



## Comparison Of The Effect Of Acid Type Of Cuko Pempek On The Surface Hardness Of Microhybrid And Nanohybrid Composite Resin

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### Abstract

**Introduction:** Hybrid composite is often used nowadays because of the combination of filler size results in restorations with good aesthetic and mechanical strength. Various factors in the oral cavity such as acid solution can affect the surface hardness of composite resin. Cuko pempek is an additional sauce that served for pempek which has acidic pH. This study aimed to compare the effect of acid type of cuko pempek on surface hardness of microhybrid and nanohybrid composite. **Materials and Methods:** Thirty-six samples of microhybrid (n=18) and nanohybrid composite (n=18) with 5 mm in diameter and 2 mm in thickness were immersed in three different solutions: artificial saliva (control), cuko pempek using vinegar, and cuko pempek using tamarind. Surface hardness was measured before and after immersion using Vickers Hardness Tester. **Results:** The result of this study showed a significant difference on the surface hardness of composite resin before and after immersion ( $p < 0,05$ ). The surface hardness of microhybrid and nanohybrid composites after immersed in artificial saliva and cuko pempek ( $p < 0,0167$ ) showed statistically significant differences, while there were no significant differences between cuko pempek using vinegar and tamarind ( $p > 0,0167$ ). This study also showed that there were significant differences on the surface hardness of microhybrid and nanohybrid composite after immersion in artificial saliva, cuko pempek using vinegar, and cuko pempek using tamarind ( $p < 0,05$ ). **Conclusion:** The surface hardness of nanohybrid composite was lower than microhybrid composite after immersed in cuko pempek using vinegar and tamarind.

Keywords: cuko pempek, composite resin, surface hardness.

### Introduction

One of the most commonly used restoration materials in dentistry is composite resin. This material is the choice because it has aesthetics, is easy to manipulate, and is relatively resistant to abrasion than other aesthetic materials.<sup>1,2</sup> Composite resins are composed of 3 main components, namely a resin matrix, filler particles, and a coupling agent.<sup>1</sup> There are several types of composite resins based on the size of the filler particles, namely traditional composites (10-100  $\mu\text{m}$ ), composites made from small particle fillers (0, 1-10  $\mu\text{m}$ ), composites made from microfillers (0.01-0.1  $\mu\text{m}$ ), and hybrid composites.<sup>1,2</sup>

Hybrid composite resins are now often used because the combination of the two filler particles results in restorations with good aesthetic and mechanical strength.<sup>1</sup> These composite resins can be applied to both posterior and anterior teeth.<sup>3</sup> Hybrid composite resins



are divided into microhybrid and nanohybrid. Microhybrid composite resin is a combination of fine particles with a particle size of 0.1-10  $\mu\text{m}$  and several microfine particles with a particle size of 0.01-0.1  $\mu\text{m}$ . Nanohybrid composite resin combines microparticles measuring 0.1-2  $\mu\text{m}$  and nano-particles of  $\leq 100$  nm.<sup>1</sup> The size of the filler particles can affect the physical and mechanical properties of a material. Small filler particle has a larger surface area which causes an increase of the interaction between the matrix and the filler particles, thereby increasing the properties of the composite.<sup>1,4</sup>

One of the mechanical properties of composite resin is surface hardness. It is an important property used to predict material wear resistance and its ability to abrade the opposing teeth.<sup>1</sup> Factors that can affect surface hardness include solubility, water absorption, polymerization of materials, thickness of composite resin, and exposure time.<sup>5-7</sup> It is also affected by food or beverages consumed.<sup>8,9</sup>

Composite resin has decreased surface hardness after immersion in orange juice and carbonated drinks.<sup>9</sup> Both drinks have a low pH or are acidic.  $\text{H}^+$  ions in acidic solution can diffuse into the composite resin and bind the negative ions contained in the siloxane (Si-O-Si) bonds, causing degradation of the siloxane bonds. Degradation of the siloxane bonds breaks the bonds formed between the filler particles and the coupling agent. It causes the release of filler particles and a decrease in the surface hardness of the composite resin.<sup>9,10</sup> It may lead to the malfunction of a restoration.<sup>11</sup>

Cuko pempek is an acidic solution that is commonly used as a companion sauce for eating pempek, a typical Palembang culinary made from fish and sago.<sup>12</sup> The consumption of pempek in Palembang City in 2010 reached 243.91 tons per month. Online sales of pempek through PT Pos Kota Palembang even reached 8 tons per month for both domestic and foreign destinations, especially ASEAN countries such as Malaysia, Singapore, and Thailand.<sup>13</sup> Cuko pempek has a distinctive sour, sweet, and spicy taste.<sup>12</sup> The sour taste of cuko pempek is obtained from vinegar (acetic acid) or tamarind (*Tamarindus indica*).<sup>14</sup>

Vinegar used for cooking usually contains acetic acid with a concentration of  $\pm 25\%$ .<sup>15</sup> Acetic acid in food vinegar is obtained by utilizing *Acetobacter* bacteria to oxidize ethanol found in wine or other fruit juices. Food vinegar sold in the market nowadays is mostly a dilution of concentrated acetic acid.<sup>16</sup> Vinegar has a pH of 3.0.<sup>17</sup>

Tamarind obtained from the *Tamarindus indica* plant. It is a kind of sour fruit, commonly used as a spice in many Indonesian dishes as an enhancer of a sour taste in food.<sup>18</sup>



Tamarind contains apple acid, citric acid, grape acid, tartaric acid, succinic acid, pectin, and invert sugar. The pH of tamarind extract is 2-3.<sup>18,20</sup>

When exposed to acid continuously, the filler particles in the composite may be leached out. It will lead to a decrease in the mechanical properties of the composite resin. Filler particle size can also affect the strength of the composite resin as a restoration. This study aims to determine the effect of the type of acid on cuko pempek on the surface hardness of the micro-hybrid and nanohybrid composite resins.

### Methods

This is an experimental research design with a pretest-posttest with a control group. The research was conducted at the Laboratory of Materials Mechanical Engineering, Sriwijaya University, and the Laboratory of Microbiology, Faculty of Medicine, Sriwijaya University.

Two types of composite resin used as permanent dental fillings, micro-hybrid (Filtex™ Z250 3M ESPE, USA) and nanohybrid (Filtex™ Z250 XT 3M ESPE, USA) composite resin, were tested and divided into 6 groups (n=6). Each type of composite were immersed in artificial saliva, in cuko pempek made with vinegar acid, and in cuko pempek made with tamarind.

A total of 36 specimens were prepared using metal ring mold (5 mm in diameter, 2 mm in height). Seluloid strip was placed on the bottom and on top of the mold, closed with a cover glass and then the excess material was removed. Specimens were light cured for 20 minutes, the tip of the light curing unit is perpendicular to the surface of the cover glass. Specimens were removed from the mold and marked with a marker. The hardness test on the upper surface of the sample before immersion was carried out with the Vickers Hardness Tester for three indents at different points.

Group A, B, and C are microhybrid whereas group D, E, and F are nanohybrid. The control groups (A and D) were immersed in 30 ml of artificial saliva for 14 days and stored in an incubator at 37 ° C. Artificial saliva was renewed every 24 hours. The test groups (B, C, E, and F) were immersed in 30 ml of cuko pempek for 13 minutes every day at room temperature ( $\pm 25^\circ$ ). The length of immersion time is based on observing the average consumption habit of pempek at one time. Then the sample was rinsed with 30 ml of distilled water, immersed in 30 ml of artificial saliva, and stored in an incubator at 37°. Immersion



was carried out for 14 days and the solution was renewed every 24 hours. The surface hardness test after immersion is carried out in the same manner.

The data obtained were analyzed using the Shapiro-Wilk test for normality test and Levene's Variance test for homogeneity test. Followed by Paired T Test to determine the significance of changes in surface hardness of micro-hybrid and nanohybrid composite resins before and after immersion in 1 group. One Way ANOVA test was conducted to determine the significance of differences in surface hardness between groups of micro-hybrid and nanohybrid composite resins after immersion, complemented by Independent t-test.

**Results**

This study involved 18 samples of micro-hybrid composite resin and 18 samples of nanohybrid composite resin. Each sample was divided into three groups based on the immersion solution. Before immersion, the pH of each solution was measured. The results can be seen in Table 1.

**Table 1.** pH value of immersion solution.

The type of immersion solution	pH
Artificial saliva	7
Cuko pempek made with vinegar acid	4,4
Cuko pempek made with tamarind	3,7

Table 1 shows that cuko pempek made with tamarind has the lowest pH value or most acidic compared with other solutions. Comparison of the surface hardness values of micro-hybrid and nanohybrid composite resins before and after immersion can be seen in Table 2.

**Table 2.** The Mean of Surface Hardness (VHN) Value of Microhybrid and Nanohybrid Composite Resins Before and After Immersion

Group	Before immersion (Mean±SD)	After immersion (Mean±SD)	P value
A	2,646±0,268	2,047±0,069	0,002*
B	2,696±0,247	1,917±0,041	0,001*
C	2,695±0,189	1,801±0,078	<0,0001*
D	2,571±0,246	1,871±0,056	0,001*
E	2,541±0,245	1,717±0,048	<0,0001*
F	2,510±0,242	1,603±0,092	0,001*

- Group A: micro-hybrid immersed in artificial saliva
  - Group B: micro-hybrid immersed in cuko pempek made with vinegar
  - Group C: micro-hybrid immersed in cuko pempek made with tamarind
  - Group D: nanohybrid immersed in artificial saliva
  - Group E: nanohybrid immersed in cuko pempek made with vinegar
  - Group F: nanohybrid immersed in cuko pempek made with tamarind
- (\*) showed significant difference (p<0,05)



Paired T-test results showed significant difference between micro-hybrid and nanohybrid before and after immersion ( $p < 0.05$ ). The lowest surface hardness of the composite resin after immersion occurred in micro-hybrid and nanohybrid composite resins immersed in cuko pempek with tamarind.

The differences in surface hardness between groups after immersion based on the type of composite resin used were analyzed. The surface hardness value of micro-hybrid composite resin after immersion was tested for normality to see the distribution of data and homogeneity test to see data variance. The results of the normality test using the Shapiro-Wilk test showed normal distribution ( $p > 0.05$ ), while the homogeneity test using Levene's Variance test showed that the variance was different ( $p < 0.05$ ). The data analysis used was Welch's One Way ANOVA test (Table 3).

**Table.3** Value of Surface Hardness (VHN) of Microhybrid Composite Resins After Immersion

Group	N	Mean±SD	P value
A	6	2,047±0,069	
B	6	1,917±0,041	0,001*
C	6	1,801±0,078	

(\*) showed significant difference ( $p < 0.05$ )

The Post Hoc Games-Howell test was then carried out to see the average difference in surface hardness values between groups. The results showed that there was a significant difference in the surface hardness of the micro-hybrid composite resin between the artificial saliva and cuko pempek with vinegar, and between artificial saliva and cuko pempek with tamarind. There was no significant difference between the two test groups ( $p > 0,0167$ ).

Normality test and homogeneity test were also carried out on the surface hardness value of the nanohybrid composite resin after immersion. The results of the normality test using the Shapiro-Wilk test showed a normal distribution ( $p > 0.05$ ) and the homogeneity test using the Levene's Variance test showed the same variance ( $p < 0.05$ ). The data analysis used was the One Way ANOVA test (Table 4).

**Table 4.** Value of Surface Hardness (VHN) of Nanohybrid Composite Resins After Immersion

Group	N	Mean±SD	P value
D	6	1,871±0,056	
E	6	1,717±0,048	<0,0001*
F	6	1,603±0,092	

(\*) showed significant difference ( $p < 0.05$ )



The difference in mean surface hardness values between groups was analyzed by using the Post Hoc Bonferonni test (Table 5). The results shows that there are significant difference in surface hardness of nanohybrid composite resin groups immersed in artificial saliva and in cuko pempek with vinegar, and between groups immersed in artificial saliva and cuko pempek with tamarind ( $p < 0,0167$ ). There are no significant difference between groups immersed in cuko pempek.

Table 5. Post Hoc Bonferonni Results on NanoHybrid Before And After Immersion

Groups		Mean difference	P value
Group D	Group E	0,154	0,004*
	Group F	0,268	<0,0001*
Group E	Group F	0,114	0,033

(\*) showed significant difference ( $p < 0,0167$ )

Independent T test analysis was then performed to see the significance of the difference in the mean surface hardness of the micro-hybrid and nanohybrid composite resins after immersion in each immersion solution (Table 6).

Groups		Mean Difference	P value
Group A	Group D	-0,177	0,001*
Group B	Group E	-0,200	<0,0001*
Group C	Group F	-0,198	0,002*

(\*) showed significant difference ( $p < 0,05$ )

The results showed that there was a significant difference in hardness between micro-hybrid and nanohybrid composite resins after immersion in artificial saliva (Groups A and D), cuko pempek with vinegar (Group B and E), and cuko pempek with tamarind material (Group C and F).

## Discussions

The durability of restorative materials in the mouth is affected by the surface hardness to predict the resistance of materials to wear.<sup>1</sup> Water absorption is one of the factors that can affect the surface hardness of composite resin.<sup>21</sup> Water molecules can diffuse into the resin matrix polymer chain that causes hydrolysis events. The polymer has a carboxyl group that will bind with water to form weak hydrogen bonds, and breaking the bond between the resin matrix and the filler particles. This degradation process causes a decrease in the surface hardness of the composite resin.<sup>21,22</sup>



Based on this study, the micro-hybrid and nanohybrid composite resins experienced a decrease in hardness after immersion in artificial saliva, cuko pempek with vinegar, and cuko pempek with tamarind ingredients. It will lead to malfunctions in restoration.<sup>11</sup>

The surface hardness of the micro-hybrid and nanohybrid composite resins immersed in artificial saliva had a significant difference with the composite resins immersed in cuko pempek. Cuko pempek made from vinegar and tamarind has a lower pH than artificial saliva. The results of the research of Tanthanuch et al.<sup>23</sup> and Erdemir et al.<sup>24</sup> showed that composite resins experienced a greater surface hardness reduction after immersion in several types of beverages with low pH value.  $H^+$  ions contained in low pH or acidic solutions diffuse into the composite resin. These ions bind to negative ions contained in the siloxane bonds resulted in degradation of the siloxane bonds. It causes a break in the bond between the filler particles and the coupling agent and may cause debonding of the filler particles that leads to a decrease in surface hardness.<sup>10</sup>

The surface hardness of the micro-hybrid and nanohybrid composite resins immersed in cuko pempek with tamarind was lower than that of the specimens immersed in cuko pempek with vinegar, but the difference was not statistically significant. Cuko pempek with vinegar (pH 4.4) and tamarind (pH 3.7) classified as a weak acid. Weak acids are acidic compounds that only partially ionize in solution.<sup>25</sup>

The vinegar contained in cuko pempek is the result of dilution of pure acetic acid which is made industrially.<sup>16</sup> Acetic acid ( $CH_3COOH$ ) is a weak acid.<sup>25</sup> Tamarind (*Tamarindus indica*) used as an acidic ingredient in cuko pempek contains organic acids that also classified as weak acid, such as tartaric acid, acetic acid, citric acid, formic acid, malic acid, and succinic acid.<sup>26</sup> This combination is thought to cause the pH of cuko pempek with tamarind to be lower than cuko pempek with vinegar.

The results of this study indicated that the nanohybrid composite resin had lower surface hardness than the micro-hybrid composite resin after immersion in the same type of solution. This is presumably due to differences in the particle size of the filler in the composite resin. The nanohybrid composite resin has a smaller filler particle size than the micro-hybrid.<sup>1</sup> The smaller filler particles have a larger surface area that allows increased fluid accumulation around the surface between the filler particles and the resin matrix, thereby increasing water absorption.<sup>1,5</sup> Absorption of water causes matrix degradation that affecting the surface hardness of the composite resin. In line with this study, Yudhit et al. also



stated that composite resin with larger filler particles has a lower water absorption value than composite resin with smaller filler particles.<sup>22</sup>

The micro-hybrid composite resin used in this study contained filler particles as much as 60% by volume, while the nanohybrid composite resin contained filler particles as much as 68% by volume. The higher the filler particle content, the lower the water absorption.<sup>27</sup> However, the resin matrix contained in the composite resin can also affect water absorption. The micro-hybrid composite resin has monomers such as Bis-GMA, UDMA, and Bis-EMA, while the nanohybrid type contains Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA. The resin matrix is hydrophilic. Based on its hydrophilic properties, TEGDMA is the most hydrophilic followed by Bis-GMA and UDMA. TEGDMA has a very dense polymer form but the network is heterogeneous that causes spaces to form between polymer groups and facilitates water absorption.<sup>28</sup> This may explain the water absorption in nanohybrid composite resins to be higher than micro hybrids.

### Conclusion

Based on the results, it can be concluded that cuko pempek can reduce the surface hardness of composite resin and nanohybrid micro-hybrid. The surface hardness of the nanohybrid composite resin was lower than the micro-hybrid composite resin after being immersed in both cuko pempek with vinegar and tamarind.

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