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ORIGINAL ARTICLE

Effect of Natural and Industrial Juices on Surface Microhardness of Microhybrid and Nanohybrid Composites

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ABSTRACT

Numerous studies have been conducted on the effect of various beverages on the mechanical properties of tooth-colored materials, however, little is known about the effects of these materials on composites, particularly newer types such as nanocomposites. Objectives: This study aimed to evaluate the surface microhardness of two types of composites, micro-hybrid (point 4) and nanohybrid (premise), after exposure to natural and industrial juices. Methods: In this experimental study, 90 disc-shaped specimens with a thickness of 2 mm and a diameter of 10 mm were taken from two composites, micro-hybrid Point4 (Kerr) and nanohybrid premise (kerr) (two groups of 45). Then, the specimens of each group were divided into 5 subgroups of 9 and were immersed for 7 days for 6 hours in 5 solutions of industrial orange juice, natural orange juice, industrial pomegranate juice, natural pomegranate juice, and distilled water (control group). Surface microhardness of specimens was measured by Vickers device at baseline, one day and one week after immersing. Data was measured by ANOVA, repeated measure test, and independent t-test. A significant level of α was 0.05. Results: The surface microhardness of two types of composites exposed to beverages was reduced significantly. However, no significant difference was found between natural and industrial juices in none of the composites. Conclusions: Natural and industrial juices can affect the surface microhardness of composites, which varies depending on the type of composite and the type of juice and immersion time.

Key words: juice, microhybrid composite, nanohybrid composite, restorative dentistry, surface microhardness

INTRODUCTION

Despite preventions, dental caries is still one of the most prevalent chronic childhood diseases worldwide.1 Deciduous tooth is important in creating and developing the ideal occlusion, nutrition, and baby health, thus it is important to maintain them using a variety of restorative methods.2

A suitable restorative material should have features such as acceptable mechanical properties, protection against tooth decay, easy use in the clinic, aesthetics, and maintaining the inherent properties in the oral environment.3 Therefore, the use of composites has increased due to the ability to bond to the tooth structure, meet aesthetic needs, and improve mechanical properties in restorative dentistry.4 Composite resins consist of four main parts, including organic polymer matrix, non-organic filler particles, coupling agent, and initiator-accelerator system.5,6 One of the methods of classifying composites is based on the size and shape of filler particles and how they are distributed. Accordingly, three groups of composites that are more widely used today include microfill, micro-hybrid, and nanohybrid composites.5,6

The mouth is considered as an ideal environment for predicting the behavior of restorative materials, and
these restorative materials require durability in this environment.\textsuperscript{7,8} Since composites change after being placed in the oral environment, the role of diet has been considered, and among these, soft drinks and juices play a more effective role.\textsuperscript{3,9}

Children and adults are large consumers of non-alcoholic beverages; thus these substances can have a significant effect on restorative materials.\textsuperscript{7,10} Numerous studies have been conducted on the effect of various beverages, especially alcoholic beverages, carbonated drinks, and orange juice on the mechanical properties of tooth-colored materials, including glass ionomers and compomers.\textsuperscript{9,11-13} However, little is known about the effects of these materials on composites, particularly newer types such as nanocomposites. Surface microhardness of restorative materials is an important feature for predicting mechanical properties such as abrasion resistance\textsuperscript{4,8} thus studies that examine the hardness of materials are important. Therefore, this experimental study tends to evaluate and compare the effect of natural and commercial beverages (pomegranate juice and orange juice) on the surface microhardness of micro-hybrid and nanohybrid composites.

### METHODS

#### Preparing specimens

Two composite, Point 4 micro-hybrid (Kerr, Bolzano, Italy) and Premise nanohybrid (Kerr, Bolzano, Italy) shade A3 were used; specifications of the used materials are listed in Table 1. The sample size was computed based on a previous study by Hashemikamangar et al., which studied the effect of organic acids in dental biofilm on the microhardness of a silorane-based composite.\textsuperscript{14} With a 90% power and 95% confidence interval, the minimum sample size was statistically derived as 43. Forty-five specimens of each composite were taken by a cylindrical mold with a thickness of 2 mm and a diameter of 10 mm.

When taking the specimens, two glass slabs below and above the mold were used to create a smooth surface and prevent the formation of non-polymerized layers. After placing the composite pieces inside the mold it was packed by a condenser to prevent the formation of the composite bubble. Once the mold was full, another glass slab was placed on it and a 5kg weight was placed on it for 3 min to ensure complete removal of bubbles and uniformity of specimen.\textsuperscript{15}

The specimens were then cured for 60 seconds on both sides (120 secs in total) by a 550 mW/cm\textsuperscript{2} light cure device (Bonart Co Ltd, New Taipei City, Taiwan). In the next step, the specimens were polished by silicon carbide paper disks (Sof-Lex Pop On; 3M ESPE, St. Paul, MN, USA) to provide a smooth surface of composites. The final thickness of the discs was 2 mm and all areas were measured with a caliper (Mitutoyo, Japan).

In the next step, all specimens were kept in distilled water for 48 hours for primary water absorption and full polymerization process, and proximity to oral conditions. After this time, the initial microhardness of the composites was measured by the Vickers hardness test, Micromet (Buehler, Lakebluff, USA).

#### Microscopic evaluation

To measure the hardness, the specimens were first placed on the desired position on the device and their surface was examined by 40x magnification (Olympus CX1, Tokyo, Japan) so that the surface on which force was applied was free of any bubbles and other defects. Vickers Hardness Test

The 100 g force was applied on the specimen for 20 seconds by a pyramid-shaped diamond roller 16. The point on which force was applied by roller was recorded on the specimen in the form of a positive mark, and this was done at three different points on the surface of each specimen at a distance of more than one millimeter from the margin. Then the horizontal and vertical

### Table 1. Characteristics, manufacturers, and constitutes of the composites used in this study

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Resin system</th>
<th>Filler type</th>
<th>Filler size/ filler content</th>
<th>Category</th>
<th>BRAND</th>
<th>Shade</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kerr, Bolzano, Italy</td>
<td>Bis GMA- BisEMA-TEGDMA</td>
<td>Bariumaluminaboro-silicate glass, silica nanofiller, PPF, barium glass, discrete nanofiller</td>
<td>Glass:0.4µm Silica:0.02µm 68% vol 82% wt</td>
<td>Nanohybrid composite</td>
<td>Premise (A3)</td>
<td></td>
</tr>
<tr>
<td>Kerr, Bolzano, Italy</td>
<td>Bis GMA-BisEMA-TEGDMA</td>
<td>Bariumaluminaboro-silicate glass silicon dioxide Barium glass</td>
<td>0.4µm 57% vol 76% wt</td>
<td>Microhybrid composite</td>
<td>Point 4 (A3)</td>
<td></td>
</tr>
</tbody>
</table>
dimensions of this mark were measured separately and substituted in the following formula:

$$HV = 1.854F/d$$

where $F$ is the force applied and $d$ is the average impact diameter of each roller. For each specimen, 3 numbers were obtained; to increase the accuracy of the study, the mean of these three numbers was measured and recorded.

Specimens of each type of composite were randomly divided into 5 subgroups ($n=9$)

- **Group 1**: 9 micro-hybrid composites immersed in natural pomegranate juice.
- **Group 2**: 9 micro-hybrid composites immersed in industrial pomegranate juice (Sunich, Iran).
- **Group 3**: 9 micro-hybrid composites immersed in natural orange juice.
- **Group 4**: 9 micro-hybrid composites immersed in industrial orange juice (Sunich, Iran).
- **Group 5**: 9 micro-hybrid composites immersed in distilled water.
- **Group 6**: 9 nanohybrid composites immersed in natural pomegranate juice.
- **Group 7**: 9 nanohybrid composites immersed in industrial pomegranate juice (Sunich, Iran).
- **Group 8**: 9 nanohybrid composites immersed in natural orange juice.
- **Group 9**: 9 nanohybrid composites immersed in industrial orange juice (Sunich, Iran).
- **Group 10**: 9 nanohybrid composites immersed in distilled water.

Then, the microhardness of the specimens was examined after 6 h (one day) and 42 h (7 days) being immersed in the beverages. The specimens were immersed 6 hours a day in the beverages and kept in distilled water at 37° when not immersed in the beverages.

The beverages were used at their usual temperature at 4°. The beverages were also replaced every day to prevent possible interactions, and their pH was checked regularly and no difference was seen in pH during this week.

**Statistical analysis**

Data obtained from the present study were analyzed using SPSS22. ANOVA test was used to compare different groups of composites and drinks, RM (repeated measure) test was employed to check the trend of time variation. An independent t-test was used to compare the effect of natural and industrial juices. p-value $<0.05$ was statistically considered as a significant level.

### RESULTS

**Effect of natural and industrial juices on surface microhardness of micro and nanohybrid composites**

Mean values and standard deviations of surface hardness of different composites in the immersion solutions at baseline, after 6 and 42 hours are presented in table 2 and Figures 1 and 2. The effect of all 5 beverages was significant on the surface microhardness of micro and nanohybrid composites at three periods ($p<0.001$).

A pairwise comparison of time intervals in each group of drinks showed a significant decrease in each measurement stage compared to the previous stage. Only in water beverages, no significant difference was found between the baseline time and 6 hours after immersion steps in both composites ($p>0.05$).

**Effect of natural versus industrial juice**

An independent t-test was used to compare the effect of natural and industrial juices on the surface
Table 3. Effect of natural versus industrial juices on the surface microhardness of micro-hybrid and nanohybrid composites

<table>
<thead>
<tr>
<th>Composite</th>
<th>Beverage</th>
<th>Period</th>
<th>Natural Mean ±SD</th>
<th>Industrial Mean ±SD</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microhybrid</td>
<td>Pomegranate juice</td>
<td>Baseline</td>
<td>34.05±1.40</td>
<td>34.96±1.59</td>
<td>0.214</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 hours</td>
<td>32.62±1.93</td>
<td>32.52±1.93</td>
<td>0.339</td>
</tr>
<tr>
<td></td>
<td></td>
<td>42 hours</td>
<td>31.22±2.09</td>
<td>29.83±1.96</td>
<td>0.266</td>
</tr>
<tr>
<td>Orange juice</td>
<td>Pomegranate juice</td>
<td>Baseline</td>
<td>40.42±4.35</td>
<td>38.97±8.39</td>
<td>0.652</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 hours</td>
<td>38.50±4.14</td>
<td>37.73±8.85</td>
<td>0.816</td>
</tr>
<tr>
<td></td>
<td></td>
<td>42 hours</td>
<td>32.68±3.83</td>
<td>33.95±7.67</td>
<td>0.662</td>
</tr>
<tr>
<td>Pomegranate juice</td>
<td>Baseline</td>
<td>57.06±2.04</td>
<td>55.79±1.88</td>
<td>0.189</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 hours</td>
<td>56.08±2.13</td>
<td>54.64±2.12</td>
<td>0.156</td>
</tr>
<tr>
<td></td>
<td></td>
<td>42 hours</td>
<td>54.34±2.58</td>
<td>51.94±1.87</td>
<td>0.120</td>
</tr>
<tr>
<td>Nanohybrid</td>
<td>Orange juice</td>
<td>Baseline</td>
<td>55.11±2.67</td>
<td>53.97±1.57</td>
<td>0.287</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 hours</td>
<td>52.66±1.88</td>
<td>52.34±1.87</td>
<td>0.954</td>
</tr>
<tr>
<td></td>
<td></td>
<td>42 hours</td>
<td>48.60±2.60</td>
<td>49.17±1.83</td>
<td>0.350</td>
</tr>
</tbody>
</table>

SD: standard deviation
As shown in Table 3, there was no significant difference between means of surface microhardness of micro and nanohybrid composites in natural and industrial juices at baseline, 6 hours, and 42 hours after being immersed (p>0.05). Comparison of surface microhardness of micro-hybrid and nanohybrid composites after the impact of natural and industrial juices. For this purpose, first, the number of microhardness changes of micro-hybrid and nanohybrid composites from the baseline stage up to 24 hours was calculated and then an independent t-test was used to compare them. The results showed that beverages led to more reduction in surface microhardness of micro-hybrid composite than nanohybrid composite; however, no significant difference was found (p>0.05) (Table 4 and Figure 3).

**DISCUSSION**

In this study, the micro-hardness of both composites increased slightly after 6 hours of immersion of the composites in distilled water, which was not statistically significant. This slight increase in surface microhardness can be due to cross-reactions in the post-curing resin matrix that cause the monomer to form and allow the chemical bond to continue to form. This result has been obtained in some studies, including Abubakr et al.\(^1\) and Okte et al.\(^17\)

According to the results of the present study, the initial surface microhardness of Premise nanohybrid composite was greater than that of microhardness Point 4 composite, and these different behaviors could be due to differences in filler composition and distribution in their matrices. The filler content in the Point 4 composite is 76% by weight, which is less than the premise composite. According to studies, the lower the filler content, the lower the hardness.\(^11,18,19\)

This result has been obtained in some studies, including Ahmadizenouz et al.\(^20\) and Badra et al.\(^21\)

However, Erdemir et al. reported that the initial hardness of Premise nanohybrid composite was less than that of Z250 micro-hybrid composite,\(^22\) which was inconsistent with the present study. The difference can be due to the difference in the type of filler particles of these two composites. It is noteworthy that the present study used the same filler particles for both composites. The main results of the study were that hardness of both point 4 and premise composites showed a significant reduction following 6 and 42 hours of immersion in natural and industrial juices compared to baseline, which could be due to several factors. Juices contain water and absorption of water can cause swelling and reduce the frictional forces between the polymerized chains and soften the resin, thus removing filler particles from the material surface and causing surface roughness, and reducing surface hardness.

The acidic pH of beverages leads to softening of the matrix, surface abrasion, and destruction of structural ions. The type of acid in beverages, including citric acid used in beverages present in this study, can penetrate the resin matrix and release non-reactive monomers and reduce surface hardness.\(^23,24\) Therefore, water absorption by resin matrix and acidic pH and acid matrix solubility have a synergistic effect in reducing the surface hardness of the composite.\(^11\) Another factor that can be noted is the presence of bubbles that can be created during the placement of composites and decompose the composite material by absorbing focal water.\(^11\)

This finding is consistent with Ahmadizenouz et al., who found that surface microhardness of all composites (p90, Z250, Z350 XT) significantly decreased in energy drinks at both times (1 week later and 1 month later).\(^20\) Coinciding with this study, Fatima N and Hussain M examined the effect of two common energy drinks on surface micro-hardness of tooth-colored restorative materials and showed that surface micro-hardness of all three restorative materials (Vitrofill, Vitremere, Filtek Z350 XT) was significantly reduced.\(^25\)

Erdemir et al. reported that surface hardness of all restorative materials studied, including premise nanohybrid composite, micro-hybrid composite...
(filtek Z250), nanofilled composite (Supreme XT), and compomer (Compoglass), was significantly reduced after 6 months of immersion in Energy drinks.\textsuperscript{22} Yesilyurt et al. investigated the effect of food-simulating liquids on the mechanical properties of four different types of composites and reported similar results.\textsuperscript{26}

This study also found that both natural and industrial juices reduced the surface microhardness of composites, although there was no significant difference between them. According to studies, the abrasive ability of a beverage depends on pH and acidic composition of the beverage \textsuperscript{23,27} and pH of the juices used in this study are very close to each other (natural orange juice = 4.5, industrial orange juice = 4.2, natural pomegranate juice = 4, industrial pomegranate juice = 5) and their main acidic compound was citric acid; this could be a convincing reason for the result.

Tanthanuch S et al. investigated the effect of different beverages (apple juice, orange juice, carbonated drinks, coffee, wine) on surface hardness of nanohybrid composite and glass ionomer and observed that carbonated drinks had a stronger acidic composition of carbonic acid and phosphoric acid in the structure and the lowest pH; thus, it had the greatest effect on microhardness of both materials.\textsuperscript{24} Erdemir et al. also found that energy and sports drinks reduced surface hardness more than the distilled water control group and this was attributed to their low pH and the presence of acidic compounds (citric acid) in beverages. They also found that there was no significant difference between the effects of beverages due to the close pH of the beverages.\textsuperscript{28}

In this study, all beverages reduced surface microhardness of Point 4 micro-hybrid composite compared to premise nanohybrid composite, but no significant relationship was observed. As noted earlier, these two composites are the same in many respects, including the type of resin system, the type of filler, the manufacturer, and so on. Moraes et al. compared the behavior of nanohybrid composites with micro-hybrid and nanofilled composites; all evaluations showed that the composite behavior of nanohybrid composites was more similar to that of micro-hybrids; in fact, nanohybrid composites do not behave like nanofillers, but are similar in behavior or even better than micro-hybrids.\textsuperscript{29} Consistent with this study, Poggio et al. observed in their study that surface microhardness of micro-hybrid composite (Gradia Direct Registered) was further reduced than nanohybrid composite (admiria fusion) after one week of immersion in beverages.\textsuperscript{30}

In contrast to the present study, Erdemir et al. found that surface hardness of nanohybrid composite (premise) was more reduced than micro-hybrid composite (Z250) under the influence of beverages which could be due to the differences in the composition of these two composites.\textsuperscript{22} The chemical characteristics of the composites tested, as the presence of nanofillers can alter the mechanical behavior of the materials tested. Additionally, also surface alterations due to wear or brushing can alter the composite structure. Therefore, future studies are needed to test the effect of wear and filler size on adhesion repair.\textsuperscript{30,31,32}

Within the limitations of this in-vitro study, it can be concluded that natural and industrial juices have a significant damaging effect on the surface microhardness of composite resins, and these effects are increased with duration of exposure so the children who have a regular diet of such drinks should consider this issue. In our study, the composition of composite resins had not a noticeable effect on the surface microhardness changes.

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