Research Article

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Preparation and characterization of oil palm ash from boiler to nanoparticle

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Abstract: This study aims to determine the characteristics of oil palm boiler ash (OPBA) after processing with ball mill and coprecipitation methods. The method used is OPBA from a palm oil mill, processed using a ball mill for 1 hour at a speed of 250 rpm. Then prepared with the coprecipitation method by dissolving it in 2M HCl solution and NaOH solution with variations (2; 2.5; 3) M. Particles were characterized by XRD, SEM, XRF, and FTIR. XRD characterization shows the size of each crystal (83,79; 72,30 and 56,31) nm, with trigonal crystal structure with the SiO_2 phase. SEM shows a homogeneous mixture. EDX shows the biggest elements are Si, O and C content. XRF shows the amount of silica is 31.45%. FTIR shows absorption peaks which are the characters of molecular vibrations of the sample.

Keywords: OPBA, Nanoparticle, Coprecipitation

1 Introduction

The characteristics of a filler will be compatible with the polymer matrix and also influenced by several factors, one of which is the particle size of the filler material. The particle size of a small filler can increase the degree of reinforcement of a polymer compared to a larger size. The smaller the particle size the higher the bond between the filler material and polymer matrix. The surface area can be increased by the presence of a porous surface on the filler. Nano addition can improve nanoscale and thermal composite properties [1]. Several methods can be used to make nanoparticles such as thermal decomposition, microemulsions, coprecipitation, sol-gel, hydrothermal, and sonochemical [2].

Oil palm boiler ash (OPBA)can be used as an economical and environmentally friendly filler. OPBA is ash derived from shells and fruit fibers which have been ground and burned at a temperature of 500 to 700°C in a boiler furnace [3]. Palm Oil mills are equipped with boilers as steam generators which are used for the production process and driving a steam turbine as an electric power plant to run crude palm oil and other processing machines. OPBA's increasing amounts are becoming an environmental problem [4]. OPBA is biomass with silica (SiO_2) content that has the potential to be utilized. OPBA contains chemical elements of silica (SiO_2) of 49.50%, Al_2O_3 of 5.45%, Fe_2O_3 of 5.73%, and SiO_2 of 45.55% and Fe_2O_3 of 10.53% [5, 6].

Boiler ash has a chemical composition that resembles other aluminosilicates, such as clay. This material solidified while in the natural gas and collected using an electrostatic precipitator. Because these particles solidify during the suspension in exhaust gases. The ash particles collected in the electrostatic precipitator are usually silt-sized (0.074-0.005 μm). This material consists of SiO_2, Al_2O_3, Fe_2O_3, Na_2O, MnO, MgO, P_2O_5, CaO, and K_2O [7]. researching with the use of industrial waste which has great benefits. Alumina is an important ceramic oxide material with immense potential for use in an extensive range of engineering products [8]. Research has been carried out on the use of various types of natural rubber compound materials and thermoplastic elastomers [9–12]. The preparation of composite materials based on OPBA waste powder is not much done while research on the use of various types of natural rubber compound fillers and thermoplastic elastomers has been widely carried out [13–15]. However, this material has disadvantages such as expensive prices and a limited amount of material availability. Therefore, the use of silica originating from OPBA as a filling material can overcome these problems because it is quite abundant and easily obtained. Research on making carbon black from OPBA has been carried out with the ball mill method, among others [5, 11], making nanoparticles using the co-

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precipitation method has been widely carried out, among others [17–22].

In this case, the researcher will use the coprecipitation method and combine it with a ball mill process to obtain OPBA nanoparticles. The coprecipitation method is one method of synthesis of inorganic compounds based on the deposition of more than one substance together when it passes the saturation point. The coprecipitation method is promising because it uses low temperatures so that the time needed is relatively shorter around 12 hours. Besides, the coprecipitation method is the most simple and easy method to do. Coprecipitation methods are used in making Palm Oil Empty Bunches Powder, the results show homogeneous particle distribution [16]. Tools and materials that are easy to obtain, so that the synthesis process can be carried out flexibly. Some of the most commonly used substances as precipitating substances in coprecipitation are hydroxide, carbonate, sulfate, and oxalate [17].

The purpose of this study was to obtain nanoparticle size and characteristics of OPBA using coprecipitation and ball mill methods. In this study acid and base will be used. 2M HCl as a solvent and for removing impurity levels. Molar variations of NaOH (2; 2.5 and 3) M are used as settling and neutralizing the acid.

2 Experimental

2.1 Materials

OPBA from PT. DPI (Dhajaja Putra Indonesia) Asahan District North Sumatra Indonesia, 2M HCL, NaOH (2; 2,5 and 3) M, and distilled water.

2.2 Preparation of OPBA nanoparticles by ball mill and coprecipitation methods

OPBA from the palm oil processing plant was dried using a furnace for 60 min at 150°C then at a ball mill with a Retch type 200 for 1 h at a speed of 250 rpm. Then the ash filtered using a 200 mesh (74μm) sieve. OPBA sized 74 μm as much as 10 g was dissolved in 40 ml of 2M HCl, and stirred with a magnetic stirrer at 70°C for 40 min then filtered. The reactions that occur are:

$$SiO_{2(s)} + 4HCl_{(l)} \Longrightarrow SiCl_{4(s)} + 2H_2O_{(l)} \quad (1)$$

Then after being filtered, OPBA which settles on filter paper is put into a glass beaker, and then mixed with NaOH 2 M as much as 40 ml, then it is stirred for 40 min with the temperature at 70°C using a magnetic stirrer. After that the NaOH solution with OPBA was separated by filtering using filter paper and repeated washing for 5 times using distilled water to obtain a neutral pH then the precipitate was dried in an oven at 70°C for 6 h with the following reaction:

$$SiCl_{4(s)} + 2H_2O_{(l)} + 4NaOH_{(l)} \Rightarrow SiO_{2(s)} + 4NaCl_{(l)} + 4H_2O_{(l)} \quad (2)$$

Similarly for 2.5 M and 3 M NaOH solutions.

3 Results and discussion

3.1 Analysis of OPBA Nanoparticles

3.1.1 XRD analysis of OPBA nanoparticles

X-Ray Diffraction (XRD) characterization was carried out to obtain diffraction patterns, crystalline structures and particle sizes of OPBA nanoparticles. Shimadzu XRD 6100 (40 kV, 30 mA) with Cu – Ka1 wavelength = 1.5405 Å = 0.15406 nm. at a rate of 2° / min in the angle range, 2θ = 5° to 70° used in this study. XRD characterization is carried out at room temperature and uses nickel to filter CuKa radiation. The sample crystallite size was calculated based on the Scherrer method of X-ray diffraction patterns. From the XRD diffraction pattern, particle size is obtained by calculating the amount of Full Width at Half Maximum (FWHM) from the diffraction peak through the Scherrer equation approach. FWHM is converted into radians by multiplying $\pi/180$.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (3)$$

With β is the line broadening at half the maximum intensity, K is the Scherrer constant (0.9), λ is X-ray wavelength (1.5406 Å), and D is the diameter of the crystal (nm). From equation 3, particle size was obtained from variations of 2, 2.5 and 3 M NaOH solutions of (83.79, 72.30 and 56.31) nm. The OPBA nanoparticle size in this study is better than the previous study [1, 15] where the OPBA size was 85.35 nm and previous research obtained OPBA size of 100 nm [1], this was due to the method used in this processing OPBA is different from previous research methods.

The results of OPBA X-ray diffraction patterns with variations of NaOH solution are shown in Figure 1 and Table 1.

The results of OPBA X-ray diffraction patterns with variations of NaOH solution are shown in Figure 1.

Table 1: XRD Analysis of OPBA Nanoparticles with ball mill processes and coprecipitation methods

Date	OPBA +HCl NaOH 2M	OPBA+HCl NaOH 2,5M	OPBA+HCl NaOH 3M
Crystal system	Trigonal	Trigonal	Trigonal
Space group	P 31 2 1 (152)	P 31 2 1 (152)	P 31 2 1 (154)
The lattice meter	A= 4.9019 A	A= 4.9158 A	A= 4.9115 A
	c=5,3988 A	c=5,4091 A	c=5,4038 A
Density	2,664 g/cm^3	2,644 g/cm^3	2,649 g/cm^3
2 theta angle	26,7552	26,6603	21,9040
Maximum d_{hkl} Intensity 1	011	011	011
Lattice distance d (Å)	3,3371	3,3454	3,3423
2 theta angle	21,9656	37,9906	26,6200
Maximum d_{hkl} Intensity 2	100	100	100
Lattice distance d (Å)	4,2452	4,2572	4,2535
2 theta angle	26,52	21,281	25,70
Maximum d_{hkl} Intensity 3	112	112	112
Lattice distance d (Å)	1,8146	1,8190	1,8173

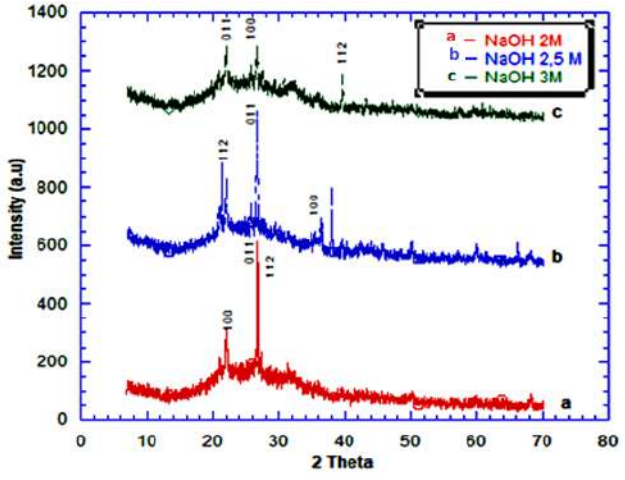

Figure 1: OPBA diffraction pattern with variation of NaOH solution

3.1.2 SEM Analysis

Based on the results of observations in Figure 2(a) pure OPBA show morphological surfaces in the form of solids that are fused or coagulated, in contrast to (Figure 2b) OPBA using 2M NaOH, the clotting on the surface begins to separate, in (Figure 2c) OPBA using 2.5 M NaOH shows shape small and tight circles, in (Figure 2d) using 3M NaOH shows a circular surface morphology smaller than (Figure 2a, Figure 2b, Figure 2c) and neatly arranged and classified as polycrystalline.

3.1.3 EDS analysis of OPBA nanoparticles

Table 2: The results of OPBA analysis with EDS

Composition	OPBA +HCl NaOH 2M (wt.%)	OPBA+HCl NaOH 2,5M (wt.%)	OPBA+HCl NaOH 3M (wt.%)
O	29,74	25,17	23,83
Si	17,42	6,89	16,78
C	22,70	48,50	14,67
Ca	14,82	5,18	26,40
Mg	4,40	1,77	4,02
Al	3,93	2,51	3,16
Fe	3,15	1,37	6,72
P	2,94	-	3,09
K	0,91	-	1,33
Nb	-	4,25	-
Zr	-	3,72	-
Na	-	0,65	-
Total	**100**	**100**	**100**

3.1.4 XRF analysis

From the XRF analysis the contents of the nanoparticle elements are shown in Figure 3 and Table 3.

Making nanoparticles by coprecipitation method serves to eliminate impurity levels in OPBA so that it is

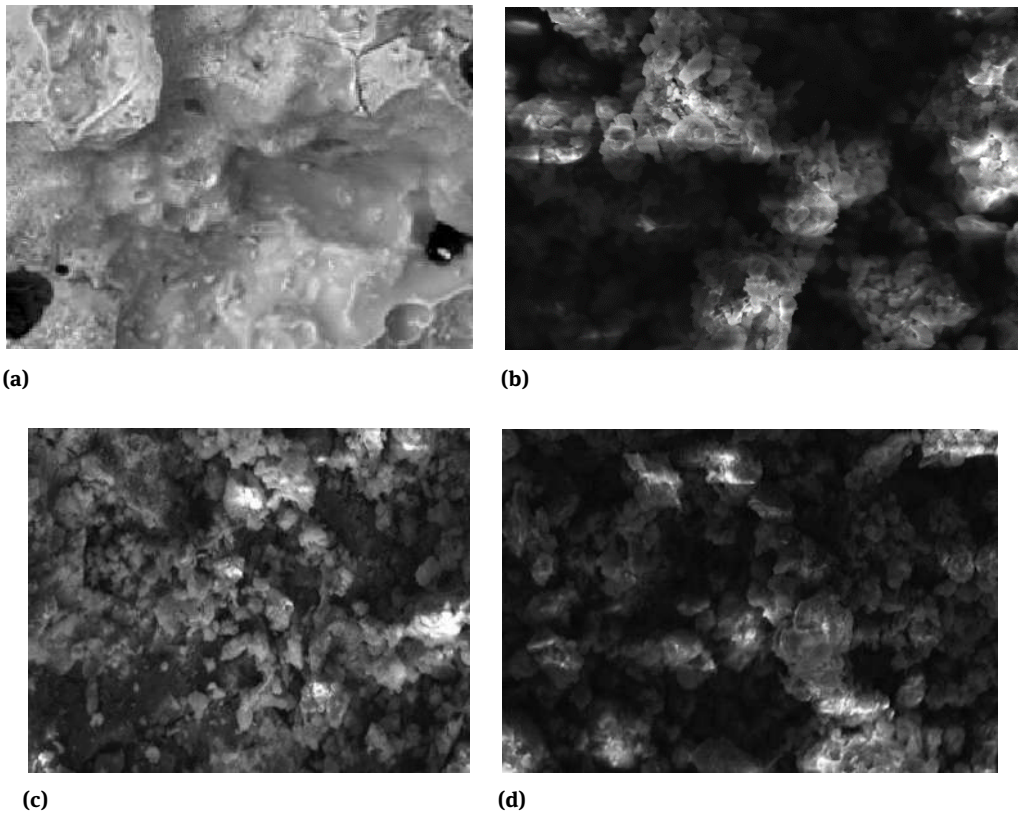

Figure 2: Morphology a. Pure OPBA ,b .OPBA with NaOH 2M c .OPBA with NaOH 2,5M d.OPBA with NaOH 3M

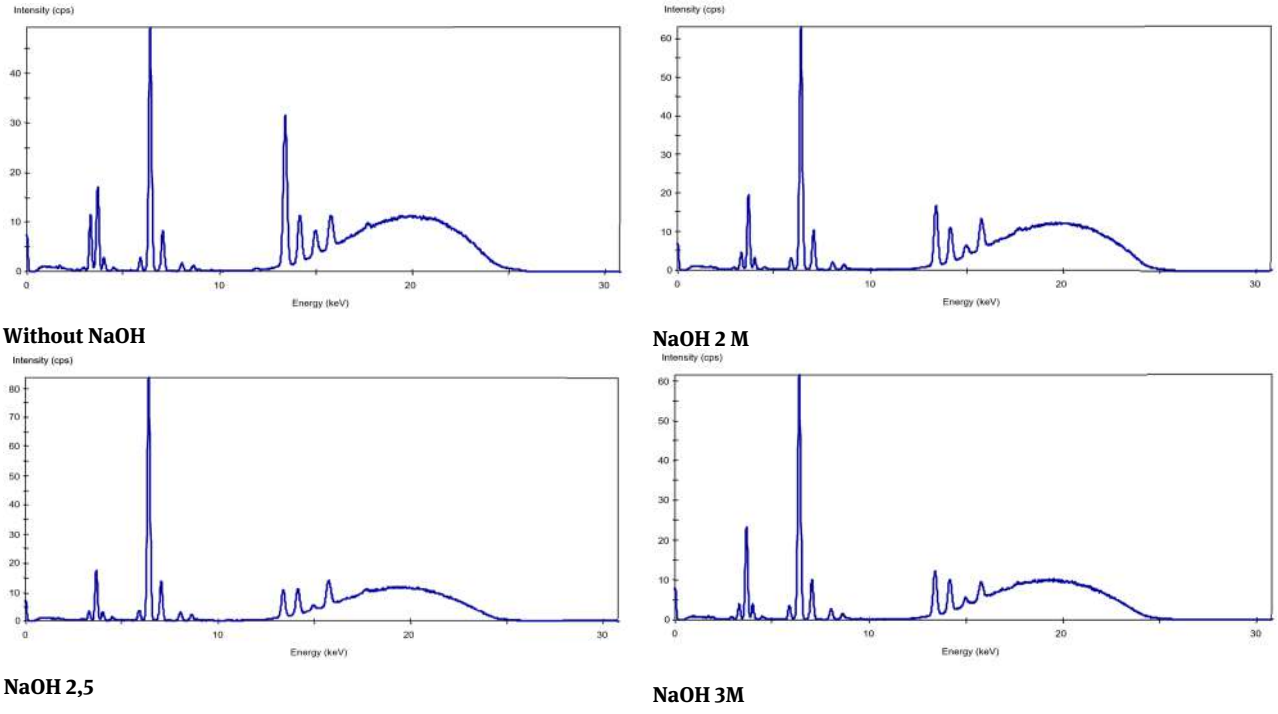

Figure 3: OPBA nanoparticle XRF analysis

Table 3: Elements obtained from XRF analysis

No	elements	Composition (wt%)without NaOH	Composition (wt%) NaOH 2M	Composition (wt%) NaOH 2,5M	Composition (wt%) NaOH 3M
1	Si	46.956	58.749	65.277	56.827
2	Fe	27.789	25.549	18,663	27.770

Table 4: Palm Oil Boiler Ash Nanoparticle Elements Contents

No	Element	Composition (wt%)
1	Mg	1.552
2	Al	16.520
3	Si	37.031
4	P	1.889
5	S	0.951
6	Ti	1.264
7	Mn	0.974
8	Fe	19.509
9	Co	0.080
10	Cu	0.428
11	Zn	0.197
12	Zr	0.265
13	Ag	4.117
14	Sn	13.765
15	Sb	1.458

expected to produce more levels of silica. Table 4 shows the highest content of Nanoparticles Si 37,031 wt%, Fe 19,509 wt%, Al 16,520 wt% and Sn 13,765 wt%.

In this research, there is a higher amount of silica compared to the research of Nanda *et al.* [12] which had a silica content of 31.45%. However, it was found that the higher silica content was 45.55% in OPBA [5].

3.1.5 FTIR analysis of the OPBA nanoparticles

Fourier Transform Infra-Red (FTIR) characterization using the Perkin Elmer spectrum one type of FTIR device. This characterization aims to find out the functional groups of material. The information obtained from this characterization is transmittance and wavenumber spectra, so that from the FTIR results it can be seen that the bond or functional group of OPBA nanoparticles. the functional group of NaOH 2 M OPBA nanoparticles is shown in Figure 4

FTIR characterization shows that there are absorption peaks from the sample. The peaks show absorption groups which are characteristic of molecular vibrations at wave numbers (468, 796, 1096, 1651, 3467) cm^{-1}.

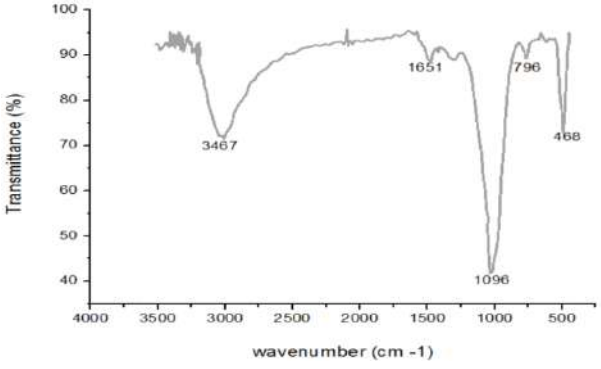

Figure 4: FTIR characterization graph

Table 5: Functional group and Wave Numbers

No	peak number	Group	Vibration
1	468	Si-O-Si	Bending
2	796	C-Cl	Stretching
3	1096	Si-O	Stretching
4	1651	Si-O	Stretching
5	3467	O-H	Stretching

In principle, FTIR is used to determine functional groups that exist in a compound, so that it can be used to determine a compound that has no known content. The wavenumber peaks and functional groups are shown in Table 5.

Table 5 lists the peak wave numbers and functional groups obtained for this study

4 Conclusion

The results of OPBA characterization processed by ball mill and coprecipitation methods using XRD showed that OPBA particles had nano size of 83.76, 72.30, 56.31 nm with quartz crystal types and trigonal crystal structures. Morphological characterization showed a homogeneous mixture, XRF showed the amount of silica was 31.45%. FTIR analysis shows an absorption peak which is a character of the molecular vibration of the sample. Increasing the molarity of NaOH solution makes OPBA particle size decrease

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