value (Ra).

These results may be due to the difference in the chemical composition of the tested materials and this hypothesis agree with (9) who suggest that the increase in roughness could be as a result of loss of resinous matrix rather than load particle.

According to this hypothesis Z250 and Z250XT affected more than the other tested materials as both of them contain BIS-GMA, UDMA, BIS-EMA. The resin technology of Z250XT is based on the Filtek Z250 restorative resin, replacing some of the TEGDMA with PEGDMA (3M ESPE), so both of them has the same resin composition, studies have reported that (UDMA) and (BIS-EMA), which are contained in Filtek Z250 and Z250XT, form fewer double bond, which result in a slightly softer matrix (11), and this softer matrix will cause debonding of the filler from the resin resulting in high rough surface.

Z350XT its resin is also affected but the presence of clusters protect the resin from degradation so the bleaching agent has lower effect than the other three materials. The addition of engineered nanoparticles to formulations containing nanoclusters reduces the interstitial spacing of the filler particles leading to higher filler loadings. The filled matrix (resin plus engineered nanoparticles) is harder and more wear resistant than resin alone. It has been noted that the largest particles present in the composites (clusters) provides a protective shoulder to the remaining resin matrix this finding agree with authors (12).

The Silorane has an intermediate effect as it is affected more than Z350 and less than Z250 and Z250XT, and that’s due to its different monomer (hydrophobic) that is affected less than the methacrylate resin monomer. The inorganic content of resin composites however, offers resistance to bleaching. Form, amount and distribution of fillers are all aspects that determine the clinical performance of these restorative materials agree with (13).

Microhardness

For Z250 there is a significant increase in micro hardness. In addition to the same reason said previously in surface roughness, that may be attributed to that the resin matrix undergo softening and removal by bleaching leaving heavily loaded filler surface with less matrix. These findings agree with (14) who found increase in hardness of micro hybrid resin after bleaching so he claimed that the active ingredients of bleaching can remove the surface resin layer and leave a rich of filler particles so a harder surface.

For Silorane there a highly significant decrease in surface Microhardness (VHN) this result may be due to many reasons one of them is may be attributed to the effect of hydrogen peroxide on the Silorane resin, and that peroxide may affect the resin filler interface and cause filler matrix debonding. This may cause microscopic cracks, resulting in increased surface roughness and decrease hardness of Silorane.

The other reason is the filler to matrix ratio which plays an important role in the effect of bleaching agent on the composite resin. The filler weight and volume ratio determines this effect as Silorane has the lower filler loading between the tested materials so its matrix is easily subjected to bleaching deterioration. The organic matrix of Filtek P90 is composed mainly by Silorane resin and the inorganic particles are quartz and yttrium fluoride, 0.1–2 μm Average 0.47 μm. Silane-treated silica filler, ytterbium fluoride, 76% wt. 55%Vol., so this material has less filler loading than the other tested materials and this finding agree with many authors (3,16).

For Z250 XT there is highly significant decrease in surface microhardness and this may be attributed to resin monomer, as the resin of Z250XT is composed of Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA. Bis-GMA and TEGDMA are both hydrophilic monomers so the reduction in VHN values may be attributed to the swelling and hydrolytic degradation of the matrix leading to filler /matrix cracking, also the incorporation of TEGDMA in the resin result in an increase water uptake in Bis-GMA this finding agree with authors (17). Hydrophilic groups such as the ethoxy group in TEGDMA are thought to show affinity with water molecule by hydrogen bonding to oxygen. These results agree with the findings of authors who found that the
Microhardness decrease related to the structure of the resin matrix (18).

For Z350 XT there was no significant alteration in its Microhardness and this finding agree with (3) who said that Nanobased composites were affected less than the hybrids and Silorane and also agree with authors (19) who said that Nanocomposite samples showed no significant alteration (color and microhardness) after bleaching. Thus, no replacement of restorations is required after bleaching. The nanofilled composite was developed for use in all areas of the mouth with high initial polish and superior polish retention (typical of microfills), as well as excellent mechanical properties suitable for high stress-bearing restorations (typical of hybrid composites). Changes in the structure or composition of this restorative material may have provided more resistant surface against bleaching treatments. The composite resin Filtek Z350XT (3M ESPE) has a nanofilled composite has a very small particle size. This may be another reason why nanofilled with smaller filler size has the highest polishing and consequently, smaller effect from bleaching agents, and disagree with Wang et al (1) who said the bleaching gels affected nanofilled and microhybrid composite resins.

It has been noted that the largest particles present in the composites(clusters) provides a protective shoulder to the remaining resin matrix (10) due to the shorter inter-particle spacing (19). According to heavily loaded Jorensen (20) reported that when the distance between neighbor filler particles is around 0.1 µm, it protects against matrix wear.

REFERENCES


