Comparative Evaluation of Micro-hardness and Surface Roughness of Different Composites Resins and Polishing System (In-Vitro Study)

Sahar Abd El Halim*

ABSTRACT:

Objectives: The purpose of this study was to evaluate the microhardness and surface roughness of Nano hybrid and Nano-fill composites with and without polishing technique (in-vitro study).

Materials and Methods: sixty-four samples were prepared in disc-shaped stainless-steel molds with a uniform size of 6 mm in diameter and 4 mm in thickness. The samples were divided according to the materials used into two groups of 32 samples for each material, and each group was then subdivided into subgroups according to the polishing instruments with 16 samples in each subgroup: Group I (control group) (Mylar’s strip) with no finishing and polishing. Group II, the specimens’ surfaces were finished with an ultrafine diamond finishing bur and polished with Sof-lex discs. Each subgroup was divided into two groups according to measurements of surface roughness and microhardness (n=8). The surface roughness was measured by using Mitutoyo Japan Surftest SJ-210 Tester and surface microhardness was measured using Vickers microhardness tester (Wilson TukonTM1102, Germany). The data were analyzed using two-way ANOVA for both the surface roughness and microhardness tests, followed by pairwise test for multiple comparisons

Results: the Mylar’s strips (control group) exhibited significantly lower roughness values (smoothest surface) than the polishing systems (p <0.0001). Nano-fill composite showed statistically significantly lower surface roughness. Nanohybrid composite showed statistically significantly higher microhardness value. Conclusions: The control group had the lowest surface roughness and microhardness values compared to the polishing group. Nano-fill composite showed lower surface roughness compared to nanohybrid composite. Nanohybrid composite had a higher microhardness value when compared with nano-fill composite.

Keywords: Resin Composite; Surface Roughness, microhardness

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Introduction:

Currently, resin composites continue to be the most widely used restorative materials due to their excellent esthetics, functional capacity, and mechanical properties \[^{1,2,3}\]. Application of nanotechnology in composites with nano-particles and nano-clusters have been introduced \[^{4,5}\]. The clinical success of composite restorations is related to surface smoothness, thus, finishing and polishing is of paramount importance for the success and longevity of these restorations \[^{6,7}\]. Surface roughness, as a consequence of irregularities in the application of restorative materials, it is a clinical problem, making it necessary to perform some finishing and polishing techniques to avoid later stains, plaque presence, recurrent deterioration, etc. \[^{8,9}\] surface roughness allows accumulation of biofilm which resulted in gingivitis and discoloration of the restoration \[^{10}\]. It is dependent on the type of the composite material and polishing system used \[^{11}\]. An increased surface roughness will lead to accumulation of plaque, increasing the occurrence of recurrent caries \[^{12}\]. The step of finishing and polishing composite restorations aims to provide adequate occlusal anatomy, remove small excesses and get a smooth, flawless surface that allows for adequate light reflection\[^{13}\].

The surface roughness depends on various factors, such as: the amount and size of the filler particles and the type of resin matrix of composite restoration, also the type and particle size of the abrasives \[^{14,15}\]. The mechanical properties of a composite, such as hardness and flexural strength, are fundamental to the material in resisting masticatory forces and providing greater longevity. The microhardness of a composite is directly related to the depth of cure of the restorative material. A lower microhardness of a resin composite indicates that the material is more susceptible to scratches and surface defects that can reduce the materials flexural strength and cause premature failure of the restoration \[^{16}\]. The superficial microhardness of resin composites is important for the clinical success of restoration, since the higher the microhardness of restorative material, the better the resistance to surface wear and scratching \[^{17}\].

Therefore, the aim of the present study was to assess the effect of polishing system on the surface roughness and microhardness of NanofillerComposite Z350 and Nanohybrid composite Z250.
MATERIALS AND METHODS:

Two widely used commercial resin composites (Table 1) were evaluated in this study.

Sixty-four samples were prepared in disc-shaped stainless-steel molds with a uniform size of 6 mm in diameter and 4 mm in thickness. Fig.(1) The samples were divided according to the materials used into two groups of 32 samples for each material, and each group was then subdivided into two subgroups according to the polishing instruments with 16 samples in each, Group I, (control group) (Mylar’s strip transparent non glazed) with no finishing and polishing. Group II, polishing with Sof-l ex Pop-on discs. A single operator prepared the samples. Each subgroup was divided into two groups according to measurements of surface roughness and microhardness (n=8). Restorative materials were handled according to the manufacturers’ instructions.

The molds were placed on flat glass plates covered with Mylar’s strips and then were filled with restorative materials. The materials were covered with Mylar’s strips, and a glass slide was pressed against the mold to adapt the materials completely to the inner portions of the molds. The excess material was removed, and the samples were photo-activated for 40 sec at the top surface using high intensity Elipar TM LED light curing unit (3M ESPE), all samples were light cured following the manufacturers’ instructions and, transparent Mylar’s strips were removed immediately after light polymerization and the surface facing the light-curing unit was marked with a small dot using a permanent pen. The specimens’ surfaces in groups II were finished with an ultrafine diamond finishing burrs (859-018-10-UF, Diatech Dental), which were used with a high-speed hand-piece and a water coolant spray. Each bur was applied using light hand pressure in multiple directions for 20 s and was discarded after three times being used.

Then the group II specimens were polished using descending 29 μm(M) 14 μm(F) 5 μm (SF) Sof-L ex Pop On XT aluminum oxide discs, strictly following the manufacturer’s instructions. Fig.(2) Each disc was discarded after use. All of the groups were stored in saline for 24 hr. All of the specimens in each subgroup were equally subdivided for both the surface roughness and micro-hardness tests.
Fig. (1) Disc-shaped stainless-steel mold

Fig. (2) Sof-Lex Pop On XT aluminum oxide discs

TABLE (1) The commercial names, compositions and manufacturers of the materials used

<table>
<thead>
<tr>
<th>Materials</th>
<th>Manufacturer (Lot No.)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z350XT Universal Restorative</td>
<td>3M ESPE, St. Paul, MN, USA (N734682)</td>
<td>Nanofilled composite Bis-GMA, UDMA, TEGDMA, Bis-EMA, discrete nonagglomerated and nonaggregated silica and zirconia fillers of 20 nm and 4-11 nm in size. Filler loading: 63.3% by volume Shade: A2</td>
</tr>
<tr>
<td>Filtek™ Z250XT Universal Restorative</td>
<td>3M ESPE, St. Paul, MN, USA (N764893)</td>
<td>Nanohybrid composite Bis-GMA, UDMA, Bis-EMA, PEGDMA and TEGDMA resins. Fillers: Combination of surface modified zirconia/silica. The inorganic filler loading is 81.8% by weight (67.8% by volume) with a particle size of 20 nm for the silica and approximately 0.1 - 10 µm for the zirconia/silica. Filler loading: 67.8% by volume Shade: A2</td>
</tr>
<tr>
<td>Ultrafine finishing diamond stones</td>
<td>859-018-10-UF, Diatech Dental</td>
<td></td>
</tr>
<tr>
<td>Sof-Lex discs</td>
<td>3M ESPE, St. Paul, MN, USA (46817)</td>
<td>Al2 O3 flexible discs: 29 µm (Medium) 14 µm (Fine) 5 µm (Super Fine)</td>
</tr>
</tbody>
</table>
Surface roughness measurements (Ra): The surface roughness was measured by using Mitutoyo Japan Surftest SJ-210 Surface Roughness Tester (Fig.3). Each specimen is fitted to the specimen holder in which the surface to be measured in horizontal direction, then the specimen holder moves in vertical direction up to the specimen surface just touch the measuring tip. Device calibration is done using the standard calibration specimen before use. Testing parameters:

1- Measuring distance 12 mm
2- Measuring Speed 0.5 mm/s. Returning 1mm/s
3- Measuring force 0.75 mN
4- Stylus profile: tip radius 2-micron, tip angle 60 degree
5- Evaluation parameter Ra values expressed in microns

Three readings are recorded for each specimen at a distance 500 microns each.

Fig. (3) Mitutoyo Japan Surftest SJ-210 Surface Roughness Tester.

Microhardness measurements:

Surface microhardness was measured using Vickers microhardness tester (Wilson Tukon TM1102, Germany) Fig.(4) the 100 gm load is applied smoothly, without impact, forcing the indenter into the test specimen. The indenter is held in place for 10 seconds. The physical quality of the indenter and the accuracy of the applied load must be controlled in order to get the correct results. After the load is removed, the indentation is focused with the magnifying eye piece and the two impression diagonals are measured, usually to the nearest 0.1-μm with a micrometer, and averaged. The Vickers hardness (HV) is calculated using: \[ HV = \frac{1854.4L}{d^2} \]
Where the load L is in gf and the average diagonal d is in μm.

Fig. (4) Vickers microhardness tester (Wilson TukonTM1102, Germany)
**Statistics analysis:**

The data were analyzed using two-way ANOVA used to compare between tested groups and subgroups. Followed by pairwise test for multiple comparisons. A significant level was set at p=0.05 (SPSS IBM, version 23, Armonk, NY, USA).

**Results:**

1- **Surface roughness Ra (um)**

Effect of composite type regardless of other variables Regardless of polishing system; Filtek Z350 XT showed statistically significantly lower mean Ra than Filtek Z250 (P-value = 0.008, Effect size = 0.134) (Table 2 and fig. 5)

**Table 2:** The mean, standard deviation (SD) values and results of ANOVA test for comparison between Ra of the two composite types regardless of other variables. *: Significant at P ≤ 0.05.

<table>
<thead>
<tr>
<th>Composite Type</th>
<th>Mean</th>
<th>SD</th>
<th>Mean</th>
<th>SD</th>
<th>P value</th>
<th>Effect size (Partial eta squared)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z350 XT</td>
<td>0.6125</td>
<td>0.2015</td>
<td>Filtek Z250 XT</td>
<td>0.7267</td>
<td>0.325</td>
<td>0.008*</td>
</tr>
</tbody>
</table>

**Fig. (5):** Bar chart representing mean and standard deviation values for Ra of the two composite types regardless of other variables.
Effect of polishing system regardless of other variables Regardless of composite type, there was a statistically significant difference between mean Ra of polishing system and control group (P value = 0.001, Effect size = 0.256). Pair-wise comparisons revealed that, there was statistically significant difference of Sof-Lex discs; it showed higher mean Ra than mylar’s strip (Table 3 and fig. 6)

Table 3: The mean, standard deviation (SD) values and results of ANOVA test for comparison between Ra values of different polishing systems regardless of other variables *: Significant at P ≤ 0.05

<table>
<thead>
<tr>
<th></th>
<th>Mean</th>
<th>SD</th>
<th>Mean</th>
<th>SD</th>
<th>P-value</th>
<th>Effect size (Partial eta squared)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sof-Lex</td>
<td>0.7033</td>
<td>0.292</td>
<td>0.5490</td>
<td>0.306</td>
<td>0.001*</td>
<td>0.256</td>
</tr>
</tbody>
</table>

Fig. (6): Bar chart representing mean and standard deviation values for Ra of different polishing systems regardless of other variables

2- Microhardness (HV): The mylar’s strip control group showed lower microhardness values compared to the polishing system. The nanohybrid group had an overall higher average microhardness compared to the nano-fill group. (Table 4 and fig.7)
### TABLE (4) Microhardness (HV) in different groups

<table>
<thead>
<tr>
<th></th>
<th>Mylar’s strip</th>
<th>Sof-lex disc</th>
<th>F value</th>
<th>P1 value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Nano-fill composite Z350</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>62.17&lt;sub&gt;B&lt;/sub&gt;</td>
<td>84.21&lt;sub&gt;C&lt;/sub&gt;</td>
<td>41.2</td>
<td>&lt;0.0001*</td>
</tr>
<tr>
<td>Std Dev</td>
<td>9.20</td>
<td>4.11</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min</td>
<td>42.74</td>
<td>79.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Max</td>
<td>68.06</td>
<td>88.33</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Nano-hybrid composite Z250</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>92.65&lt;sub&gt;A&lt;/sub&gt;</td>
<td>99.75&lt;sub&gt;B&lt;/sub&gt;</td>
<td>15.65</td>
<td>&lt;0.0001*</td>
</tr>
<tr>
<td>Std Dev</td>
<td>4.97</td>
<td>7.67</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min</td>
<td>88.47</td>
<td>89.11</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Max</td>
<td>100.14</td>
<td>111.64</td>
<td></td>
<td></td>
</tr>
<tr>
<td>F value</td>
<td>88.69</td>
<td>11.78</td>
<td>F= 62.08</td>
<td></td>
</tr>
<tr>
<td>P value</td>
<td>&lt;0.0001*</td>
<td>0.0002*</td>
<td>P&lt;3&lt;0.0001*</td>
<td></td>
</tr>
</tbody>
</table>

Significance level p<0.05*significant

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**Fig. (7)** Surface hardness (Vickers hardness number) of the control and polished groups
**Discussion:**

Proper finishing and polishing are critical steps to enhance the esthetics and long-lasting quality of resin composite restorations. \(^{18-19}\) The surface roughness of the restorations can influence dental biofilm retention, staining, gingival inflammation, and secondary caries, thus affecting the clinical performance of restorations. Composite resin restorations have evolved rapidly over the past decade. Advances, such as nanohybrids and nano-filled composites.

As regards results of surface roughness in the current study were showed that control group had exhibited significantly lower roughness values (smoothest surface) than soflex disc (P<0.0001). This finding is in agreement with other studies that showed that mylar’s strip group exhibited significantly lower roughness values than the polishing systems (P<0.0001). \(^{20,21,22,23}\) However, resin composite surface not treated with any cutting instruments and didn’t use any finishing and polishing systems. The filler particles that were not abraded away from the resin matrix, which finally led to the creation of the smoothest surface of the tested resin composites \(^{24}\). Moreover, the smoothest surface of resin composite is achieved under Mylar’s strip, but this surface cannot be stabled clinically due to no flat tooth surface exists; otherwise, the complex tooth morphology will necessitate the clinician to make finishing and polishing for the restoration to reassemble the tooth complex morphology \(^{25}\). Furthermore, control group (Mylar’s strip) the resulting surface is polymer-rich and provides the restoration relatively unstable. Moreover, this resin-rich surface should be removed since it can easily wear in the oral environment. In addition, the oral environment will be exposed to inorganic filler content if no polishing procedure is carried out. So, this layer clinically is abolishing during removal of excess material or contouring of the restoration. The step of finishing and polishing procedures is an important factor in the clinical success of composite resin restorations \(^{20}\)

Filtek Z350 XT composite showed statistically significantly lower surface roughness (0.6125) than Filtek Z250 XT composite (0.7267) (Table 2 and figure1)

This result could be attributed to different fillers’ composition, size and loading of both tested materials. During the polishing procedure, in Filtek Z350 XT, nanomer and nanocluster particles were abraded easily along with the resin matrix. The nanomer bond which constructs nanoclusters would detach, providing a smoother surface. Also,
nanomer was added with silane on its surface, which creates a strong bond with the matrix during curing. The matrix system contains more Bis-GMA and UDMA with less double bonds, increasing the degree of polymerization \[22,26,27\]. While in Filtek Z250 XT resin composite, larger and irregular filler size was obtained by grinding larger particles and causing a lot of space between fillers. The larger filler would appear protrusive on the surface during curing. Pressure would gather more on the irregular filler and increase the chance of the filler detaching from the resin surface. When the larger filler detached from the matrix, it would create a large hole on the surface and increase surface roughness \[22,26\].

Additionally, Filtek Z250 XT resin composite still uses PEGDMA as a main matrix with more double bonds than Bis-GMA and UDMA, making the curing process less adequate than Filtek Z350 XT resin composite \[22,26\].

Difference in surface roughness between Filtek Z350 XT and Filtek Z250 XT resin composites on the basis of differences in their chemical composition; nano-filled Filtek Z350 XT resin composite contains nanoparticles with an average size of 11 nm while nanohybrid Filtek Z250 XT resin composite has an average particle size of 0.6 μm. \[22\]

**Microhardness** is defined as the blocking resistance that prevents the creation of permanent deformation and hardness is the most important feature contributes the success of clinical utilizations. A high microhardness value eventuates increasing the scratch and abrasion resistance, meanwhile prevents the material easily deformed against various forces \[27\].

In the present study, it was found that the control group finished with Mylar strip showed lower microhardness values than the groups that have undergone polishing. This in agreement with other studies that reported that Mylar strip produced perfectly smooth restoration surface, although it is rich in the resin organic binder. Finishing and polishing in such a case result in harder, more wear resistant and esthetically pleasing surface which is attributed to the removal of the superficial resin layer \[28,29,30\].

The nanohybrid group had a higher microhardness than the nano fill group. This could be due to that the nanohybrid resin, has a higher filler loading of 82% by weight (68% by volume) as compared with the nanofill resin which has an Inorganic filler loading is 78.5% by weight (63.3% by volume). \[31\]
Conclusions:
Within the limitations of the current study, it could be concluded that:

1- The control group (Mylar’s strip) had the lowest surface roughness and microhardness values compared to the polishing group.
2- Nano-fill composite showed lower surface roughness compared to nanohybrid composite.
3- Nanohybrid composite had a higher microhardness value when compared with nano-fill composite.

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