

The Effect of Addition of Blood Cockles (*Anadara granosa*) Shell Nano-hydroxyapatite on Hardness of Heat Cured Acrylic Resin

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Abstract: Blood cockles shell (*Anadara granosa*) nano-hydroxyapatite (NHA) powder is a bioceramic that can increase the mechanical strength of heat-cured acrylic resin. This study aims to determine the effect of adding NHA powder from blood clam shells on heat-cured acrylic resin's hardness. This type of research used laboratory experimental, research design posttest-only control group design. Twenty-four heat-cured acrylic resin samples were used and divided into three groups: the acrylic resin group with 1%, 3% blood cockles shell NHA, and acrylic resin without NHA with simple random sampling. TEM characterization results showed that NHA had a spherical shape and tended to experience agglomeration. TEM characterization with magnifications ranging from 5,000x to 150,000x and particle sizes ranging from 20 nm to 200 nm. The highest mean value of hardness was in group III at 27.7375 VHN. One-Way ANOVA test on hardness showed a significant difference between groups with $p = 0.000$ ($p < 0.05$). This study concludes that adding NHA could increase the hardness of heat-cured acrylic resin.

1 INTRODUCTION

Heat-cured acrylic resin or polymethylmethacrylate (PMMA) is the most commonly used denture base construction material since 1930. This material is not ideal in every respect, and it is the combination of various rather than one desirable property that accounts for its popularity and usage (Hameed & Rahman, 2015).

The heat-cured acrylic resin as denture base material has some benefits such as its ease of processing, low cost, lightweight, excellent aesthetic properties, low water sorption, solubility, and ability repaired quickly. However, low thermal conductivity, insufficient mechanical strength, brittleness, high coefficient of thermal expansion, and relatively low modulus of elasticity make it more prone to failure during the clinical service (Alla et al., 2015). The drawback of heat-cured acrylic resin is monomer residue, which can reduce its mechanical properties,

making it easier to fracture (Kenneth J Anusavice, C Shen, 2013)

One of the mechanical properties of heat-cured acrylic resin is hardness. Hardness is a mechanical property used to describe the resistance of a material to a load. Surface hardness is closely related to abrasion, erosion, and attrition of the restorative material in the oral cavity (Kenneth J Anusavice, C Shen, 2013). Not adequate hardness can cause the acrylic resin not to be able to withstand the pressure produced during mastication so that it can crack and break. Overcome these problems; several attempts were made to modify and improve the strength, thermal properties, and hardness of the heat-cured acrylic resin (Hameed & Rahman, 2015).

The high use of acrylic resin in dentistry has led to various attempts to improve these properties, one of which is a reinforcing agent in the form of hydroxyapatite. One of the potential sources of hydroxyapatite due to its high calcium carbonate content and high density is the blood cockles or

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Anadara granosa (*A granosa*) shell, which is commonly consumed by the public. The mineral composition of blood cockles, such as calcium carbonate and carbon, is more than 98.7% of the total mineral content. The content of Mg²⁺, Na⁺, P³⁻, K⁺, Fe²⁺, Cu⁺, Ni²⁺, consists of about 1.3% (Awang et al., 2007).

Hydroxyapatite can be made in various forms, one of which is nanoparticles (Mollazadeh et al., 2007). Hydroxyapatite can be made in various forms, one of which is nanoparticles. Nanoparticles has large surface-to-volume ratio (specific surface area), which improves mechanical properties (Okada & Matsumoto, 2015). Research on the effect of adding 1% and 3% of blood cockles shell nano-hydroxyapatite to the hardness of heat-cured acrylic resin has not been conducted. Therefore, researchers are interested in conducting this research.

2 MATERIAL AND METHODS

This research is an experimental laboratory study with a post-test only control group design. Twenty-four heat-cured acrylic resin samples were used and divided into three groups: the acrylic resin group with 1%, 3% blood cockles shell nano-hydroxyapatite (NHA), and acrylic resin without NHA *with simple random sampling*. Ethical eligibility permit from the Ethics Commission for Medical and Health Research, Faculty of Medicine, Jenderal Soedirman University with registration number 316 / KEPK / VII / 2019. The research sample was in the form of discs measuring 10 mm in diameter and 2 mm thick. The polymerized acrylic resin cured using a water bath at 70°C for 90 minutes, then continued to 100°C for 30 minutes and allowed to stand at room temperature. Furthermore, sample finishing uses a tungsten carbide bur at a speed of 15,000 rpm for 60 seconds for each sample.

The Vickers Hardness test was performed on all samples. The strength was applied to the midpoint of base materials by a diamond tip. Then, diagonals of trace whose shape is square measured by microscope and evaluated their hardness values. The statistical analysis of obtained data was performed with a one-way ANOVA analysis of variance test.

3 RESULTS

The Transmission Electron Microscope (TEM) test could determine the shape and size of NHA. Figure 1 below is a picture of the results of TEM

characterization with weak and strong magnification. The results of TEM characterization showed that NHA (*A granosa*) had a spherical shape and tended to experience agglomeration. TEM characterization with magnifications ranging from 5,000x to 150,000x and particle sizes ranging from 20 nm to 200 nm.

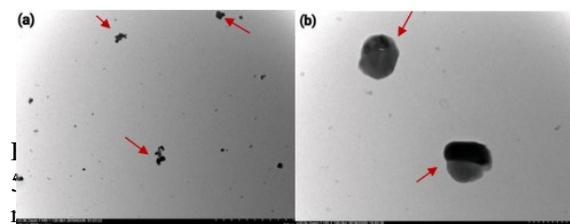


Figure 1.

Hardness testing uses the Vickers microhardness test. The test was carried out on 3 sample groups with a cylindrical sample with a diameter of 10 mm and a thickness of 2 mm. The mean and standard deviation of the test results showed in Table 1.

Table 1. Mean and standard deviation of Vickers hardness number for heat-cured.

| No | Sample groups | N | Mean | ±SD |
|----|---------------|---|---------|---------|
| 1 | Without NHA | 8 | 22.6538 | 0.39192 |
| 2 | 1% NHA | 8 | 26.6950 | 0.83816 |
| 3 | 3% NHA | 8 | 27.7375 | 0.57918 |

Based on Table 1, the highest mean value of hardness was in group III at 27.7375 VHN, while the lowest hardness value was in a group I at 22.6538 VHN. The Shapiro-Wilk test and Levene test showed that the data were normal and homogeneous. Furthermore, the One-Way ANOVA test showed a significant difference in the sample group hardness value. The test results showed in Table 2. The One-Way ANOVA test results show a p-value of 0.000 (p < 0.05). There is a significant difference in the value of hardness between the sample groups. The results of the post hoc LSD follow-up test showed in Table 3.

Table 2. The One-Way ANOVA analysis

| V H N | | Sum of Squares | df | Mean Square | F | Sig. |
|-------------|----------------|----------------|----|-------------|---------|------|
| | Between Groups | 115.368 | 2 | 57.684 | 145.231 | 0.00 |
| | Within Groups | 8.341 | 21 | 0.397 | | |
| Total | 123.709 | 23 | | | | |

Table 3. Results of post hoc LSD hardness test of Heat-Cured Acrylic Resin

| Sample groups | Without NHA | 1% NHA | 3% NHA |
|---------------|-------------|--------|--------|
| Without NHA | | 0,000* | 0,000* |
| 1% NHA | | | 0,003* |
| 3% NHA | | | |

The LSD test results in Table 3 show a significant difference ($p \leq 0.01$) in all treatment groups with the control group. Besides, there was a significant difference between groups II and III.

4 DISCUSSION

TEM characterization determined the size and shape of the particles. Figure 5.2 shows an overview of NHA particles. A weak magnification of 5,000x indicates a spherical particle with a size of about 20 nm. A strong magnification of 30,000x shows a clearer picture of the particle shape, a 163 nm spherical shape. The morphological description of hydroxyapatite particles is indeed round and tends to be irregular. NHA also tends to experience agglomeration, namely the fusion of several hydroxyapatite nanoparticles.

The agglomeration of the hydroxyapatite particles occurs during the synthesis process of making hydroxyapatite. Agglomeration of nano-sized particles usually occurs because nanoparticles have a high surface area and a more significant number of particles, so they tend not to be quickly stabilized, so they easily clump. Agglomeration also occurs due to the lack of stirring speed during the synthesis of hydroxyapatite. The description of NHA particles as per the research conducted by (Dong et al., 2009) that the particle morphology formed from hydroxyapatite is an irregular sphere, but this study only reports at a magnification of 5,000x.

The results showed that the addition of NHA particles with a concentration of 1% and 3% could increase the hardness of heat-cured acrylic resin. The most significant increase in hardness occurred in the group with the addition of 3% nano hydroxyapatite. The hardness of heat-cured acrylic resin was influenced by several factors, one of which was the particle size. The particle size ranges from 20-200 nm. This result is per the research conducted by Afrizal et al. (2016) that hydroxyapatite with a small and delicate grain size will fill the space between the atoms of heat-cured acrylic resin and inhibits the rate

of dislocation of heat-cured acrylic resin atoms, thereby increasing the density and mechanical properties of heat-cured acrylic resin (Afrizal, 2016). The nano-hydroxyapatite that fills the space between the atoms of the heat-cured acrylic resin is a mechanical bond. The use of nanofillers with a particle size of 10-100 nm can increase mechanical properties such as abrasion resistance, hardness, and flexural strength, so that the use of dentures with a sufficient period long expected to withstand abrasion (Rodrigues et al., 2008).

5 CONCLUSIONS

The addition of blood cockles nano-hydroxyapatite could increase the hardness of heat cured acrylic resin.

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