International Journal of Advanced Engineering and Management Research
Vol. 3 Issue 3; 2018

www.ijaemr.com ISSN: 2456-3676

THE EFFECT IN USING BIOCERAMIC POWDER OF PROCESSED COCKLE (BLOOD CLAM) SHELL AND ACRYLIC RESIN AGAINST THE ARTIFICIAL TOOTH MECHANICAL PROPERTIES

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Abstract

Cockle (Anadara granosa) is a kind of sea shell which can be used together with acrylic resin as artificial tooth material. Within this study, cockle bioceramic was taken as the replacement particles of artificial tooth. Bioceramic powder precursor was prepared by burning, grinding, honing to make it smooth and sift in order to have bioceramic precursor particles with the roughness number sieve level #125. The concerning mechanical properties within this study were compressive strength and micro structure of BACK (cockle bioceramic)+TRA (acrylic resin powder) replacement material. Then, these mechanical properties were compared to tooth mechanical properties. BACK powder blend composition is 0.5; 0.75; 1.375; and 1.875 gram of BACK+TRA+CRA (acrylic resin liquid) material weight. The optimum mechanical properties were obtained in the blend with 0.75 grams BACK. BACK+TRA blend had higher compressive strength than tooth natural strength, so it had potency to be applied as tooth replacement material referring to its mechanical properties.

Key Words: Anadara granosa, Bioceramic, Acrylic resin, Artificial tooth, Mechanical properties

INTRODUCTION

Dental damage is a serious problem as it can disturb the mastication function. The more and the longer the damage can risk the disruption on the dental structure [1]. Due to this damage, the demand on artificial tooth is increasing. The artificial tooth used is commonly the one of fixed or removable shapes. Economically, the making of these teeth need much additional fee which is costly [2]. Most of this kind of teeth are made of self cured acrylic and heat cured acrylic resin. Recently, acrylic resin is used extensively as the main substance for the artificial tooth. However, their availability in Indonesia should be imported and costed. Therefore, an effort is needed in finding alternative for replacement substance with appropriate biocompatibility on human body, and one of them is hydroxyapatite substance (HA) [3].

On the other hand, Kuala Tungkal, Jambi produces 267.7 ton cockles per year [4, 5].

Certainly, it can cause enviromental issue because of unutilized shell waste. Thus, the waste treatment becoming hydroxyapatite is very promising and can prevent enviromental damage. Several previous researches reported that synthesized hydroxyapatite mechanical properties has 917 MPa and 196 MPa on average for compressive and tensile strength [6]. Then Rodri LM, *et al*., (2001) also stated that hydroxyapatite has 3000 kg/mm2 (294 MPa) compressive strength, 1500 kg/cm2 (147 MPa) buckling strength, and 350 kg/mm2 (3.43 MPa) Vickers hardness [7]. Meanwhile, Suchanek, Yoshimura (1998) highlighted that dense hydroxyapatite has 38-250 MPa buckling strength, and 120-900 MPa compressive strength [8].

Moreover, factors which influence the HA mechanical properties are; pulverulent, pores and grain size. HA powder has a precise stoichiometry which is Ca: P molar ratio as much as 1.67% [9], and with this ratio can produce superior HA mechanical properties. According to Willman (1996), irregular and unrelated (unattached) HA pores are the factors weakening the HA strength [10]. Up to this time, the substance which is equal with mechanical and biological properties of human tooth yet found. Due to complicated human tooth structure which consists of enamel, dentin, cementum, and pulp. The first three teeth is human dental hard tissue marked by unique mechanical properties [11]. The hardness level of enamel and dentin on all teeth of adult and teenage is 343-683 VHN (3.36-6.69 GPa) [12].

The uniqueness of human tooth makes the artificial tooth that can resemble original tooth properties requiring acrylic resin as additional material. This acrylic contains of some pigment that can be matched on human tissue of several races. The temperature value can be varied from one product to another, is depended upon the average molecule mass and residual monomer level. Common temperature value of heat cured acrylic resin is 105^{o}C. Elastic modulus value decreases and creep potential raises further at the temperature degree close to its temperature. Even, it can cause distortion by soaking the artificial tooth in the boiling water. The temperature value for self cured acrylic resin is usually lower than heat cured acrylic resin, which is about 90^{o}C. The use of water at 65^{o}C can be avoided for soaking the artificial tooth. The temperature can be lowered down to 60^{o}C or even lower than that if the amount of molecule or residual monomer is low as well. This can be occured when the substance is not neatly cured and mostly happened in self cured acrylic resin [13].

Since the availability of cockle shell raw material as the HA source is overflow, thus alternative to utilize the piled up waste should be considered to save the environment. One of the alternatives is as bioceramic powder precursor which has been prepared by burning, grinding, honing to make them smooth and sift so that these precursor particles (BACK) are obtained with the roughness number sieve level #125. Nevertheless, this BACK substance yet can be utilized in order to make the artificial tooth as the precise mixture of BACK and acrylic resin has not been apprehended. Thus, more researches should be done referring to the effect of additional BACK powder on acrylic resin as artificial tooth material against the mechanical properties for artificial tooth application.

METHODOLOGY

2.1 Material and Equipment

The chosen cockle shell in this study was taken from the coast in Mendahara Kuala Tungkal Tanjung Jabung Timur Jambi. Chemical composition analysis of this shell was conducted using foundry Master Expert 52Q0070, where surface morphology using Scanning Electron Microscopy (SEM-EDX) Hitachi S-3400N, EMAX X-Act type, and using pulvarizer machine and ball mill FRITSCH serial No.06.2000/03075 in order to refine it. Then the sample was

characterized to determine its mechanical properties which were hardness test using Vicker Hardness Tester Shimadzu HMV-2, compressive test using Universal Hydraulic Press and micro structure using microscope Olympus GX71F.

2.2 Procedure

Initially, the cockles were washed, dried, and analyzed for its chemical composition before refining it, and the testing result can be viewed in Table 1. Afterward, these cockle shells were pounded using the hammer until they were smooth, then grinded using pulvarizer machine, and continued using ball mill until the powder with 62 µm fineness obtained. This cockle powder then was calcinated in furnace at 200, 400, 600 dan 800^{o}C, 30 minutes for heating time and 10 minutes holding time in order to get equalization on CaO grain fineness perceived within this process. Next, this CaO powder was sieved to get the appropriate size of the fine grain.

The sieving process was conducted in three stages to get micron of CaO grain particle which was going to be made into bioceramic (BACK). The first stage was fining the particle using pulvarizer machine to reduce its mass becoming 4.32 gram from 145.08 gram initial mass. The second stage took ball mill machine with 156.87 gram particle mass due to additional of new powder as much as 11.79 gram to fill the lack of fine powder when it was necessary. In this second stage, the obtained fine powder was 9.85 gram. To optimize the needed BACK particle amount, then the third stage was undertaken, which was heating 145.61 gram fine grain BACK particle using ball mill machine. So the process could attain more fine grain BACK as much as 58.08 gram with 62 μm as the finest grain size, and its process detail can be seen in Table 3. There was a temperature setting and grain size examination using optical microscope on each stage. The result data of the grain sizing examination on 5 observation points was shown in Table 3 and its dispersion in Figure 1. BACK fine particle preparation process was conducted regularly in order to have necessary amount for making 30 speciments.

This CaO powder was ready to use as the calcium of BACK synthesis source. This BACK powder then was blended with resin acrylic powder, the material of artificial tooth (TRA), and varied composition mass percentage of resin acrylic liquid (CRA) as seen in Table 4. The mixture of these three materials then were molded with the diameter of (10x10) mm, and dried afterward in the drying machine with average temperature of 85°C. The mold had been through dimension adjustment process with the standard speciment size for compressive test, hardness and micro structure. Molded speciment then was dried out in electric rice cooker at 74-85°C. This study design applied ANOVA (Analisis of Varians) with perfectly random design as the speciment design, BACK and TRA variables, CRA fixed variable, and speciment drying temperature control variable in the rice cooker.

RESULT and DISCUSSION

3.1 Analysis of Chemical Composition

The chemical composition examination result of Kuala Tungkal Jambi cockle shell using SEM-EDX is shown in Table 1. Calcium carbonate and carbon were more than 98.7% of total mineral content, where Mg, Na, P, K, and others (Fe, Cu, Ni, B, Zn dan Si) were 1.3%.

Table 1. Speciment Chemical Composition (BACK+TRA+CRA)

Unsure	Specimen											
	1A (%)		2A (%)		3A (%)		1B (%)		2B(%)		3B (%)	
	mass a	atom	mass a	atom	mass a	atom	mass a	atom	mass a	atom	mass a	atom
C	30.00	43.07	64.04	73.19	17.54	27.85	27.88	40.58	20.78	**32.14**	29.35	41.39
O	**43.80**	**46.18**	**28.11**	**24.12**	**45.57**	**54.32**	**42.59**	**46.54**	**46.76**	**54.30**	**44.79**	**47.42**
Ca	25.54	10.75	7.86	2.69	36.15	17.19	29.53	12.88	24.17	**11.21**	25.05	10.59
Na	-	-	-	-	0.77	0.64	-	-	-	-	0.81	0.60
Zn	-	-	-	-	-	-	-	-	8.28	2.35	-	-
Total	100	100	100	100	100	100	100	100	100	100	100	100

The BACK+TRA+CRA mixture speciment was then undergone micro structure investigation. BACK investigated the speciment composition of blender material 1.375 gram; 1.5 gram; and 1.875 gram with TRA: 0.5 gram and 0.75 gram utilizing SEM-EDX. The oxygen (O) element was the one that had the most composition on each BACK and TRA blends, except for 1.5 gram BACK blend and 0.5 gram TRA that had the most composition on Carbon (C) element with the weight of 64.04% and 73.19%. The highest composition of Calcium (Ca) was on BACK, 1.875 gram with 0.5 gram TRA and 36.15% composition. However, the lowest calcium composition on BACK was 1.5 gram with 0.5 gram TRA and 7.86% composition.

Previous studies mentioned that shell mineral composition from west coast of Malay Peninsula consisted of $CaCO_3$, Mg, Na, P and 0,2% of others as viewed in Table 2 [2]. Seen in the table that C and O contents on the two shells were high, while other mineral contents like Na, Mg, P, Cu, Fe and Zn were in low amount.

Table 2. Chemical Composition of Cokle Shell Powder

No	Component	Content (% weight)
1	$CaCO_3$	98.70
2	Na	0.90
3	P	0.02
4	Mg	0.05

5	Fe, Cu, Ni, B, Zn, Si	0.20

3.2 Analysis of Sieving

The stages of sieving process is stated in Table 3. Each stage was undergone tempertaure control, and grain sizing investigation through optical microscope. The result data on the grain investigation on 5 observation points was shown in Table 3 and Figure 1. BACK fine particle preparation process was conducted regularly in order to have necessary amount for making 30 speciments in diameter of (10 x 10) mm. Further, these speciments were going to be utilized for hardness, compressive and micro structure examination tests.

Tabel 3. Stages of sieving process on cockle shell powder to get the finest grain size

No. of Sieving	BACK Powder Mass (gram)		
	Pulvarizer Machine **Stage 1**	**Ball Mill Machine** **Stage 2**	**Furnace** **Stage 3**
Initial Mass	145.08	156.87	145.61
A1. 35 (500 μm)	54.57	4.97	13.15
A2. 60 (250 μm)	49.51	68.04	3.25
A3. 120 (125 μm)	45.57	70.02	69.71
Ayak Dasar (62 μm)	4.32	9.85	58.08

3.3 Analysis of Calcination

Back powder was calcinated and observed during the process occured, then proceeded by sintering process. From before and after observation, it was seen that the color during the calcination process changed. Before undergoing the process, the color of cockle shell at room temperature was cream with mass of 200 gram. On the contrary, after 3 hours calcination process at 800°C and slow cooling reaching 27°C room temperature, its color changed becoming white and 132 gram mass. The color changes presented the conversion on element filler composition during calciantion process. Taken from previous studies that there was mass reduction on element filler during calcination process [14]. The increasing of heating temperature (anil) caused the color changes and the darker color presented complete decomposition of organic composition. The BACK powder changing during sintering process was due to the organic decomposition within the bone powder. At 500℃, the bone powder color changed togrey and then white at 800°C. Afterward, HA color became white when sintering was conducted at (900-

1100)°C. This white color represented the loss of organic substance (such as collagen and protein) from BACK powder. Taken from that reference, it could be concluded that the color changing from cream to white after experiencing calcination process was due to organic substance decomposition [14, 15]. Hence, calcination process stages and obtained particle grain can be viewed in Table 4 and its particle dispersion in Figure 1.

Table. 4. BACK powder size at heating time 30 minutes and holding time 10 minutes

Temp. (°C)	Grain observation point (μm)					Average grain size (μm)
	1	2	3	4	5	
200	19.56	19.56	25.14	27.94	33.52	25.14
400	25.14	25.14	33.52	30.73	39.11	30.72
600	19.58	27.94	25.14	19.58	25.14	23.47
800	27.94	27.94	16.76	15.76	18.76	21.43

Sieved bioceramics powder of cockle shell used vibrating sieve in gradual starting from 500µm (No. 35), 250µm (No. 60), and 125µm (No. 120) then put up together with acrylic resin powder of artificial tooth, and added acrylic resin liquid (CRA) with varied blend comparison, after that his blend was stirred evenly, as shown in Table 5.

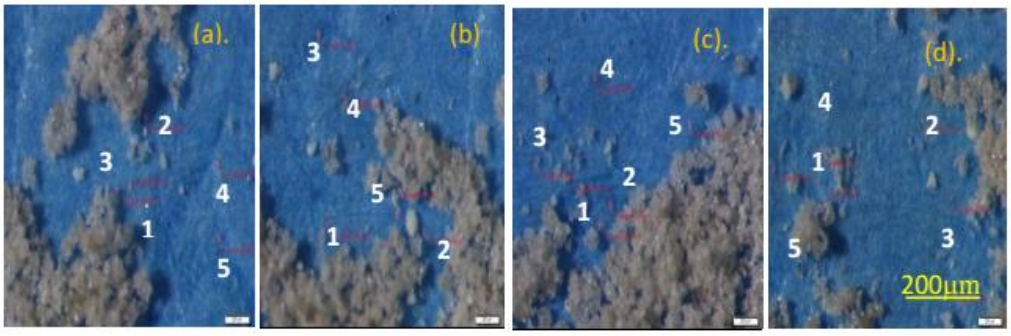

Figure 1. Grain size examination result of cockle shell powder using optical microscope (a) 1st treatment BACK powder; (b) 2nd treatment BACK powder(c) 3rd treatment BACK powder; and (d) 4th treatment BACK powder

Table 5. Speciment composition comparison (BACK+TRA+CRA)

(10x10) mm in size, 40 minutes drying time

Speciment	BACK (gram)	TRA (gram)	CRA (gram)
1A	1.375	0.50	0.01
1 B	1.375	0.75	0.01
2 A	1.500	0.50	0.01
2 B	1.500	0.75	0.01
3 A	1.875	0.50	0.01
3 B	1.875	0.75	0.01

3.4 Analysis of Mechanical Properties

Compressive test result of BACK, CRA blend composition, and varied TRA was presented in Table 6. The table described that the increase of BACK and TRA blend amount was going to decrease its compressive strength. 1.375 gram BACK powder blend with 0.5 gram TRA, and 1.375 gram BACK blend with 0.75 gram TRA had the strongest compressive strength. It was due to BACK grain dispersion against TRA was more even and finer based on SEM-EDX examination result as seen in Figure 1. Here, the uneven grain dispersion, rougher grain size and clod was reducing its compressive strength as viewed in Figure 1.

On the other hand, hardness mechanical properties usually was utilized in determining material ability characteristic against deformation because of pressure or crack on certain part of the tooth. The tooth strength was grouped into static, and dynamic strengths. Here, this study conducted static hardness test using Vicker Hardness Tester. Table 6 viewed the hardness testing result of tooth replacement material speciment.

Table 6. Result of compressive test with BACK and TRA blend treatment

BACK(gram) / **TRA (gram)**	**Compressive Test (Mpa)**		
	1.375	1.5	1.875
0.50	172	166	141
0.50	182	178	132

BACK(gram) / TRA (gram)	Compressive Test (Mpa)		
	1.375	1.5	1.875
0.75	178	132	132
0.75	182	132	132
Average	176	149	137

From Table 7, can be seen that the increase of blend composition (BACK+TRA) was decreasing its hardness level. It was because of the bigger the blend composition made uneven particle dispersion, and rougher particle due to agglutination, taken from the SEM-EDX examination result in Figure 1.

Table 7. BACK and TRA Blend speciment hardness

BACK (gram) / TRA (gram)	Hardness (Gpa)		
	1.375	1.5	1.875
0.50	0.17	0.19	0.12
0.50	0.32	0.15	0.15
0.75	0.26	0.17	0.17
0.75	0.27	0.25	0.15
Average	0.26	0.19	0.15

This study result indicated that there was an increase on BACK+TRA blend compressive and hardness strengths, with blend composition increase as shown in Table 5 and 6. Table 5 showed the highest compressive strength was on speciment with 1.375 gram BACK composition and 0.5 gram TRA, reaching 182 MPa. This compressive strength was higher than average compressive strength on tooth parts: *superficial dentin* 61.6 MPa; *middle dentin* 48.7 MPa; *deep dentin* 33.9 MPa; *dentin-enamel junction* 46.9 MPa, *Parallel enamel* 42.2 MPa and *transversal enamel* 11.5 Mpa [16, 17]. As the increasing strength is higher than the original tooth strength, therefore, concerning on strength, this blend could be utitlized as the tooth replacement. This strength was also better than the strength obtained from the tooth replacement research (cow bone powder blend and Zirconium powder), where the highest strength only arriving at 89.11 MPa [18]. In

Table 6, seen that the lowest compressive strength was on the speciment with 1.5 gram BACK composition and 0.75 gram TRA, and 1.875 gram BACK and 0.75 gram TRA, which was 132 MPa. This strength was still higher than tooth strength or biomaterial strength of the tooth material replacement from previously investigated cow bone and Zr powder [18, 19].

The data in Table 6 explained the average strength of tooth replacement material with1.375 gram BACK mass composition reaching 176 MPa; 1.5 gram BACK mass composition reaching 149 MPa, and 1.875 gram BACK mass composition reaching MPa. The percentage of TRA additional mass in the speciment was considered effective enough against speciment strength increase. This precentage only gave 7% on the strength difference, the average strength with 0.5 TRA mass percentage was 159 MPa, and 148 MPa at 0.75TRA. Comparing to HA blend strength material originating from shell flower polymer and commercial HA powder in the form of porous sample, thus the cockle BACK blend was best among others. Sintering process was done to this polymer so the sample composition was unchanged. All porous samples were in (0.2-1) *n*m diameter and (100-500) µm macro size. The processing parameter effect like sintering level, stirring time and HA concentration against physical property on porous part, the average strength was increasing varied from 1.8 to 10.5 MPa. Porosity is decreasing (59.8–34.3)%; the compressive strength is inversely proportional to porosity. Higher compressive strength caused density level of its crystal structure was higher as well. Faster sintering process made atom crystal density becoming higher. Here, this process created higher increase on compressive strength [20].

The hardness testing result in Table 7 exposed that the highest hardness was on the speciment with 1.375 gram BACK composition and 0.5 ram TRA, where the obtained hardness was up to 0.32 GPA. It was lower compared to average strength of tooth parts; *peritabular dentin* 4.7 GPa, between *mid-intertabular dentin* and *intertabular dentin* 1.8 ± 0.4 GPa, and *tabular dentin* (1.2 ± 0.4) Gpa [21]. Meanwhile in the previous studies, the obtaining hardness of replacement tooth material (blend of cow bone and Zirconium powder), the maximum hardness reached 39.1VHN (0.38 GPa) [17]. The value of average hardness perceived from this study was relatively low comparing to the earlier studies. Ideally, material strength increase was in line with hardness increase. Usually, material strength was one third of tensile strength. Whereas, compressive strength usually was about 20% lower from tensile strength value. Based on the result of obtaining average compressive strength in this study reaching 176.04 MPa, ideally average strength was going to be around 5.7 GPa. On the other hand, BACK+TRA additional gave less dominant effect toward the increase of speciment hardness because in this study, the reached average strength was only 0.27 GPa. It happened because of during the speciment preparation process; moulding, there was no strength given at the time filling the blend into the mould so that it effected BACK and TRA folding composition within the blend.

CONCLUSION

Taken from a series of undergone studies, there is an effect of BACK and TRA additional powder against its hardness and compressive strengths. The increase of blend composition variation is decreasing its hardness and compressive strengths. The highest average compressive strength reaches 176 MPa, where the lower average reaches 131 MPa. Whilst, the average hardness strength attains 0.26 GPa, and the lower average attains 0.15 GPa. Nevertheless, the adding of both is not optimal yet in the increase of delivering hardness and compressive

strengths. BACK + TRA blend has higher compressive strength than tooth natural strength, so that it is potential to be utilized as tooth replacement material referring to its mechanical properties.

ACKNOWLEDGEMENT

The writer would like to express the special thanks of gratitude to the Head of Material Physiscs Laboratorium, Faculty of Science and Technology, Jambi University. As well as to the Head of Metallurgy and Material Laboratorium, Faculty of Engineering, Andalas University Padang, for the assistance, admission and privilege in utilizing their equipments for the testing processes during the research taken place.

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