Preparation And Characterization Of Oil Palm Empty Bunches Powder

E.M.Ginting*, M. Motlan1, N.Bukit1, M. T.Saragih1, A. H. Sinaga, E.Frida2

1Department of physics, Universitas Negeri Medan, Indonesia
2Faculty of Engineering, Universitas Quality, Indonesia

*email: evamarlina@unimed.ac.id, nurdinbukit5@gmail.com

Abstract. The objective of this study was to determine the characteristics of Oil Palm Empty Bunch powder (OPEBP). The method performed in OPEBP processing into nanoparticles is used by the method of ball mill and coprecipitation. The characterization results of X-Ray Diffraction (XRD) show that the OPEBP crystalline diameter size is 68.62 nm with Crystal system trigonal (hexagonal axes) Unit cell a= 8.376 Å, c= 3.884 Å, I/Ic 3.16 with density calculated 2.536 g/cm³ the elements contained are Si, O, Ca. Morphology results showed uniform and homogeneous particle distribution.

Keywords: Nano Particles, OPEBP

1. Introduction.

Oil Palm is one of Indonesia’s main commodities that has experienced rapid progress. Beside of the high palm oil production, by-products or oil palm mill waste that produced are also high. With the condition of that, there are actually many benefits that can be obtained to create oil palm as a zero waste industry. Waste is the center of world attention in increasing protection for nature, so various technologies have been developed to utilize that waste [2,3]. Oil Palm Empty Fruit Bunches (OPEFB) are the largest solid waste produced by Oil Palm Mills (OPM). After the process of oil palm processing have been done, finally it leaving the Oil Palm Empty Fruit Bunch (OPEFB), oil palm is well known as one of the most economical [4].

Availability of OPEFB is quite significant if it is reviewed based on the average ratio of OPEFB production to the number of FFB that processed. The average production of OPEFB is around 22 to 24% of the total FFB that processed in POM [5]. The results of combustion of OPEFB waste on the combustion engine will be producing the powder which is called the Oil Palm Empty Bunches Powder (OPEBP). Based on the results of the OPEBP test it can be stated that the most abundant elements in OPEBP are K2O (Potassium Oxide) which are easily oxidized. The compounds that contained in Palm Oil Mill Fly Ash (POMFA) include SiO2 39.02%, K2O 6.20%, MgO 4.11%, CaO 9.39% and P2O5 5.16% [6]. OPEBP is synthesized using the coprecipitation method with an acid base reaction, where HCl 7M as a solvent and NH4OH 3M as a settler. The result of silica gel product contains impurities of C, Na, K, Al, and S. The elements of C, K and Al are present in the POMFA and a small amount of those elements has been carried over in the silica gel [7].

The field of nanotechnology is one of the most popular fields for research today because the materials in nano-size particles usually have chemicals material or physical properties that are superior than the big size materials (bulk) [8]. Nanoparticles are microscopic particles about 1-100 nm in size.
Research in the field of nanoparticles produces unique material properties that materials with nanoscale have different properties from their original material. To get the minimum nanoparticle size, a material is carried out by several methods, one of which is the synthesis method. The nanoparticle synthesis process consists of several methods including sol-gel method, coprecipitation, microemulsion, hydrothermal / solvotherma, using mold (templated synthesis), biomimetic synthesis, supercritical fluid method and ionic liquids synthesis.

The factors that affect the synthesis of nanoparticles including reactant concentrations, coating molecules (capping agent), temperature and stirring. Researchers use the coprecipitation method because the cost is relatively cheaper. In addition, because the process is simpler based on the deposition of more than one substance together when it passes through the saturation point using a low temperature (70°C) and is easy to control the particle size so that the time that required is relatively short. Some of the substances that most commonly used as precipitating agents in coprecipitation are hydroxides, carbonates, sulfates and oxalates [9]. Through this method, the researchers combined the ball mill method for grinding OPEBP to obtain the smaller nanoparticle sizes than the previous studies.

The purpose of this study is to determine the particle size of Oil Palm Empty Bunches Powder (OPEBP), to determine the morphology and the element content of OPEBP particles. The method used in the preparation of OPEBP nanoparticles is the ball mill and coprecipitation method.

2. Experiment
2.1. Materials
OPEBP derived from PT.DPI (Dhajaja Putra Indonesia) Asahan district of North Sumatra-Indonesia. HCL 7M and NH₄OH 3M.

2.2. Preparation of Nanoparticles OPEBP with ball mill and Coprecipitation Method.
The process of making OPEBP nanoparticles was first done by drying the OPEBP in the furnace with a temperature of 150°C for 1 hour in order to separate the water content that is still contained in the OPEBP. The Results from furnace is milled with Retsch Planetary ball mill PM 200 for 2 hours at a speed of 250 rpm. Then it sifted using a 200 mesh sieve so that it produce a 74 µm. OPEBP is then synthesized by using the coprecipitation method.

Then, 10 g of OPEBP are put into a glass beaker, then dissolved in 7 M HCl as much as 40 ml, then it stirred and heated in a magnetic stirrer at 70°C for 40 minutes. The OPEBP dissolved again with a 40 ml 3M NH₄OH solution. Then it is stirred and heated in a magnetic stirrer at a temperature of 70°C for 40 minutes. The solution is washed and then precipitated using distilled water. The results of OPEBP deposits are dried in an oven for 4 hours at a temperature of 70°C. The dried of OPEBP is ground using mortal and pestle, so that the nanoparticles are obtained. Then structural analysis and particle size were carried out using X-Ray Diffraction (XRD) and morphological analysis was performed using Scanning Electro Microscopy (SEM) and analysis of OPEBP particle elements was performed using X-ray fluorescence (XRF).

3. Result and Discussion
3.1. Results of Characterization of OPEBP Nanoparticles Synthesis
OPEBP measuring 74 µm was synthesized using the coprecipitation method with an acid base reaction, where 7M HCl as a solvent and NH₄OH 3M as precipitant.

\[
\begin{align*}
\text{Na}_2\text{O} + \text{HCl} &\rightarrow 2\text{NaCl} + \text{H}_2\text{O} \\
\text{MnO} + 2\text{HCl} &\rightarrow \text{MnCl}_2 + \text{H}_2\text{O} \\
\text{MgO} + 2\text{HCl} &\rightarrow \text{MgCl}_2 + \text{H}_2\text{O} \\
\text{P}_2\text{O}_5 + 10\text{HCl} &\rightarrow \text{P}_2\text{Cl}_{10} + 5\text{H}_2\text{O} \\
\text{Al}_2\text{O}_3 + 6\text{HCl} &\rightarrow \text{Al}_2\text{Cl}_6 + 3\text{H}_2\text{O} \\
\text{CaO} + 2\text{HCl} &\rightarrow \text{CaCl}_2 + \text{H}_2\text{O}
\end{align*}
\]
\[
\text{SiO}_2 + \text{HCl} \quad \rightarrow \quad \text{SiCl}_4 + 2\text{H}_2\text{O}
\]

The OPEBP solution are dissolved again with 150 ml NH\textsubscript{4}OH with a reaction as follows:

\[
\text{SiCl}_4(\text{s}) + 2\text{H}_2\text{O}(\text{l}) + \text{Na}_4\text{OH}(\text{l}) \Rightarrow \text{SiO}_2(\text{s}) + 4\text{NaCl}(\text{l}) + 2\text{H}_2\text{O}(\text{l})
\]

The solution was washed and then precipitated using distilled water solution. Results OPEBP precipitate dried in an oven for 4 hours at a temperature of 70°C. OPEBP Particle Results are shown in Figure 1.

![Figure 1. (a)OPEBP, (b) Micro particle, (c) Nanoparticles](image)

3.2. Results of XRD (X-Ray Diffractometry) Characterization of OPEBP Nanoparticles.

XRD (X-Ray Diffraction) testing was carried out to obtain the diffraction pattern, crystalline structure and particle size of OPEBP nanoparticles. All materials containing certain crystals when analyzed using XRD will bring up specific peaks. So that the weakness of this tool cannot be to characterize the material that have amorphous properties [10,11].

The XRD diffraction pattern is obtained by particle size by calculating the amount of FWHM (Full Width at Half Maximum) from the diffraction peaks through the Scherrer equation approach. FWHM converted into units of radians by multiplying \(\pi / 180\).

\[
D = \frac{K\lambda}{\beta\cos\theta}
\]

With \(\beta\), \(K\), \(\lambda\) and \(D\) respectively is the width of the half peak of Full Width at Half Maximum (FWHM) in radians, scherrer constant (0.9), X-ray wavelength (1.5406 Å), and \(D\) is the diameter crystal (nm).

The XRD used is type 6100 Shimadzu with a wavelength of Cu – \(K_{\text{a1}} = 1.540\) Å at a rate of 2 °/minute in the 2\(\Theta\) = 5 ° - 70 ° angle range. From the results of XRD nanoparticles testing, the particle size was 68.62 nm with SiO\textsubscript{2} phase and crystal structure: trigonal (hexagonal axes) are obtained. Grid parameters: \(a = 8.375\) Å, \(b = 8.375\) Å, \(c = 3.884\) Å with fields dhkl: (110), (11-1), (001) and density: 2.536 g/cm\(^3\). The results of the X-ray diffraction pattern on the OPEBP sample are shown in Figure 2.
3.3. **Characterization result of OPEBP Nanoparticles XRF**

X-ray Fluorescence (XRF) spectrometry is a non-destructive analysis technique used for identification and determination of element concentrations that exist in solid, powder, or liquid samples. In general, the XRF spectrometer measures the wavelength of individual material components from the fluorescent emissions produced by a sample when irradiated with X-rays. The XRF test results from OPEBP nanoparticles obtained the largest Fe content as much as 39.057 wt%. The results of research that have been carried out in the manufacture of OPEBP nanoparticles with the XRF test obtained the content of OPEBP nanoparticles shown in Figure 3, and Table 1.

**Table 1. The Content Of OPEBP Nanoparticles**

| Element | HCl 7M |
|---------|--------|
| Sb      | 31.68  |
| Fe      | 39.06  |
| Others  | 29.26  |
| Total   | 100    |

**Figure 2.** Graph of X-ray diffraction patterns on OPEBP.

**Figure 3.** Test results of XRF OPEBP Nanoparticles.
3.4. Characterization result of OPEBP Nanoparticles SEM EDX (Energy Dispersive X-ray)

Scanning Electron Microscope (SEM) is used in thick samples and allows for surface analysis. The beam of files falling on the sample will be reflected and diffused. The presence of diffracted electrons can be observed in the form of diffraction patterns that are very dependent on the shape and size of the unit cell of the sample. SEM can also be used to infer crystallographic data, so that this can be developed to determine elements or compounds. The results of SEM testing on OPEBP nanoparticles are shown in Figure 4.

![SEM image of silica nanoparticles OPEBP magnification of 1000X](image1)

**Figure 4.** SEM image of silicate nanoparticles OPEBP magnification of 1000X

From the above images, it can be estimated that the range of size of the aggregate in the silica obtained

![SEM-EDS spectrometry of the of Nanoparticles OPEBP EDX](image2)

**Figure 5.** SEM-EDS spectrometry of the of Nanoparticles OPEBP EDX
EDS Analysis of OPEBP Nanoparticles.

Table 2. The composition of the OPEBP the coprecipitation product

| Element | Wt(%) |
|---------|-------|
| O       | 24.21 |
| Si      | 11.36 |
| C       | 1.23  |
| Ca      | 49.26 |
| Mg      | 2.20  |
| P       | 9.09  |
| Fe      | 2.65  |

The results of this study in accordance with the results of previous research [7]

4. Conclusion

From the results of the study obtained Ca 49.26 % wt silica content of 11.36% wt and the average particle size of 68.62 nm density: 2.536 g / cm³. In general, the results of morphology are distributed evenly and homogeneously.

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