

### The different effects of preheating and heat treatment on the surface microhardness of nanohybrid resin composite

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#### ABSTRACT

**Background:** A composite resin is used as restorative material in dentistry because it has the same colour as dental enamel, is easy to use in an oral cavity and offers good biocompatibility. Based on the size of filler, composite resin is divided into types, one of which is a composite resin nanohybrid. An important mechanical property of restorative material is microhardness. The mechanical properties of restorative material is highly affected by both polymerisation and heating process. There are many methods to improve composite resin's microhardness, including preheating and heat treatment. **Purpose:** The purpose of this study was to evaluate different effects of preheating and heat treatment on the microhardness of nanohybrid composite resin. **Methods:** This study is an experimental laboratory research with post-test-only group design. Samples were divided into six groups: preheating groups at temperatures of 37°C and 60°C, heat treatment groups at temperatures of 120°C and 170°C, a negative control group and a positive control group. Afterwards, the resulting data were analysed using one-way ANOVA. **Results:** The result based on the one-way ANOVA test indicated that there was a difference in microhardness in each group with a significance of 0.000 ( $p < 0.005$ ) between preheating and heat treatment. **Conclusion:** The conclusion of this study was the best microhardness of composite resin nanohybrid is the heat treatment group at temperature 170°C.

**Keywords:** heat treatment; microhardness; nanohybrid resin composite; preheating

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#### INTRODUCTION

Nanohybrid composite resin is a composite resin with nano-sized particles of 0.005-0.020 µm in a resin matrix.<sup>1</sup> The advantages of nanohybrid composite resin includes good colour stability, good surface finish and less cure shrinkage.<sup>2</sup> Nanohybrid composite resins have additional components in the resin matrix, namely nanoparticles and nanocluster.<sup>3</sup> This combination can reduce particle interstitial distance, thereby increasing filler resistance, physical properties, mechanical properties and retention.<sup>4,5</sup> Nanohybrid composite resin also offers mechanical properties such as high compression strength, good surface hardness, resistance to fracture, abrasion resistance and high diametral tensile strength.<sup>4,6</sup>

The mechanical properties of composite resins are influenced by polymerisation, heat treatment and a mixture

of both.<sup>7</sup> The polymerisation process determines the number of changes in the double bond of the monomer to a single bond of polymer called the degree of conversion.<sup>8</sup> Ideally, the dental restorative resin would have all of its monomer converted to polymer, but, in reality, polymerised composite resins only have a 55–75% degree of conversion.<sup>9</sup> Composites with insufficient hardness will be easily cracked.<sup>10</sup>

The method used to improve the mechanical properties of composite resins is preheating and heat treatment. Preheating is a method of heating composite resin using a composite warmer or an oven at a certain temperature before curing.<sup>11</sup> Preheating can reduce the viscosity of composite resins, thus reducing microleakage and increasing adaptation to the edge of the lift.<sup>12</sup> The method of preheating has an effect on increasing the degree of conversion.<sup>13</sup> According to some studies, radical and

monomer mobility will increase after preheating,<sup>11</sup> affecting the degree of polymerisation, which can increase polymer crosslink.<sup>14</sup>

Heat treatment is secondary curing in the laboratory, which aims to improve the mechanical and physical properties of a material.<sup>15</sup> Some tools that can be used for heat treatment include conventional ovens, autoclaves and porcelain furnaces.<sup>16</sup> The results of heat treatment can increase the degree of conversion and the mechanical properties of composite resins, as well as minimise polymerisation shrinkage.<sup>13</sup> Studies comparing preheating and precooling at certain temperatures have indicated the flexural strength and flexural modulus of composite resins. The results of this study demonstrate that preheating at 45°C using a composite warmer for 15 minutes can increase the flexural strength and flexural modulus of nanohybrid composite resins.<sup>13</sup>

Other studies have suggested that preheating at 37°C and 60°C can reduce shrinkage during polymerisation and increase the surface hardness of a nanohybrid composite resin.<sup>17</sup> Preheating composite resins at 37°C and 54°C using an oven for 30 minutes can increase the resin’s hardness.<sup>11</sup> Research into nanohybrid composite resins with heat treatment at 170°C has indicated an increase in the surface hardness of composite resins and flexural strength. Other studies have shown that a heat treatment temperature of 170°C for 10 minutes for composite resins can reduce water absorption and solubility, enabling the composite resins to become more flexible.<sup>15</sup> Heat treatment with a temperature of 120°C for 10 minutes on composite resins can increase the flexural strength and hardness of composite resins.<sup>19</sup>

To date, there is no research comparing treatments between preheating and heat treatment on the microhardness of nanohybrid composite resins. Hence, the purpose of this study was to evaluate the different effects of preheating and heat treatment on the microhardness of a nanohybrid composite resin. The temperatures used for preheating in this study were 37°C and 60°C. The temperatures used for heat treatment in this study were 120°C and 170°C.

## MATERIALS AND METHODS

This study is an experimental laboratory research with post-test-only group design. According to the American Society

for Testing Materials (ASTM) E384 standard, a cylindrical specimen was used with a diameter of 5 mm and a thickness of 2 mm. The total specimens used in the study were 48, divided into six groups such as 1A nanohybrid composite resin (*dentsply*, Indonesia) preheating 37°C, 1B nanohybrid composite resin preheating 60°C, 2A nanohybrid composite resin heat treatment 120°C, 2B nanohybrid composite resin heat treatment 170°C, 3A negative control with resin nanohybrid composite resin and 3B positive control with a laboratory composite resin as a microhybrid composite resin.

Preheating groups were treated for 30 minutes in the oven before curing; they were then cured for 20 seconds and put into an incubator for 48 hours at 37°C. The heat treatment group was cured for 20 seconds first; thereafter, the heat treatment group was put into the porcelain furnace for 10 minutes and incubated at 37°C for 48 hours. The negative control group was put into an incubator at 37°C for 48 hours after being lightly cured. The positive control group was incubated at 37°C for 48 hours and then put into a porcelain furnace for 10 minutes at 170°C.

The microhardness composite resin test was carried out using a Vickers microhardness test with a load of 100gF in 15 seconds. The unit value of the surface hardness of the composite resin was Kg/mm<sup>2</sup>. The research data were tested for normality using the Shapiro-Wilk test because the number of samples in the study was fewer than 50, and homogeneity tests were carried out using the Levene test. The researcher used the one-way ANOVA test to compare each preheating and heat treatment group. Furthermore, the data were tested additionally: namely, the post hoc test used the LSD test to determine the value of comparisons between groups.

**Table 1.** One-way ANOVA test results: preheating and heat treatment of microhardness of nanohybrid composite resins

Groups	N	δR <sub>q</sub>		Sig
		Mean	SB	
1A	8	22.73	3.11	
1B	8	49.98	4.69	
2A	8	28.43	2.66	0.000*
2B	8	51.75	4.54	
3A	8	19.95	2.40	

**Table 2.** Post hoc preheating and heat treatment test results for microhardness of nanohybrid composite resins using the LSD methods

Groups	1A	1B	2A	2B	3A	3B
1A		0.000*	0.005*	0.000*	0.183	0.000*
1B			0.000*	0.376	0.000*	0.011*
2A				0.000*	0.000*	0.000*
2B					0.000*	0.001*
3A						0.000*
3B						

## RESULTS

The results of the nanohybrid composite resin microhardness test via the Vickers microhardness tester can be seen in Table 1. Per the one-way ANOVA test results, there were significant differences in microhardness of the nanohybrid composite resin ( $p < 0.05$ ). Next, regarding the post hoc test using the LSD test, the test summary can be seen in Table 2. The LSD test results showed significant differences between several groups, such as group 1A with groups 1B, 2A, 2B and 3B ( $p < 0.05$ ). In contrast, group 1B with group 2B and group 1A with 3A showed no significant difference ( $p > 0.05$ ).

## DISCUSSION

Heat treatment can reduce the amount of residual monomer in the composite resin which causes an increased degree conversion.<sup>18</sup> The heat treatment process will remove some carbon double-bond monomers that cannot react by evaporation because heat and some other carbon double monomers will be covalently bonded to the polymer network.<sup>20</sup> The temperature used in the heat treatment affects the increasing degree of conversion and the large amount of residual monomer present in the composite resin.<sup>15</sup>

Heat treatment at a temperature of 170°C is closer to the standard glass transition temperature ( $T_g$ ), so it is very effective in homogenising and modifying the polymer structure, increasing the number of cross bonds and condensing the polymer to become stronger.<sup>15</sup> Temperatures closer to the glass transition temperature ( $T_g$ ) can reduce stress during polymerisation when free radicals still trapped in the molecule will react again to form more cross bonds. Such polymer crosslinking has small groups of atoms on both sides. When the sides of the polymer are close to electrons, they will form covalent bonds that will join. This polymer crosslinking will improve the mechanical properties of the composite resin better.<sup>21</sup> Irregular filler particles can cause shrinkage during polymerisation, causing a decrease in the mechanical properties of composite resins.<sup>22</sup> The heat treatment can homogenise the filler particles, reducing the stress of shrinkage during polymerisation. High temperatures in the heat treatment process can increase radical mobility and the degree of polymerisation, thereby affecting crosslink density and producing dimethacrylate monomers.<sup>23</sup>

Preheating can change high viscosity to low by melting viscosity due to the vitrification of the polymer. Polymer vitrification is a polymer melting process. Vitrification of the polymer occurs because the time required for the rate of diffusion reaction is reduced by the formation of the polymer. The reduced speed of the polymerisation process aims to determine the final result of the degree of conversion.<sup>24</sup> As a result of vitrification, free radicals are trapped in the polymer, but this will not stop the mobility

of free radicals because some free radicals are still present in the polymer.

Due to increased system mobility caused by temperature, free radicals will still react with the remaining double bonds and continue to polymerise. In addition to the free radicals trapped in the polymer due to vitrification, other molecules, such as residual monomers and photoinitiators, are also trapped inside the polymer so that they can influence biological properties such as the surface roughness of composite resins and mechanical properties such as surface hardness, flexural strength and compression strength of composite resins.<sup>25</sup> Polymer vitrification will stop when the preheating temperature used has reached the standard glass transition temperature of the polymerisation. The reaction rate will decrease significantly after vitrification, and any subsequent reaction will be slower. This process will determine the end result of polymerisation.<sup>26</sup>

Preheating can increase the mobility of polymer chain molecules, delay diffusion reactions and increase the degree of conversion.<sup>11</sup> The number of monomers will decrease with the increase in preheating temperature.<sup>27</sup> Increasing the temperature during preheating can increase the degree of conversion. This is because the polymerisation process of composite resins involves free radicals that convert material from high viscosity to low viscosity. During this process, a change in the C = C double bond becomes a covalent C-C single bond between the methacrylate monomers and changes in the rate of diffusion of free radicals.<sup>22</sup>

An increase in preheating temperature can increase the amount of monomer dimethacrylate, but only to a certain temperature limit. After the preheating temperature limit is reached, the monomer conversion decreases despite the subsequent temperature rise.<sup>28</sup> The effective temperature for changing the viscosity of composite resins is between 54°C and 68°C.<sup>29</sup> This is due to reactant evaporation and photoinitiator degradation.

In addition, there are other influences, such as the time lag between removing the composite resin from the syringe into the mould and smoothing the composite resin before curing.<sup>27</sup> The temperature received by the composite resin was not achieved as expected because the temperature could drop 2–4°C from the temperature it was supposed to be acquired.<sup>30</sup> After preheating, the time needed to irradiate the composite resin is five minutes, so that it would increase 52–64% degree of conversion. This research did not observe the time needed to place the composite resin after preheating until printing.<sup>31</sup>

Several factors can cause differences in the surface hardness of composite resins, including the type of composite resin. The positive control group used laboratory composite resins, namely the microhybrid composite resin, and the negative control group used nanohybrid composite resins. Differences in the organic matrix composites of resins such as monomers and photoinitiators can affect the microhardness of composite resins.<sup>18</sup> Another influence is that nanohybrid composite resins have additional components in the form of nanoparticles and nanocluster in

the matrix resin. The combination of these two ingredients can reduce the interstitial distance of particles, thus increasing filler loading, offering better physical properties and increasing retention.<sup>7</sup> In conclusion, this study has determined that the microhardness of the nanohybrid composite resin heat treatment group is higher than the preheating treatment group.

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