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ORIGINAL ARTICLE

Evaluation of Transverse Strength and Impact Strength of Heat Cured Acrylic Resin With Addition of Skipjack Tuna Bone Nano-Hydroxyapatite

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ABSTRACT

Introduction: Hydroxyapatite made from skipjack tuna bone can be used as a reinforcing material added to the denture plate base to prevent fracture. The development of knowledge about nanoparticles shows that particles or globules at the nanometer scale have distinctive physical properties, which can improve the quality of various types of media. The purpose of this study was to determine the effect of adding nanohydroxyapatite from skipjack tuna bone on the transverse strength and impact strength of heat cured acrylic resins. **Methods:** There were 3 groups of heat cured acrylic resin samples; the group without the addition of nano hydroxyapatite as a control, the group with the addition of 2% nano hydroxyapatite, and the group with the addition of 5% nano hydroxyapatite. Each group consisted of 9 samples which were then tested for transverse strength with Transverse Testing Instrument and impact strength with Impact Testing Instrument. **Results:** The best transverse strength was in the group with the addition of 2% nano hydroxyapatite of skipjack tuna. Meanwhile, the impact strength after the addition of 2% nano hydroxyapatite did not have a significant difference compared to the control, but the addition of 5% decreased the impact strength. **Conclusion:** Nano hydroxyapatite of skipjack tuna bone has the potential as a reinforcing material from heat cured acrylic when used at a concentration of 2% because at that concentration the transverse strength shows the best performance.

Keywords: Hydroxyapatites; Acrylic resins; Mechanical testing

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INTRODUCTION

Tooth loss is a common problem in society (1). This condition can be caused by widespread dental caries, periodontal disease, trauma, infection, malignancy, or failure of endodontic treatment. Several studies have shown that tooth loss causes several consequences. The main effect of teeth that are not replaced is a reduction in the effectiveness of mastication. Meanwhile, non replaced missing posterior teeth can disrupt the stomatognathic system and cause further effects such as drifting, rotation or supereruption. In addition, tooth loss also affects speech and self-confidence due to changes in appearance. It is also possible that patients are at risk for ischemic stroke especially in patients with periodontal disease and having less than 24 teeth in the mouth compared to those with more than 25 teeth. Therefore, it is necessary to use artificial teeth to replace the missing teeth (2,3).

Acrylic-based removable dentures are still the treatment of choice for replacing missing teeth, mainly because of their lower cost. Removable dentures can replace some part or all of the teeth that have been lost and can be removed by the patient himself from the oral cavity without the help of a dentist (4). In the manufacturing process, acrylic consists of a powder called a polymer and a liquid called a monomer. Besides being biocompatible and good esthetics, denture base materials must also have good mechanical and physical properties. The basic material that is often used is Poly Methyl Methacrylate (PMMA) which is often used to make denture bases because of its various advantages, which is easy and inexpensive processing, stability in the oral environment and good aesthetics. However, PMMA also has weakness, such as lower physical and mechanical properties. The denture base can be fractured if it is dropped or due to compressive forces that occur in the oral cavity. To improve the PMMA properties, it can be done by adding filler to the polymer composition (5,6).

Adding filler as a reinforcing material have been carried out in various studies (7). One of the reinforcing materials that have good biocompatibility is hydroxyapatite. This material also has a calcium phosphate ratio similar to that of natural bone and teeth (8,9). Synthesis of hydroxyapatite can be derived from fishery waste materials, including fish bones which in this study used skipjack tuna (10–12). With the development of knowledge about nanoparticles, research on nano-hydroxyapatite (nHA) has also developed. Previous studies stated that the optimal composition of addition of nHA in PMMA is 2.5% and more than 10% will not give a better effect on the mechanical properties of acrylic (7,13). Based on this review, this study was conducted to determine the effect of adding nano-hydroxyapatite from skipjack tuna on transverse strength and heat cured acrylic strength.

MATERIALS AND METHODS

Research Design

This study used heat cured acrylic resin. The sample for the transverse strength test was in the form of a rod slab 65 mm long, 10 mm wide, and 2.5 mm thick. While the samples for the impact strength test were cylindrical in shape with a diameter of 6 mm and a thickness of 12 mm (14,15). In each test there were 27 samples divided into 3 groups, namely, group K (control) without the addition of nano-hydroxyapatite, group P1 with the addition of 2% nano hydroxyapatite, and the P2 group with the addition of 5% nano hydroxyapatite.

Preparation of skipjack tuna bone nanohydroxyapatite

Synthesis of hydroxyapatite was prepared from the basic ingredients of tuna fish bone (*Katsuwonus pelamis*) by the precipitation method. Fish bones were cleaned and then through a process of drying and soaking in acetone for 3 days. After being evaporated, the bones are baked in an oven to form calcium oxide powder. The powder was then calcined at a temperature of 900^{o}C, sieved and precipitated using phosphoric acid. The solution was then precipitated for 24 hours. The precipitate was filtered and then baked again for 2 hours at 105^{o}C and 5 hours at 900^{o}C to produce hydroxyapatite which is proved by X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) tests. The preparation of nano-hydroxyapatite particle was carried out using a ball mill and sieving and then a particle size test was carried out using Particle Size Analysis (PSA) (16).

XRD Test

XRD test was performed using PANalytical Type x'Pert PRO. This tool will shoot X-rays at the material so that interactions occur between electrons in atoms. X-ray diffraction due to atoms will produce a pattern depicted in a graph which is then matched with a standard chart of the Joint Committee on Powder Diffraction Standard (JCPDS) database (17).

FTIR Test

FTIR test with Shimadzu Type IR Prestige 21. Fourier Transform InfraRed (FTIR) is a technique used to determine the functional groups of compounds that make up a material. FTIR can detect the energy absorbed by each functional group. The apparatus will generate light by heating a radiation source with electricity to a temperature of 1500°K and 2000°K to emit an interferogram beam. The light is then absorbed by the sample and captured by the detector and then sent to a computer for further interpretation (18).

Measurement of Particle Size Analysis (PSA)

PSA test was carried out to analyze the particle size of nano-hydroxyapatite. Hydroxyapatite was weighed as much as 0.02 grams and then dissolved in 100 ml of aquabides. The mixed solution was then stirred using a magnetic stirrer for about 180 minutes at 30^{o}C until the solution was dispersed with aquabides media. Samples of the solution were taken and then put into a tube and the PSA (Particle Sizing Analyze) test process was carried out using a Microtrac Type Nanowave II tool (19).

Measurement of transverse strength

The transverse strength test was carried out on a heat cured acrylic plate of 65mm x 10 mm x 2.5mm (ADA, no. 12). The acrylic plate is placed on the Transverse Testing Instrument and then given a load until the acrylic fractures. The value shown by the monitor is then calculated using the Transverse Strength formula with the result in N/mm2 (20).

Measurement of Impact strength

Impact strength test was performed on heat cured acrylic cylindrical shape with a diameter of 6 mm and a length of 12 mm (ADA, no 17). The acrylic rod is placed on the Impact Testing Instrument and then given a pendulum or weight so that it hits the acrylic plate. The swing angle is measured. The weight of the pendulum, the initial and final angles of the pendulum and the surface area of the acrylic rod are then calculated using the Impact Strength formula with the result in kg/cm^2 (20).

RESULTS

XRD Test Results

XRD characterization test to see the hydroxyapatite phase contained in the synthesized hydroxyapatite powder.

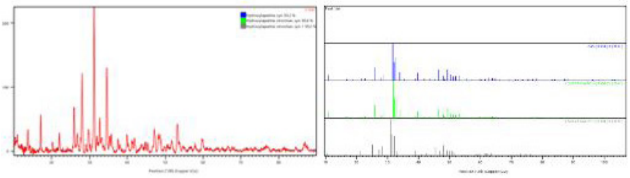

Figure 1 : XRD Test Results.

Based on ICSD and ICDD data, it can be concluded that the hydroxyapatite sample was identified as the hydroxyapatite phase because there were diffraction peaks in the sample containing hydroxyapatite.

FTIR Test Results

FTIR spectroscopic analysis which aims to determine the formation of the hydroxyapatite functional group and also to determine the number of components in a sample which is characterized by the presence of a peak at a certain wave number. The component of hydroxyapatite consists of several functional groups, namely the OH^- group, the $PO4^{3-}$ group and the $CO3^{2-}$ group.

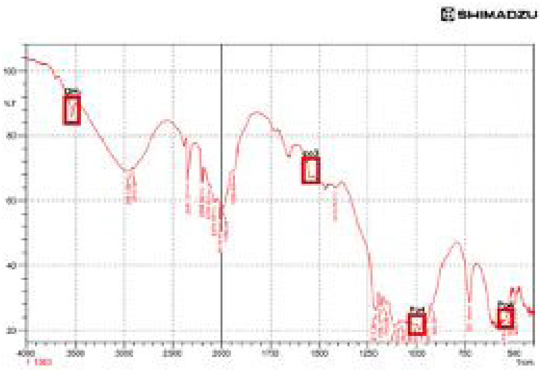

Figure 2 : FTIR Test Results.

Table I : FTIR Test Wave Number

Functional Group	Wave Number (cm^{-1})
OH^-	3500-3650 cm^{-1}
CO_3^{2-}	1500-1600 cm^{-1}
PO_4^{3-}	500-600 cm^{-1}
	1000-1100 cm^{-1}

The results of the FTIR characterization test showed that skipjack tuna bone powder could be proven to contain hydroxyapatite because the phosphate group was identified as having wave numbers between 1000-1100 cm^{-1} and 500-600 cm^{-1}.

PSA Test Results

Particle size analyzer (PSA) test was carried out to determine the size of the hydroxyapatite powder formed. This measurement uses the principle of dynamic light scattering (DLS) with the wet method, which shoots light at a sample that has been dissolved in aquabides media.

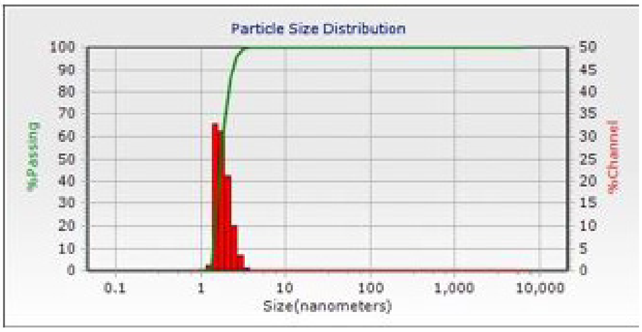

Figure 3 : PSA Test Results.

Based on the results of the PSA test, the average size of the hydroxyapatite flour is 1.73nm .

Furthermore, the results of the average transverse strength test and impact strength are shown in the following table.

Table II : Transversal strength of acrylic plate

Group	n	Mean ± SD
Group K	9	150.91 ± 8.29
Group P1	9	262.16 ± 10.19*
Group P2	9	185.52 ± 9.22*

Table III : Impact strength of acrylic rod

Group	n	Mean ± SD
Group K	9	0.586 ± 0.061
Group P1	9	0.578 ± 0.092
Group P2	9	0.484± 0.074 *

Based on the results of statistical tests, there were significant differences between the groups of the transverse strength test. Meanwhile, in the impact strength test results, there was no significant difference in the no-addition group with the 2% addition group but there was a significant difference in the 5% addition group with the other two groups (sig < 0.05).

DISCUSSION

From XRD test results based on ICSD and ICDD data in Figure 1, it can be concluded that the powder sample was identified as the hydroxyapatite phase because there were diffraction peaks in the sample, which is containing 60% of the hydroxyapatite. This percentage was due to the incomplete synthesis process. Meanwhile, an indication of the formation of a hydroxyapatite group based on the FTIR results in Figure 2 is the formation of a phosphate group complex ($PO4^{3-}$) at a wave of 900-1050 cm^{-1} with a wavelength of 530.43 cm^{-1}. The carbonate group ($CO3^{2-}$) showed an absorption band in the wave range of 1500-1600 cm^{-1} and the (OH^{-}) group was detected in the range of wave numbers between 3500-3650 cm^{-1}. The results of the FTIR characterization test showed that skipjack bone powder could be proven to contain hydroxyapatite because the phosphate group was identified as having wave numbers between 1000-1100 cm^{-1} and 500-600 cm^{-1} according to the information in table I. The particle size was then tested using PSA. The result in Figure 3 is that the average size of hydroxyapatite particles is 1.73 nm so that it can be classified as nano-sized particles (21)(22)(23).

The results of the transverse strength test in table II show that after being given the addition of nano-hydroxyapatite synthesized from skipjack tuna, acrylic samples will show an increase in transverse strength at the addition of 2%, but decreases at the addition of 5%. This value is in line with previous studies, which shown the best value of transverse strength with the addition of 2% hydroxyapatite (11,24). The increase in transverse strength can occur due to the increase in shear strength between the surface of the nanoparticles and the matrix. This causes the formation of cross-links or supramolecular bonds. Nano-sized hydroxyapatite can increase the effectiveness of particle distribution so that it can fill the space between the linear macromolecular chains of the polymer (25).

However, the addition of more nano hydroxyapatite would not increase the transverse strength. In line with other studies, the addition of 18nm nano-Al_3O_2, 15nm nano-SiO_2, and 13nm nanoTiO_2 with a concentration of 5% showed a decrease in transverse strength compared to the addition of 1% and 3%. The concentration of 5% is considered too much, causing the bond between the additive and acrylic resin to be less stable (26). Similarly, research has shown that the addition of 50 nm nano hydroxyapatite with a concentration of 5% will experience a transverse decrease (27). The addition of 5% nano concentration-hydroxyapatite will affect the magnitude of the value of the contraction pressure and the relative amount of the polymer matrix, thereby reducing the absorption of the liquid. As a result, the bond formed between the polymer and the hydroxyapatite material will be lower. The addition of too much concentration of nanoparticles can also cause particle deposition in the resin. In addition, at the saturation point of the matrix will cause the continuity of the resin matrix (28).

Meanwhile, the results of the impact strength test in table III show that the control group has a higher impact strength than the treatment group. This can be caused by the formation of agglomeration of nanoparticles. As a result, cracks will occur due to bonds in the formation of loose clusters that affect the impact strength. In addition, poor adhesion between the untreated filler particle and the matrix will decrease the energy dissipation per unit volume. Therefore, the value of the impact strength will decrease as the concentration of nano hydroxyapatite increases, especially at a concentration of 5% (29)(30).

CONCLUSION

From the results of the test of transverse strength and impact strength, nano-hydroxyapatite can be useful as a reinforcing material or filler for heat cured acrylic resin when used at a concentration of 2% because at that concentration the transverse strength shows the best performance, while the impact strength does not decrease significantly.

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