Compressive strength of nanohybrid composite resin after the application of glycerin immersed in fermented milk

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ABSTRACT

Background: The prevalence of dental caries in Indonesia in 2018 is 45.3% of the population. Tooth filling is done so that dental caries does not spread further. The restorative material that is commonly used is composite resin. The compressive strength of composite resin is affected by the polymerization process. The surface of the composite resin exposed to air causes the formation of oxygen inhibited layer (OIL). Glycerin acts as a surface coating that can inhibit contact between the composite resin and air so that polymerization can run optimally. Changes in the mechanical properties of composite resins are also influenced by salivary pH and food intake. The acid contained in fermented milk can affect the matrix and filler bonds in composite resins. Further research is needed to determine the compressive strength of nanohybrid composite resins after the application of glycerin in fermented milk immersion. Purpose: To find out whether there is a change in the compressive strength of nanohybrid composite resin after the application of glycerin immersed in fermented milk, also to find out whether the glycerin application and immersion in fermented milk play a role in causing changes of the compressive strength. Methods: Laboratory experimental research with a total sample of 28 which was divided into 4 groups. Composite resin samples were made with a thickness of 3 mm and a diameter of 5 mm, followed by measuring the compressive strength using the Universal Testing Machine. Results: Independent T-Test test showed significantly different results. The compressive strength value of the group with glycerin application was higher than without glycerin application. Meanwhile, the group immersed in fermented milk drinks had lower compressive strength compared to saline immersion. Conclusion: The compressive strength of the nanohybrid composite resin changed after the application of glycerin immersed in fermented milk.

Keywords: Glycerin, Compressive Strength, Nanohybrid Composite Resin, Immersion, Fermented Milk.

INTRODUCTION

Dental health is an important indicator that can improve the quality of human life. Healthy teeth will produce a good stomatognathic system so that the processes of speaking, chewing and swallowing can function normally.¹ Currently, dental caries is the main dental problem experienced by the Indonesian population. The results of basic health research in 2018 stated that 45.3% of people in Indonesia experienced dental caries.² If not treated immediately, dental caries can spread quickly.³ Tooth filling is the initial action that can be taken so that dental caries does not spread further.⁴ The restorative material that is widely used in various dental treatments is composite resin. Fillings with composite materials will produce a color similar to the tooth structure.⁴ Composite resin has mechanical properties that can support how long the material can last in the oral cavity, one of which is compressive strength. Poor compressive strength can lead to restoration failure.¹ Along with the times, composite resins have changed based on the size of the filler particles. The latest advancement in composite technology is the use of nanotechnology.⁶ Two types of composite resins containing nanoscale particles, namely nanohybrid and nanofiller composite resins. The difference between the two lies in the particle size. Nanohybrid composite resins contain particles of 0.4-5 microns in size and contain nano-sized particles and conventional filler particles, while nanofiller composite resins are the result of the use of nanotechnology in the development of fillers containing 1-100 nm particles throughout the resin matrix.⁶,⁷
The nanohybrid composite resins can be used for both anterior and posterior restorations. Components contained in nanohybrid composite resins can improve aesthetics, physical, chemical, and mechanical properties, such as better compressive strength and tensile strength, wear resistance, and low polymerization shrinkage.

Compressive strength is affected by the polymerization process. Polymerization involves a chain reaction induced by free radicals. If the surface of the composite resin is exposed to air during the polymerization process, there will be greater reactivity between oxygen and radicals than monomer and radicals. This causes the formation of oxygen inhibited layer (OIL). This layer can interfere with the polymerization process and reduce the quality of the composite resin.

The formation of OIL can be minimized by applying glycerin to the surface of the composite resin. In addition to its easy application procedure, glycerin can inhibit the contact between the composite resin and air so that polymerization can run optimally. Good polymerization can increase the hardness, wear resistance, and compressive strength of composite resins.

Changes in the mechanical properties of composite resin are also affected by conditions in the oral cavity, such as salivary pH and food intake. Fermented milk is a dairy product which involves a bacterial fermentation process. Based on the attachment to the regulations of the 2018 Food and Drug Supervisory Agency of the Republic of Indonesia regarding food consumption figures, it was found that the consumption rate of fermented milk is 155 grams per person per day. This figure is the largest among other dairy products and their analogues.

Lactic acid bacteria (LAB) is a microbe used in the manufacture of fermented milk. However, lactic acid which is formed from the results of LAB metabolism can cause a decrease in the pH of fermented milk. If more lactic acid is produced, the decrease in pH will also be greater so that it will be directly proportional to the level of acidity of the product.

Consumption of fermented milk which is acidic exceeds the normal daily intake and in the long term can affect the restorative material. H\(^+\) ions originating from the low pH of fermented milk will react with the methacrylate group. This can affect the matrix and filler bonds in the composite resin and have an impact on decreasing mechanical properties due to accelerated degradation of the matrix.

Based on the above background, the researcher is interested in conducting research which aims to determine the compressive strength of nanohybrid composite resins after the application of glycerin in fermented milk immersion.

**MATERIALS AND METHODS**

This type of research is laboratory experimental research. This research was conducted using Post Test Only Control Group Design. The samples used in this research were nanohybrid composite resin (Filtek™ Z250XT 3M ESPE, USA) which were placed into a disc mold (acrylic plate) with a thickness of 3 mm and a diameter of 5 mm which would form a tablet-like shape. In this research, 7 samples were used per group with a total of 4 groups so that 28 samples were needed.

The distribution of sample groups as follows: Group P1: nanohybrid composite resin without glycerin application in saline immersion (control group). Group P2: nanohybrid composite resin with glycerin application in saline immersion (control group). Group P3: nanohybrid composite resin without glycerin application in fermented milk immersion. Group P4: nanohybrid composite resin with glycerin application in fermented milk immersion.

The composite resin is placed on the disc mold and covered by a glass over the mold. The composite resin was irradiated perpendicularly using light cure with a distance of 1 mm with the provisions of the working procedures for each treatment. Each group of composite resin samples was labeled according to the group and immersed in acrylic container with saline solution. Then, stored in an incubator at 37°C for 24 hours.

Fermented milk used in this research was yogurt with a pH of 3.5 - 4.5. After that, the yogurt is placed in acrylic container. Samples were immersed either in saline or fermented milk according to the distribution of sample groups. The acrylic container filled with the sample and the immersion were placed in an incubator at 37°C for 20 hours.

Compressive strength was measured using Universal Testing Machine, by pressing the sample until the sample fractures. After that, the value shown on the monitor is calculated into the compressive strength measurement formula:

\[
CS = \frac{F \times 9.80}{\pi r^2}
\]

Statistical data testing was carried out using SPSS and data would be analyzed using the Shapiro-Wilk test. After that, an Independent T-Test was conducted to find out the relationships between groups.

**RESULTS**

The results of the compressive strength characteristics of nanohybrid composite resins can be seen in Table 1. Descriptively, the nanohybrid composite resin group with glycerin application had a higher average compressive strength value than the nanohybrid composite resin group without glycerin application, both in saline immersion and in fermented milk. After that, statistical tests were carried out to see whether there were significant differences between each group.

Table 2 shows the results of the normality test for the compressive strength of the nanohybrid composite resin in each group. Based on these results, it was found that all the groups had normally distributed data (p > 0.05) so it could be concluded that the research data fulfilled the requirements for using the Independent T-Test.
Based on Table 3, it is known that the nanohybrid composite resin without glycerin application in saline immersion has an average compressive strength of 203.14 + 19.13 MPa, while with the application of glycerin it has an average compressive strength of 354.84 + 44.19 MPa. On the results of the Independent T-Test with a significant level of 5%, the value of $p = 0.000 < 0.05$ was obtained, so it was concluded that there was a significant difference between the average compressive strength of the nanohybrid composite resin without and with the application of glycerin in saline immersion.

Meanwhile, based on Table 3 it is known that the nanohybrid composite resin without glycerin application in fermented milk immersion has an average compressive strength of 130.36 + 38.78 MPa, while with the application of glycerin it has an average compressive strength of 254.20 + 18.69 MPa. On the results of the Independent T-Test test with a significant level of 5%, the value of $p = 0.000 < 0.05$ was obtained, so it was concluded that there was a significant difference between the average compressive strength of the nanohybrid composite resin without and with the application of glycerin in fermented milk immersion.

Table 1. Compressive strength characteristics of nanohybrid composite resins

<table>
<thead>
<tr>
<th>Groups</th>
<th>Application</th>
<th>n</th>
<th>Min</th>
<th>Max</th>
<th>Mean ± SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersed in Saline</td>
<td>Without Glycerin Application</td>
<td>7</td>
<td>187.20</td>
<td>233.90</td>
<td>203.14 ± 19.13</td>
</tr>
<tr>
<td></td>
<td>With Glycerin Application</td>
<td>7</td>
<td>278.50</td>
<td>407.00</td>
<td>354.84 ± 44.19</td>
</tr>
<tr>
<td>Immersed in Fermented Milk</td>
<td>Without Glycerin Application</td>
<td>7</td>
<td>67.50</td>
<td>177.50</td>
<td>130.36 ± 38.78</td>
</tr>
<tr>
<td></td>
<td>With Glycerin Application</td>
<td>7</td>
<td>234.10</td>
<td>284.60</td>
<td>254.20 ± 18.69</td>
</tr>
</tbody>
</table>

Table 2. Normality test for the compressive strength of nanohybrid composite resins

<table>
<thead>
<tr>
<th>Groups</th>
<th>Application</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersed in Saline</td>
<td>Without Glycerin Application</td>
<td>0.065</td>
</tr>
<tr>
<td></td>
<td>With Glycerin Application</td>
<td>0.499</td>
</tr>
<tr>
<td>Immersed in Fermented Milk</td>
<td>Without Glycerin Application</td>
<td>0.783</td>
</tr>
<tr>
<td></td>
<td>With Glycerin Application</td>
<td>0.558</td>
</tr>
</tbody>
</table>

$p > 0.05$ (Normal Distribution).

Table 3. Results of independent t-test differences in compressive strength of nanohybrid composite resins based on glycerin application

<table>
<thead>
<tr>
<th>Immersion</th>
<th>Mean ± SD Without Glycerin Application</th>
<th>Mean ± SD With Glycerin Application</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saline</td>
<td>203.14 ± 19.13</td>
<td>354.84 ± 44.19</td>
<td>0.000*</td>
</tr>
<tr>
<td>Fermented Milk</td>
<td>130.36 ± 38.78</td>
<td>254.20 ± 18.69</td>
<td>0.000*</td>
</tr>
</tbody>
</table>

(*) = Significantly different at the 5% significance level ($p < 0.05$).

Table 4. Independent t-test results on differences in compressive strength of nanohybrid composite resins based on the type of immersion

<table>
<thead>
<tr>
<th>Application</th>
<th>Mean ± SD Saline</th>
<th>Mean ± SD Fermented Milk</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Glycerin Application</td>
<td>203.14 ± 19.13</td>
<td>130.36 ± 38.78</td>
<td>0.001*</td>
</tr>
<tr>
<td>With Glycerin Application</td>
<td>354.84 ± 44.19</td>
<td>254.20 ± 18.69</td>
<td>0.000*</td>
</tr>
</tbody>
</table>

(*) = Significantly different at the 5% significance level ($p < 0.05$).

Figure 1. Bar chart “average compressive strength of nanohybrid composite resin”.

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Furthermore, the results of the Independent T-Test based on the type of immersion can be seen in Table 4. Based on Table 4, it is known that nanohybrid composite resins in saline immersion without glycerin application had an average compressive strength of 203.14 + 19.13 MPa, while immersion of fermented milk without glycerin application had an average of 130.36 + 38.78 MPa. On the results of the Independent T-Test with a significant level of 5%, the value of p = 0.001 < 0.05 was obtained, so it was concluded that there was a significant difference between the average compressive strength of nanohybrid composite resin in saline immersion without glycerin and in fermented milk immersion without glycerin.

Meanwhile, based on Table 4 it is known that the nanohybrid composite resin in saline immersion with glycerin application has an average compressive strength of 354.84 + 44.19 MPa, while immersion of fermented milk with glycerin application has an average compressive strength of 254.20 + 18.69 MPa. On the results of the Independent T-Test with a significant level of 5%, the value of p = 0.000 < 0.05 was obtained, so it was concluded that there was a significant difference between the average compressive strength of nanohybrid composite resin in saline immersion with glycerin and in fermented milk immersion with glycerin.

Thus, the application of glycerin and immersion in fermented milk had a significant effect on the compressive strength of the nanohybrid composite resin. Figure 1 shows the average compressive strength of nanohybrid composite resins in each group.

**DISCUSSION**

The results of the Independent T-Test on differences in the compressive strength of nanohybrid composite resins based on the application of glycerin showed significant results. This shows that the application of glycerin has an effect on the compressive strength of the composite resin. Based on the research, the average value of compressive strength in the group without glycerin application was lower than the group with glycerin application. This occurs due to the formation of OIL during the polymerization process.⁹,¹⁰

The polymerization process involves the formation of polymer molecules through chemical reactions which are characterized by inter-monomer bonding of the composite resin.¹¹ The camphorquinone photoinitiator will trigger polymerization by absorbing blue light from the light cure activator so that free radicals will be formed.⁶,¹⁰ Polymerization at the initiation stage will support the polymer chain to develop by inducing free radicals which will activate monomer molecules so that free radicals can react with other monomers and will produce a chain reaction. In the propagation stage, there is an addition of monomers to free radicals which will be followed by elongation of the polymer chain. This process occurs quickly and the formed free radicals will react with other molecules to produce new free radicals. After that, it is followed by the termination stage, namely chain termination which marks the formation of a stable polymer chain followed by the cessation of free radical formation.⁶,²³

In groups P1 and P3 without glycerin application, the polymerization process will be disrupted when the surface of the composite resin is in contact with air. Oxygen (O₂) is one of the chemical elements contained in air. If the surface of the composite resin is exposed to oxygen, the free radical will come in contact with O₂ to produce a peroxy radical (R • + O₂ = R-OO •). The formation of bonds between free radicals and O₂ causes a decrease in the reaction on the monomer. Conversely, there will be an increase in reactivity between free radicals and O₂ which is greater than free radicals and monomers. This causes the formation of oxygen inhibited layer (OIL). The oxygen inhibited layer is a sticky layer and is classified as a resin-rich uncured layer. The formation of OIL causes polymerization to be less than optimal, which results in a decrease in the mechanical properties of the composite resin.¹¹,²⁴

Meanwhile, in groups P2 and P4, one layer of glycerin (50 μl) was applied using a brush on the surface of the composite resin and the results obtained that the average compressive strength value was higher in the group that applied glycerin compared to the group without glycerin application. Glycerin acts as a surface coating which is applied to the surface of the composite resin prior to irradiation to reduce the formation of OIL. This is because glycerin has stability in an atmospheric oxygen medium which will minimize the occurrence of changes at normal temperatures to glycerin’s bonds with surrounding objects.²⁵ Glycerin will block the contact between free radicals and oxygen (O₂) so that a bond between the two is not formed. That way, free radicals with monomers can bind maximally. Minimized OIL formation will make polymerization run optimally. In this research, glycerin prevented a greater decrease in compressive strength, as evidenced by the higher average compressive strength values of nanohybrid composite resins with the application of glycerin in fermented milk immersion, 254.20 + 18.69 MPa compared to the compressive strength values of nanohybrid composite resin without glycerin application in fermented milk immersion, 130.36 + 38.78 MPa. Glycerin also has a transparent color so it does not affect the light intensity and irradiation distance during the polymerization process.²⁶,²⁷

In addition, the results of the Independent T-Test on differences in the compressive strength of nanohybrid composite resins based on immersion showed significant results. This shows that immersion also affects the value of the compressive strength of the composite resin. Based on the results of the research, the average compressive strength in the group in fermented milk immersion was lower than in the saline immersion group. This occurs due to water absorption associated with food and beverage intake.¹³

Water absorption caused by daily food and drink intake will affect the mechanical properties of the composite
resin. Water absorption involves the loss of the chemical structure in the composite resin, bis-GMA. This is caused by hydrolysis and environmental conditions that are water-related environmental.\textsuperscript{28} Yogurt is a dairy product that is fermented in lactic acid bacteria. The formation of lactic acid in yogurt plays a role in increasing acidity and decreasing the pH value\textsuperscript{28}. In this study, yogurt was used with a pH of 3.66.

In the composite resin group immersed in fermented milk (yogurt), the H\textsuperscript{+} ions contained in the acidic nature of the yogurt will react with the dimethacrylate monomer at the end of the composite resin matrix which will cause instability in the chemical bonds of the double chain, namely the double bond carbon group (C=C) on the polymer matrix resin. It has the potential to dissolve the composite resin so that it has an impact on the degradation of its components, marked by changes in the microstructure of the composite resin which will form pores and remove residual monomers which will cause a decrease in mechanical properties, namely compressive strength.\textsuperscript{13,28}

In this study, it was concluded that there was a change in the compressive strength of the nanohybrid composite resin after the application of glycerin in fermented milk immersion. In this case, the application of glycerin and the immersion in fermented milk played a role in causing a change in the compressive strength value of the composite resin.

Nanohybrid composite resin with glycerin application has a higher compressive strength than without glycerin application. In addition, nanohybrid composite resin immersed in fermented milk will experience a decrease in compressive strength compared to saline immersion.

REFERENCES

