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PREPARATION AND CHARACTERIZATION OF OIL PALM ASH FROM BOILER TO NANO PARTICLE

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Abstract:	Solid waste from the palm oil mill comes from the remaining combustion shells and fiber in the boiler machine produces oil palm boiler ash (OPBA). This study aims to determine the characteristics (OPBA) after being processed by ball mill and coprecipitation method. OPBA from the processing plant is milled for 1 hour at a speed of 250 rpm. Then prepared with 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 and XRF and FTIR. Characterization XRD shows the size of each crystal (83,79; 72,30 and 56,31) nm, with trigonal crystal structure with SiO2 phase. SEM shows a homogeneous mixture. EDX shows the biggest elements are Si, O and C content. XRF shown the amount of silica is 31.45% . FTIR shows absorption peaks which are the characters of molecular vibrations of sample.
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PREPARATION AND CHARACTERIZATION OF OIL PALM ASH FROM BOILER TO NANO PARTICLE

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Abstract.Solid waste from the palm oil mill comes from the remaining combustion shells and fiber in the boiler machine produces oil palm boiler ash (OPBA). This study aims to determine the characteristics (OPBA) after being processed by ball mill and coprecipitation method. OPBA from the processing plant is milled for 1 hour at a speed of 250 rpm. Then prepared with 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 and XRF and FTIR. Characterization XRD shows the size of each crystal (83,79; 72,30 and 56,31) nm, with trigonal crystal structure with SiO_2 phase. SEM shows a homogeneous mixture. EDX shows the biggest elements are Si, O and C content. XRF shown the amount of silica is 31.45% . FTIR shows absorption peaks which are the characters of molecular vibrations of sample.

1 . INTRODUCTION

Nanoparticle technology in the material field is a very interesting study, because nanoscale materials have more superior properties than the bulk materials. These superior properties can be carried out by controlling material size, chemical composition regulation, surface modification and controlling the interaction between particle. The nature of a filler material will be compatible with the polymer matrix, and influenced by several factors, among others, the particle size of a filler matterial, where the particle size of a small filler matterial can increase the degree of polymer strengthening compared to a larger size. and then the smaller the size of particle, then the higher the bond between filler material and polymer matrix. The amount of surface area can be increased by the presence of porous surfaces on the surface of the filler material as well as the addition of nano can improve the nanoscale mechanical properties and composites thermal property [1]. Various types of methods that used to make nanoparticles include: thermal decomposition, microemulsion, coprecipitation, sol gel, hydrothermal, and sonochemistry [2].

Each Palm Oil Mill (POM) is always equipped with a boiler as a steam generator that is used for the purposes of the production process and for drives a steam turbine as a electric power plant in order to Running CPO processing machines, lighting and other processing machines. The fuel used for boilers is solid waste, namely palm fruit fibers and palm shells.

Fuel consumption for POM boilers with a capacity of 30 tonnes / hour is 3.8 tonnes / hour of palm fruit fiber and 1.5 tons of palm shells. From the Each Palm Oil Mill (POM) production process with a capacity of 30 tons of FFB per hour, solid waste will be obtained, 3.0 tons - 3.6 tons of palm fruit fiber and 2.1 tons - 2.7 tons of palm shells, or if averaged around 2, 4 tons of palm shells. The use of palm fruit fibers as boiler fuel, is maximally meaning all the fruit fibers of palm oil are used for boiler fuel. This means that there is still an average of around 0.9 tons / hour of palm oil shells from the PKS production process with a capacity of 30 tons of FFB per hour. Suppose that every day it operates for 10 hours, then an average of 9 tons of palm shells will be obtained which can be used for various purposes, including raw materials for making palm oil shell powder as Carbon Black [3].

The residual solid waste of oil palm combustion in boilers in the form of ash with an amount that stay increasing throughout the year is become an environmental problem. Most of the waste from the palm oil mill has not been utilized or can be said to be wasted and just in vain. Solid waste from the palm oil mill results is the residual burning of the shell and fiber in the boiler machine called Palm Oil Boiler Abu (OPBA). OPBA is a biomass with silica content (SiO_2) which is potentially 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$10.53% [4,5].

Basically boiler ash has a chemical composition resembling other aluminosilicates, such as clay. This material, solidifies while during in the exhaust gases and it is collected using an electrostatic precipitator. Because these particles solidfies during suspension in the exhaust gases, these ash particles are generally round. The ash particles collected in the electrostatic precipitator are usually silt sized (0.074-0.005). This material mainly consists of silicondioxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide ($Fe2O_3$), research on palm oil waste has been done [6-9].

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. The filler that is often used is Carbon Black [10-12]. However, this material has weaknesses. In terms of expensive prices and the amount of material availability. Therefore, silica originating from OPBA as a filler can overcome these problems because of the large number and easy to obtain. Research on the manufacture of carbon black from OPBA has been carried out by the ball mill method, among others [4,9], making nanoparticles with the cooprecipitation method [13-18].

This research uses coprecipitation method combined with ballmill process to obtain OPBA nanoparticles. Coprecipitation method is one method of synthesis of inorganic compounds based on the precipitation of more than one substance together when it passes through the saturation point. The coprecipitation process uses low temperature so that the time needed is relatively shorter, which is ± 12 hours. Coprecipitation is the simplest and easiest method to do, and uses tools and materials that are easily obtained, so that the synthesis process can be carried out flexibly. Some of the substances most commonly used as precipitating agents in coprecipitation are hydroxides, carbonates, sulfates and oxalates [13].

The purpose of this study is to obtain OPBA nanoparticle size and analyze its characteristics. Preparation uses coprecipitation method and ball mill. 2M HCl is used as a solvent and to remove impurity levels, and NaOH with variations (2; 2.5 and 3) M as precipitate and neutralize acid.

2. EXPERIMENTAL

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.

Preparation of OPBA Nanoparticles By ball mill and coprecipitation methods

OPBA from the palm oil processing plant was dried using a furnace for 60 minutes at 150^0C then at a ball mill with a Retch type 200 for 1 hour at a speed of 250 rpm. then filtered using a 200 mesh (74μm) sieve. OPBA sized 74 μm as much as 10 grams was dissolved in 40 ml of 2M HCl, and stirred with a magnetic stirrer at 70^0C for 40 minutes then filtered. The reactions that occur are:

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

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 stirred for 40 minutes with temperature at 70^0 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 70^0 C for 6 hours.

with the following reaction :

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

In the same way it is done for 2.5 M NaOH solution and 3M.

Figure 1 shows the change in OPBA after experiencing the process

a b c

Figure 1. a. OPBA b. OPBA after furnice c. Nano partikel OPBA

3.RESULTS AND DISCUSSION

Nanoparticle Synthesis Analysis (OPBA).

XRD *(X-Ray diffraction)* testing was performed to obtain the diffraction pattern, crystalline structure and particle size of OPBA nanoparticles. The XRD used by Shimadzu 6100 (40 kV, 30 mA) with a wavelength of Cu – K_{a1} = 1.5405 Å = 0.15406 nm at a rate of 2 ° / minute in the 2Θ = 5 ° -70 ° angle range. XRD testing is carried out at room temperature and uses nickel to filter CuKa radiation. The sample crystallite size is calculated based on the Scherrer method analysis of X-ray diffraction patterns.

From the XRD diffraction pattern, the particle size is obtained by calculating the amount of FWHM (Full Width at Half Maximum) from the diffraction peak through the Scherrer equation approach. FWHM is converted to radians by multiplying π / 180

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

With β, K, λ and D respectively the width of the half peak Full Width at Half Maximum (FWHM) in radians, scherrer constant (0.9), X-ray wavelength (1.5406 Å), and D is the crystal diameter (nm). With the calculation of equation 3, the average particle size for the variation of 2, 2.5 and 3 M NaOH solutions was obtained for crystal sizes (83.79, 72.30 and 56.31) nm. .

The results of OPBA particle size in this study obtained better nanoparticle size than previous studies [4], [17] obtaining OPBA size of 300 nm and [4] obtaining OPBA size of 100 nm, this was due to the method used in OPBA processing differs from previous research.

The results of data analysis for the three solution variations and the three peak intensities are shown in Table 1

Table 1. XRD analysis result data of OPBA with the ball mill process and methods of coprecipitation

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 c=5,3988 A	A= 4.9158 A c=5,4091 A	A= 4.9115 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

The results of OPBA phase X-ray diffraction pattern with variations of NaOH solution are shown in Figure 2.

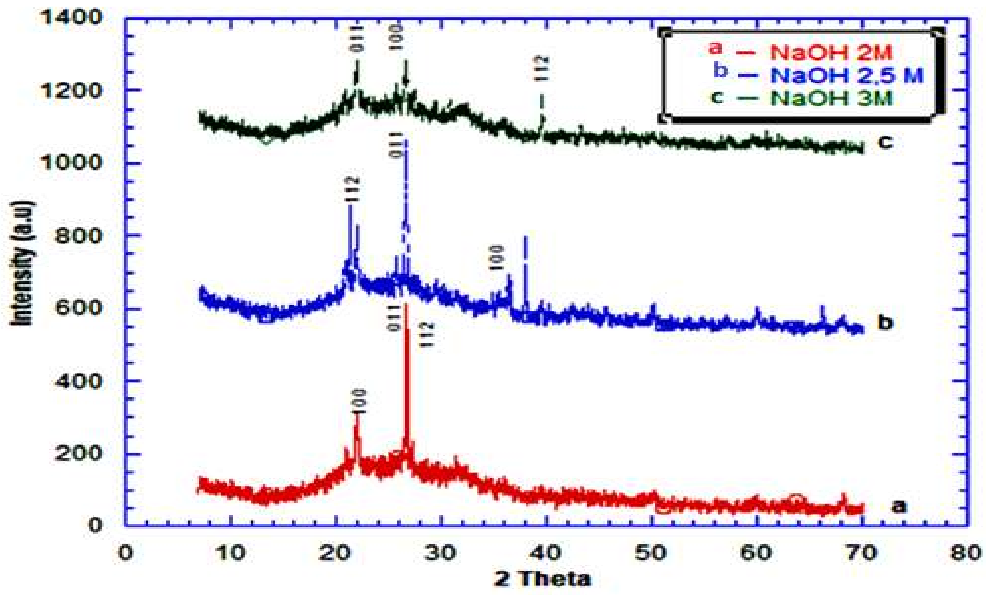


Fig. 2. OPBA diffraction pattern with variation of NaOH solution

SEM Analysis

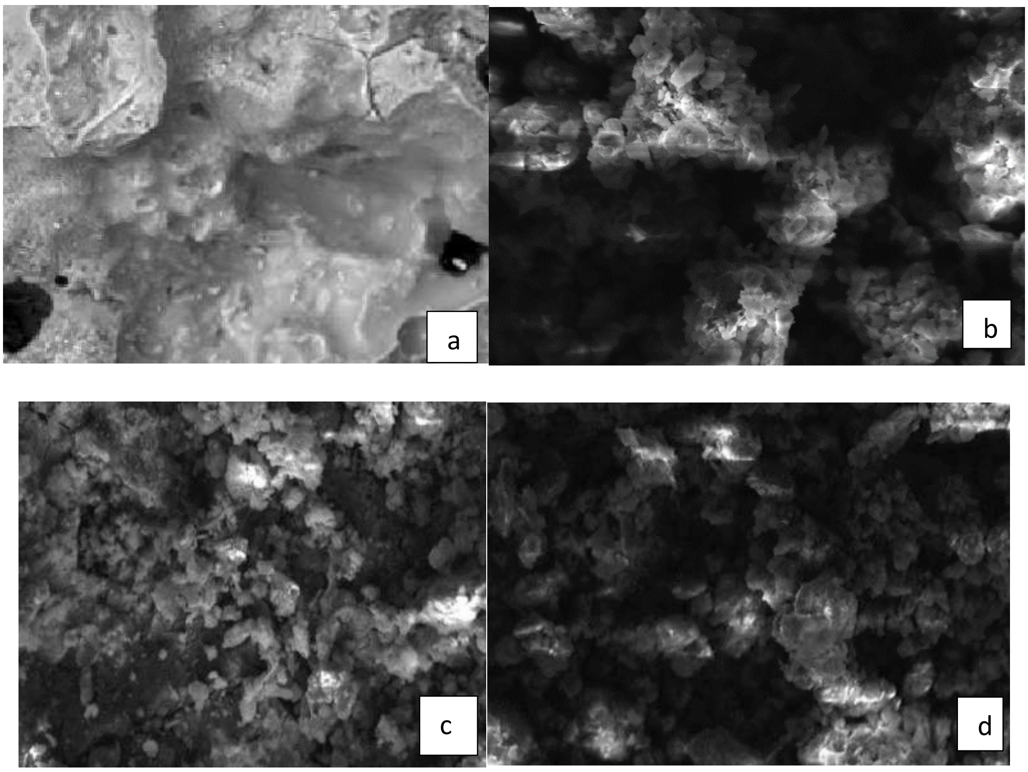


Fig. 3. Morphology a. OPBA pure ,b OPBA with NaOH 2M c .OPBA with NaOH 2,5M d.OPBA with NaOH 3M

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

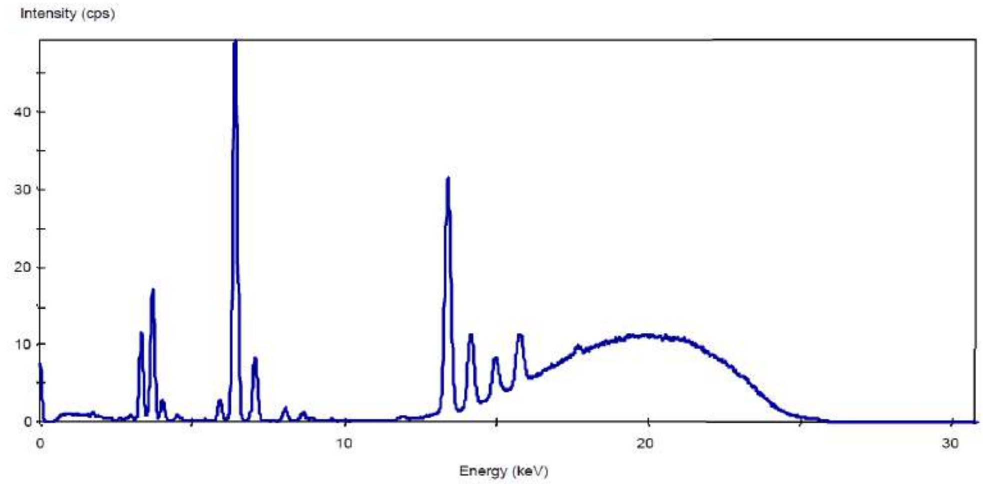
Analysis of OPBA Nanoparticles Eds.

Table 2. The results of OPBA analysis with EDS

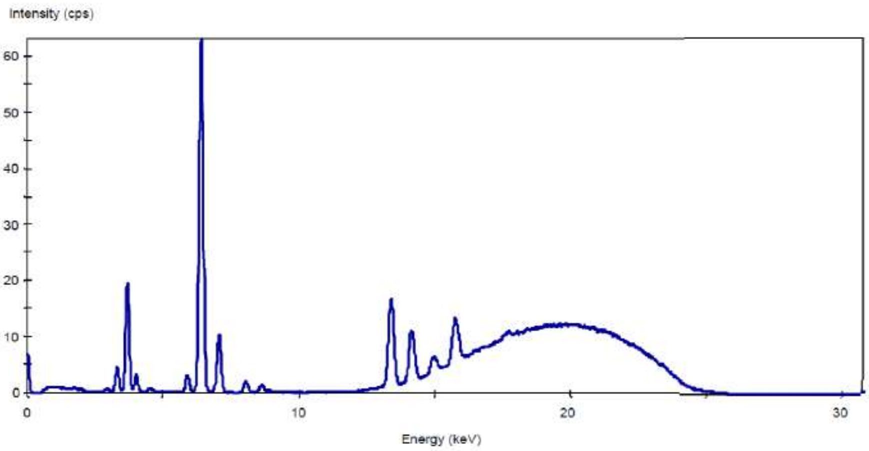
Composition	**OPBA +HCl NaOH 2M (wt.%)**	**OPBA+HCl NaOH 2,5M (wt.%)**	**OPBA+HCl NaOH 3M (wt.%)**
O	29,74	25,17	23,83
Si	17,42	6,89	16,78
C	22,70	48,50	14,67
Ca	14,82	5,18	26,40
Mg	4,40	1,77	4,02
Al	3,93	2,51	3,16
Fe	3,15	1,37	6,72
P	2,94		3,09
K	0,91		1,33
Nb		4,25	
Zr		3,72	
Na		0,65	
Total	**100**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

XRF Analysis

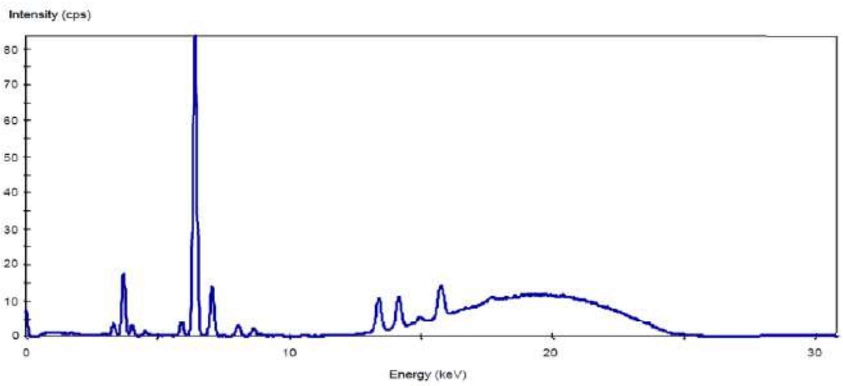
Analysis of OPBA Nanoparticles with XRF obtained Nanoparticles content as shown in Figure 5 and Table 4.



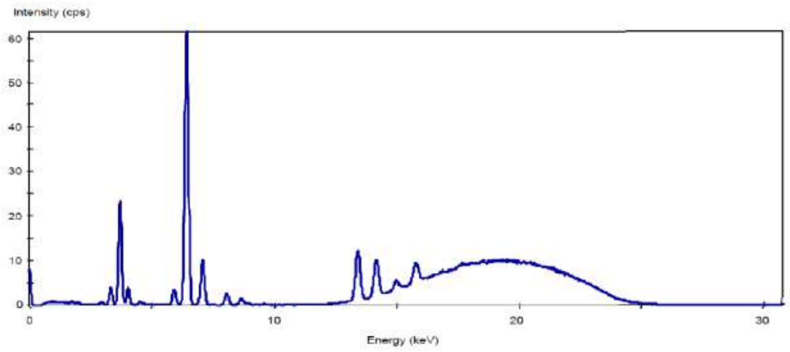
Without NaOH



NaOH 2 M



NaOH 2,5



NaOH 3M

Fig.4. XRF testing results of OPBA Nanoparticles

Table 3. Results of elements of XRF testing

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

Preparation of Nanoparticles with coprecipitation method is useful to remove impurity levels in oil palm boiler ash so that it can produce more silica content. From the results of Table. 4 obtained the contents of the elements of Nanoparticles such as Si 37,031 wt%, Fe

19,509 wt%, Al 16,520 wt% and Sn 13,765 wt%. In this study, the amount of silica was higher than the previous research [9] which is 31.45% silica and lime at 15.2%. But in the study [4] it was found that the higher silica content was 45.55%. In the table obtained the most Si content in 2.5 M NaOH Nanoparticles as much as 65.277% and the content which contains a lot of Fe that is without solution solution as much as 27.789%.

FTIR Nanoparticle Analysis

FTIR characterization using the Perkin Elmer spectrum one type FTIR device. This characterization aims to find out the functional groups of a 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 2 M NaOH OPBA nanoparticles shown in the spectra results in Figure 4

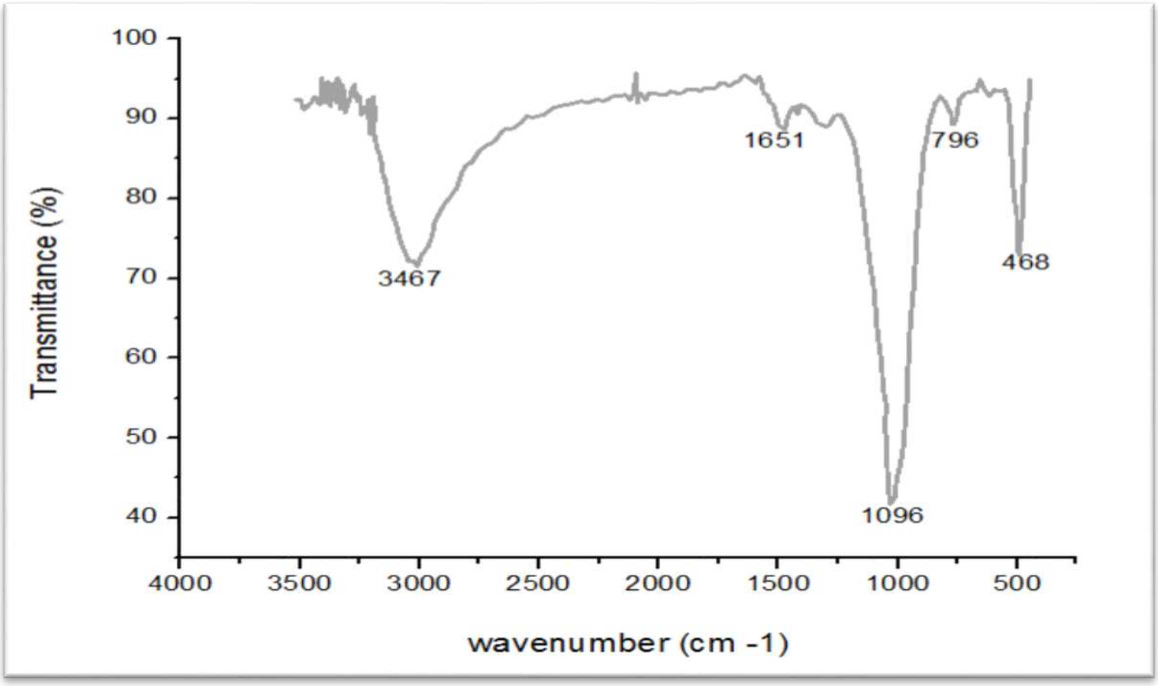


Fig. 5. FTIR reading results

FTIR testing shows absorption peaks from the sample. The peaks show the sample absorption groups which are the characters of the molecular vibrations of the sample. The peak numbers are the wave numbers (468, 796, 1096, 1651, 3467) cm-1.

In principle, FTIR is used to determine functional groups that exist in a compound, so that it can be used to determine a compound whose identity is unknown. In this study obtained the peak of wave numbers and functional groups found in Table 5.

Table 5. Table of Functional group and Wave Numbers of FTIR Results

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

OPBA analysis with FTIR shows the absorption group is 468 cm-1 which shows buckling vibration from Si-O-Si, peak wave number 796 cm-1 is a stretch vibration from C-Cl where the peak number between 850-550 is a stretch vibration from C-Cl, the peak of wave number 1096 cm-1 is the stretching vibration of Si-O, the peak of wave number 1651 cm-1 is the Si-O stretching vibration, the peak of wave number is 3467 cm-1 which shows the presence of OH group where the OH group is free to absorb strong in the area of 3550-3200 cm-1.. Several studies using FTIR (Fourier Transform Infra Red) have been conducted including observing functional groups on silica that obtained from rice husks. The results obtained show that the main peak associated with the silica functional group is the wave number 3444.6 cm-1 which is an O-H group (hydroxyl group) which indicates the presence of hydroxyl groups of hydrated water molecules . research shows the peak of wave number 1095.5 cm-1 that is the presence of Si-O-Si stretching vibration. And at the peak of 470.6 cm-1 number is Si-O stretching vibration.

4. CONCLUSION

The results of the characterization of XRD of palm oil boiler ash nanoparticles carried out using a ballmill and coprecipitation method showed that palm oil boiler ash particles can be said to be nanoparticles because they have particle sizes of (83.76; 72.3, 56.31) nm with quartz crystal types and trigonal crystal structure. Morphological characterization shows a homogeneous mixture,XRF shown the amount of silica is 31.45% . FTIR testing shows absorption peaks from which are the characters of the molecular vibrations of the sample. with the increase in the molarity of the NaOH solution, the particle size of the ash from the palm oil boiler decreases.

ACKNOWLEDGEMENTS

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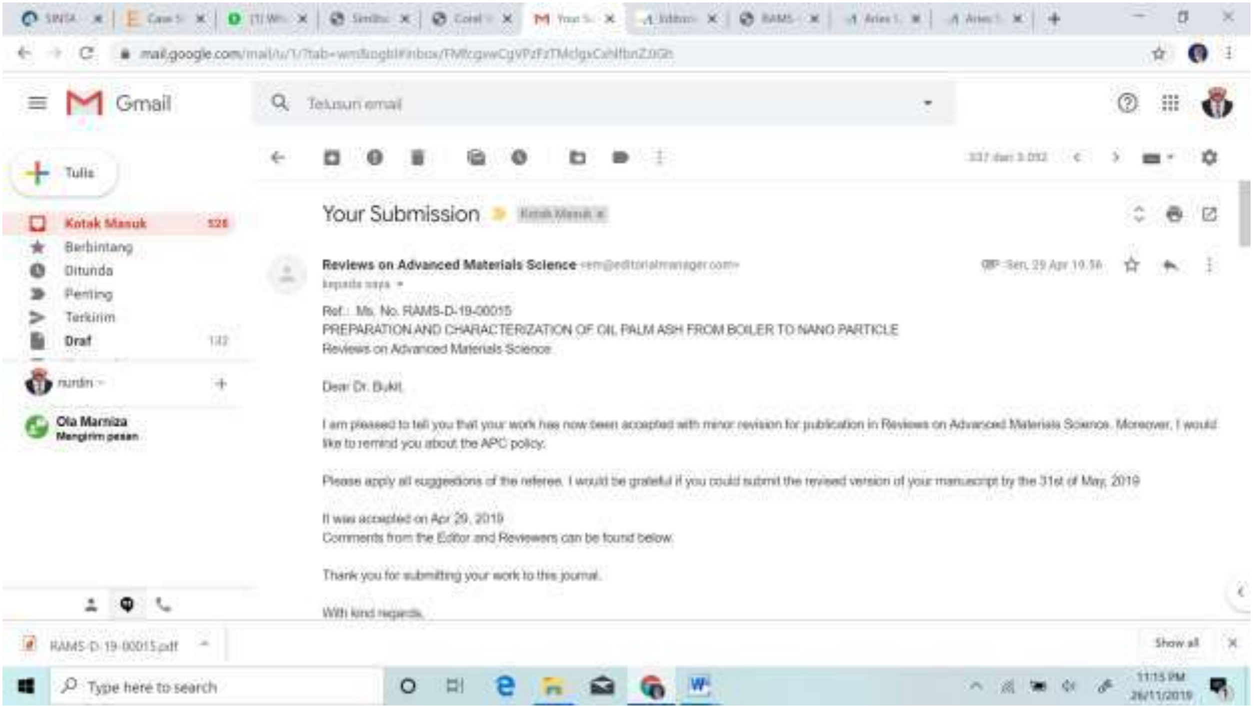


Your Submission

Reviews on Advanced Materials Science

Ref.: Ms. No. RAMS-D-19-00015
PREPARATION AND CHARACTERIZATION OF OIL PALM ASH FROM BOILER TO NANO PARTICLE
Reviews on Advanced Materials Science

Dear Dr. Bukit,

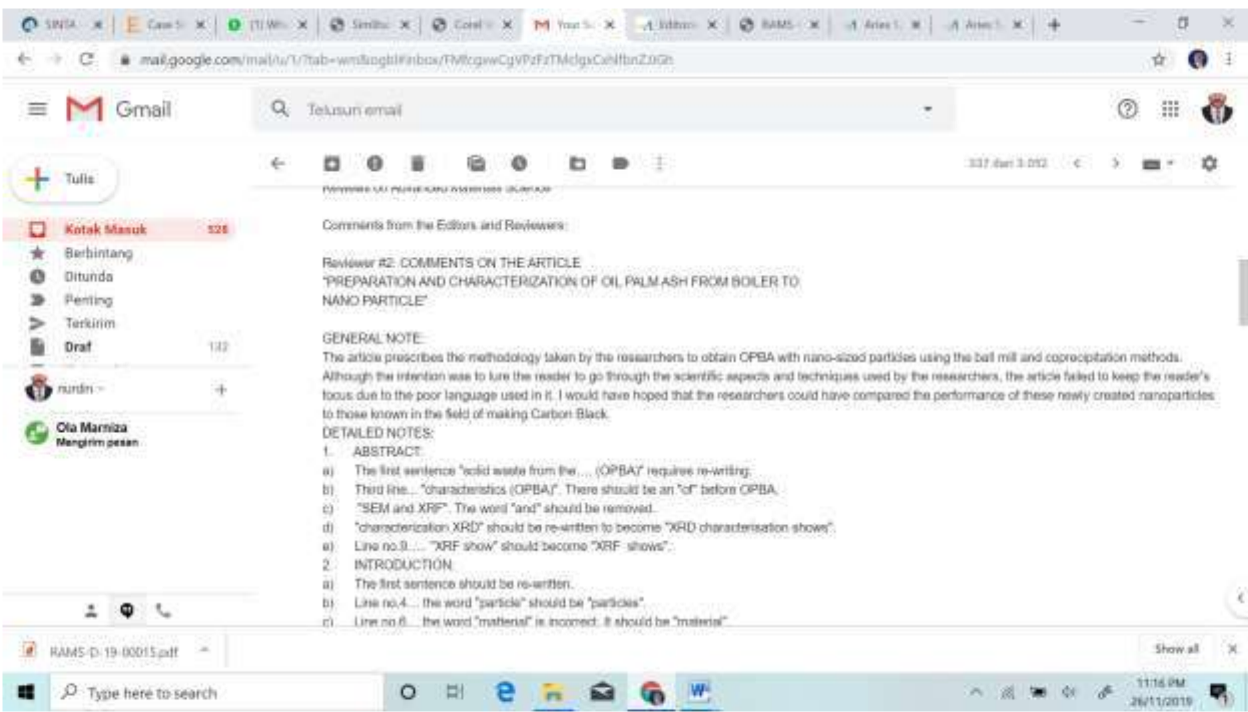
I am pleased to tell you that your work has now been accepted with minor revision for publication in Reviews on Advanced Materials Science. Moreover, I would like to remind you about the APC policy.

Please apply all suggestions of the referee. I would be grateful if you could submit the revised version of your manuscript by the 31st of May, 2019

It was accepted on Apr 29, 2019
Comments from the Editor and Reviewers can be found below.

Thank you for submitting your work to this journal.

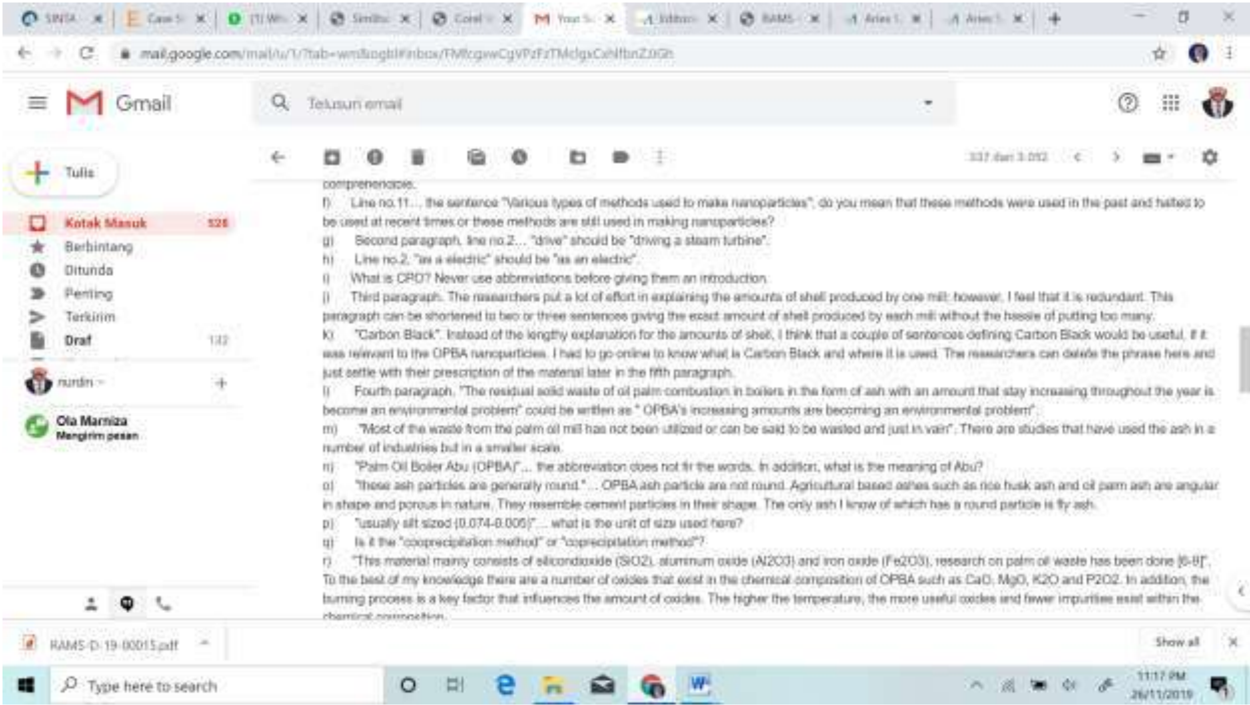
With kind regards,



Comments from the Editors and Reviewers:

Reviewer #2: COMMENTS ON THE ARTICLE
"PREPARATION AND CHARACTERIZATION OF OIL PALM ASH FROM BOILER TO NANO PARTICLE"

GENERAL NOTE:
The article prescribes the methodology taken by the researchers to obtain OPBA with nano-sized particles using the ball mill and coprecipitation methods. Although the intention was to lure the reader to go through the scientific aspects and techniques used by the researchers, the article failed to keep the reader's focus due to the poor language used in it. I would have hoped that the researchers could have compared the performance of these newly created nanoparticles to those known in the field of making Carbon Black.
DETAILED NOTES:
1. ABSTRACT:
a) The first sentence "solid waste from the.... (OPBA)" requires re-writing.
b) Third line... "characteristics (OPBA)". There should be an "of" before OPBA.
c) "SEM and XRF". The word "and" should be removed.
d) "characterization XRD" should be re-written to become "XRD characterisation shows".
e) Line no.9..... "XRF show" should become "XRF shows".
2. INTRODUCTION
a) The first sentence should be re-written.
b) Line no.4... the word "particle" should be "particles".
c) Line no.6... the word "matterial" is incorrect. It should be "material".



comprehendable.

f) Line no.11… the sentence "Various types of methods used to make nanoparticles", do you mean that these methods were used in the past and halted to be used at recent times or these methods are still used in making nanoparticles?

g) Second paragraph, line no.2… "drive" should be "driving a steam turbine".

h) Line no.2, "as a electric" should be "as an electric".

i) What is CPO? Never use abbreviations before giving them an introduction.

j) Third paragraph. The researchers put a lot of effort in explaining the amounts of shell produced by one mill; however, I feel that it is redundant. This paragraph can be shortened to two or three sentences giving the exact amount of shell produced by each mill without the hassle of putting too many.

k) "Carbon Black". Instead of the lengthy explanation for the amounts of shell, I think that a couple of sentences defining Carbon Black would be useful, if it was relevant to the OPBA nanoparticles. I had to go online to know what is Carbon Black and where it is used. The researchers can delete the phrase here and just settle with their prescription of the material later in the fifth paragraph.

l) Fourth paragraph. "The residual solid waste of oil palm combustion in boilers in the form of ash with an amount that stay increasing throughout the year is become an environmental problem" could be written as " OPBA's increasing amounts are becoming an environmental problem".

m) "Most of the waste from the palm oil mill has not been utilized or can be said to be wasted and just in vain". There are studies that have used the ash in a number of industries but in a smaller scale.

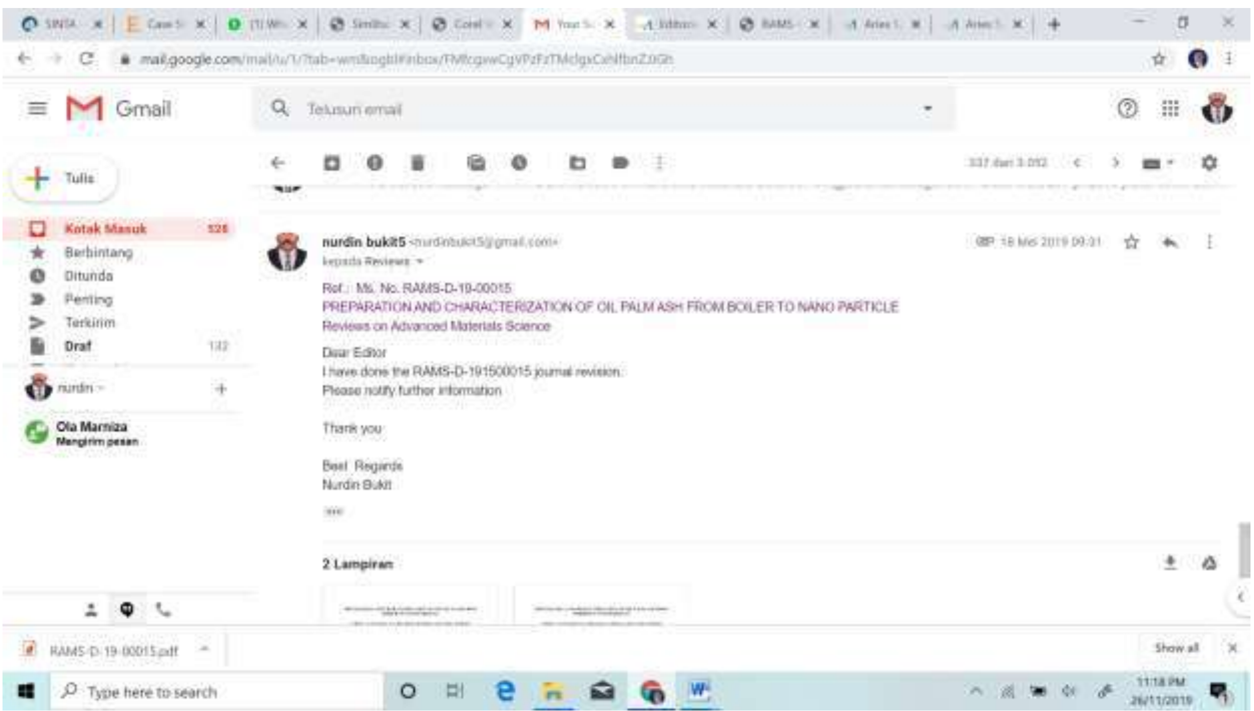
n) "Palm Oil Boiler Abu (OPBA)"… the abbreviation does not fit the words. In addition, what is the meaning of Abu?

o) "these ash particles are generally round."… OPBA ash particle are not round. Agricultural based ashes such as rice husk ash and oil palm ash are angular in shape and porous in nature. They resemble cement particles in their shape. The only ash I know of which has a round particle is fly ash.

p) "usually silt sized (0.074-0.005)"… what is the unit of size used here?

q) Is it the "coprecipitation method" or "copreciptation method"?

r) "This material mainly consists of silicondioxide (SiO2), aluminum oxide (Al2O3) and iron oxide (Fe2O3), research on palm oil waste has been done [6-8]". To the best of my knowledge there are a number of oxides that exist in the chemical composition of OPBA such as CaO, MgO, K2O and P2O2. In addition, the burning process is a key factor that influences the amount of oxides. The higher the temperature, the more useful oxides and fewer impurities exist within the chemical composition.



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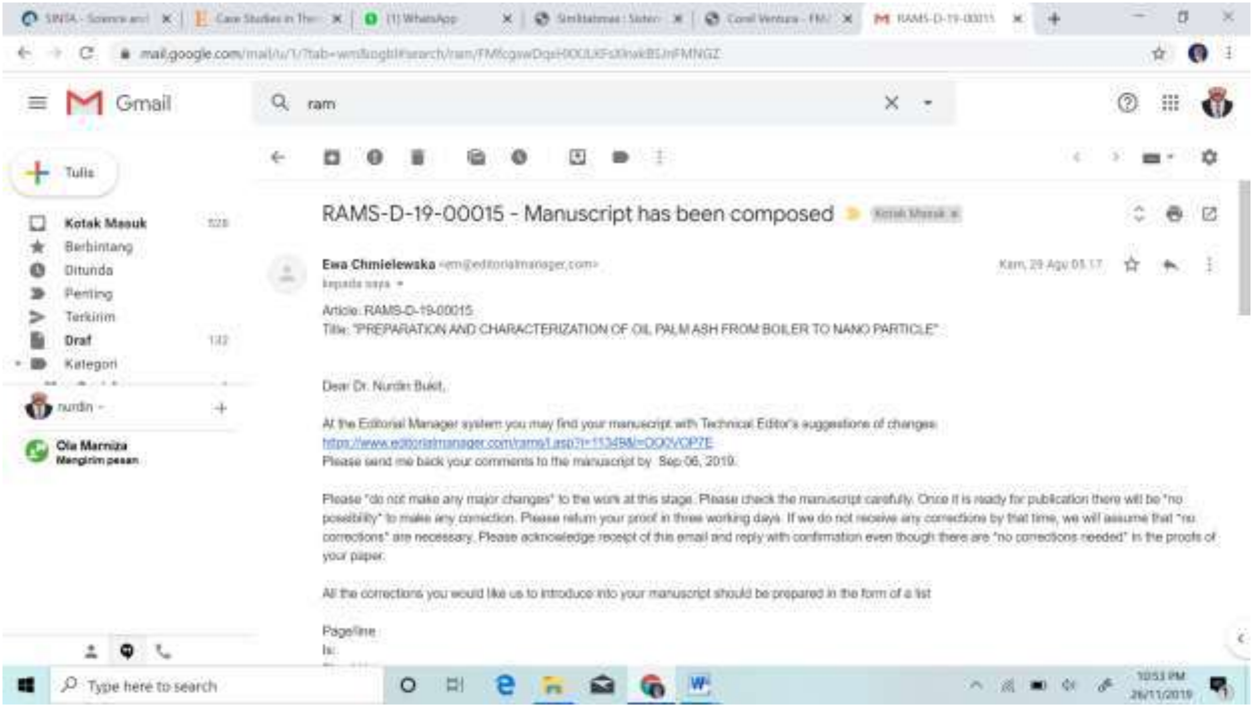

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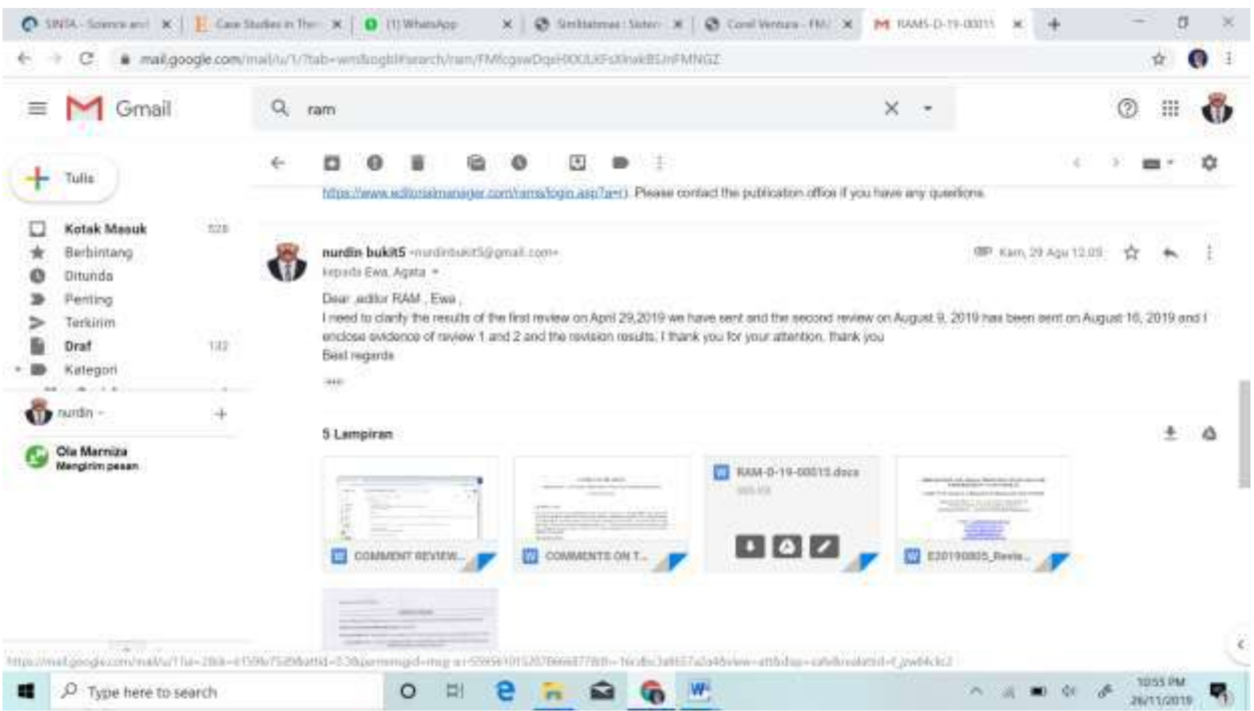

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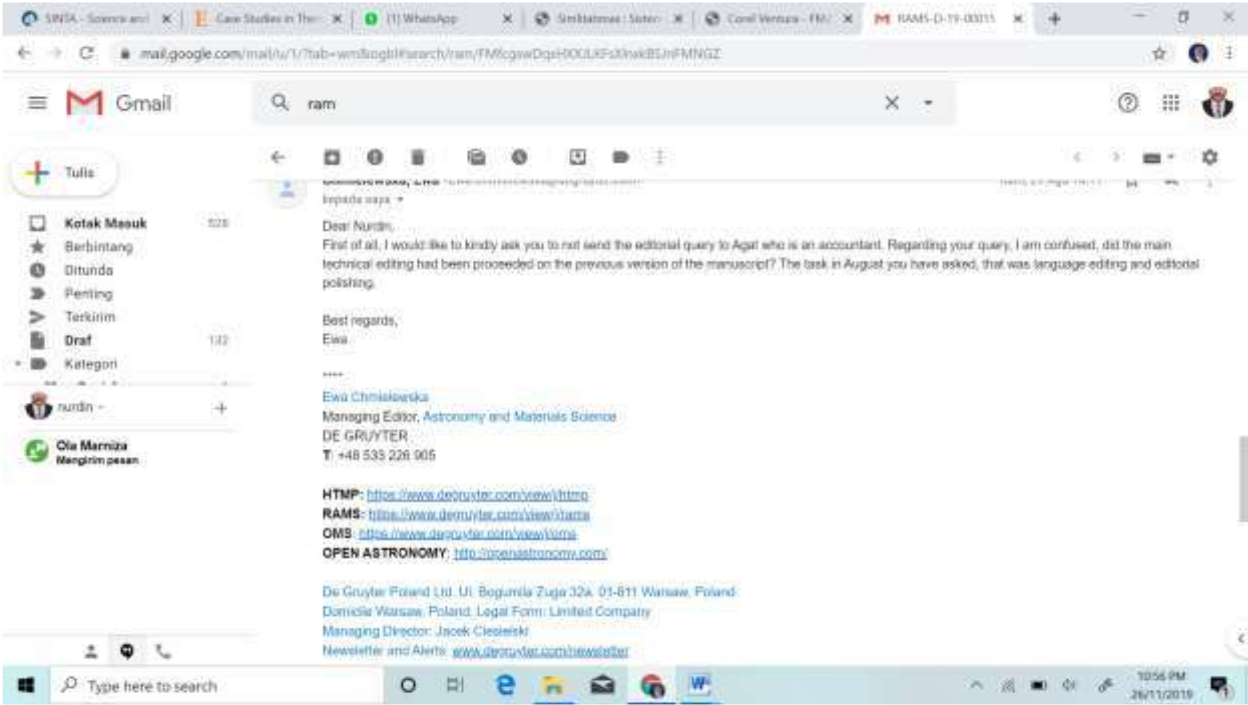

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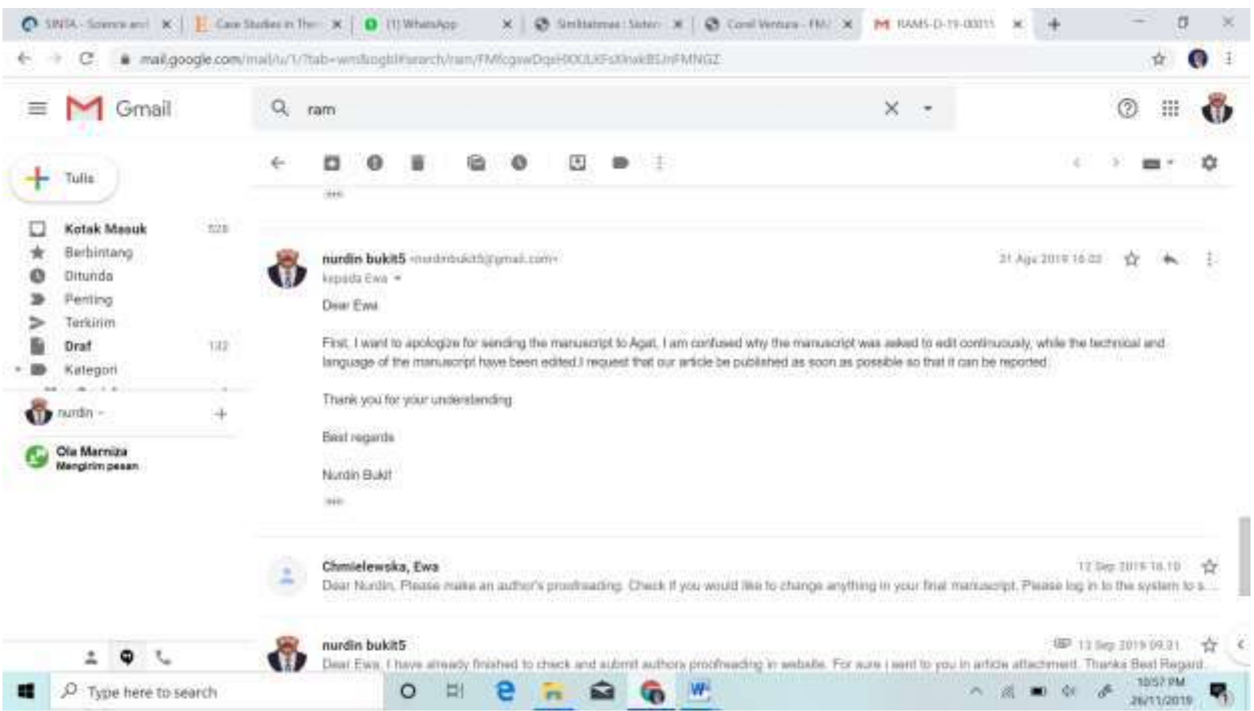

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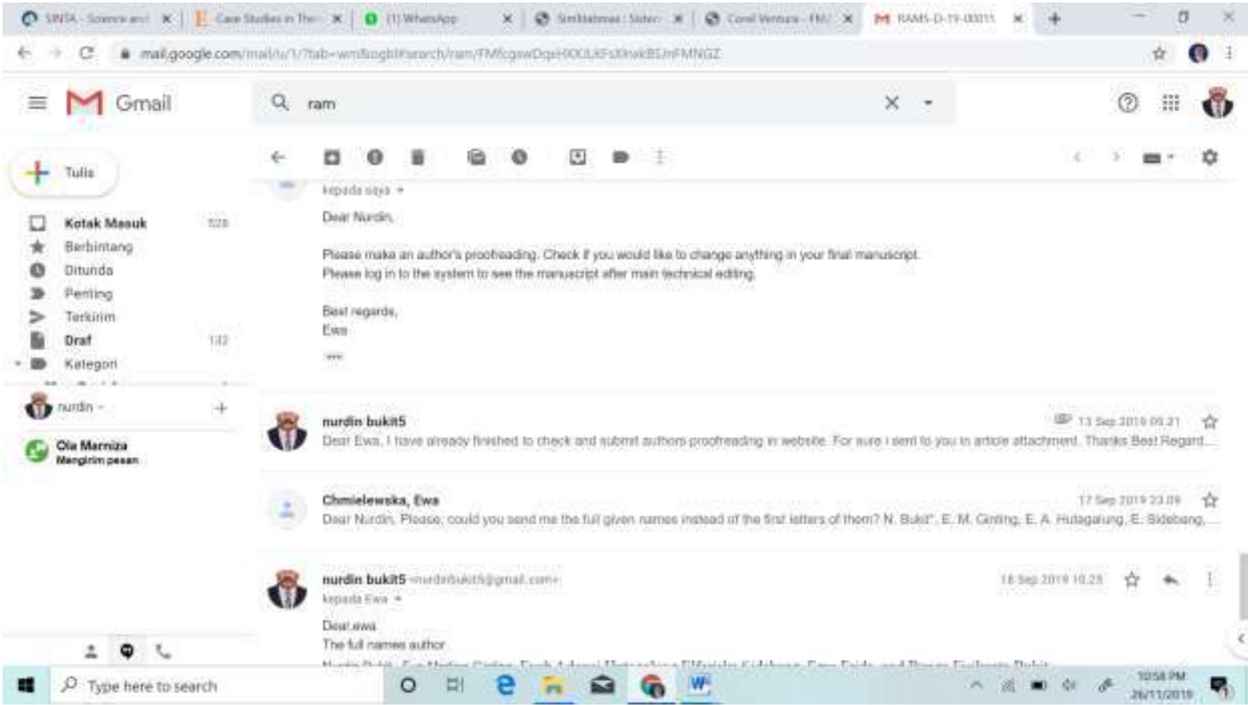

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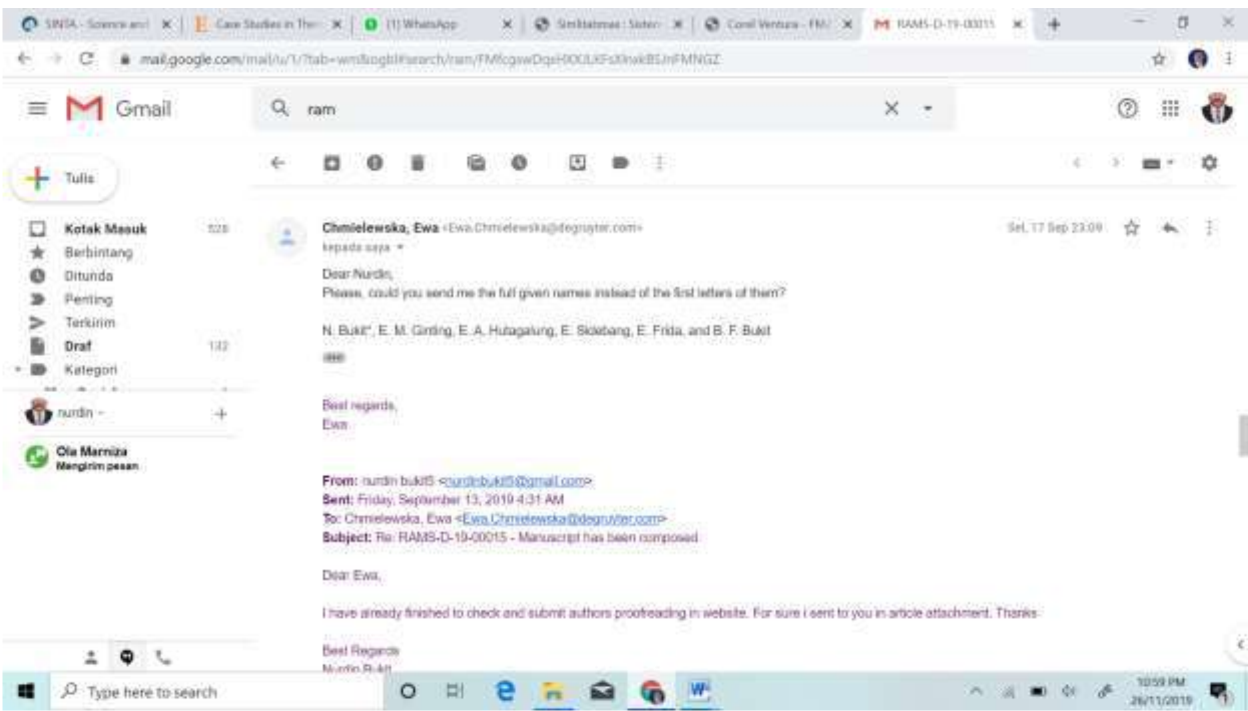

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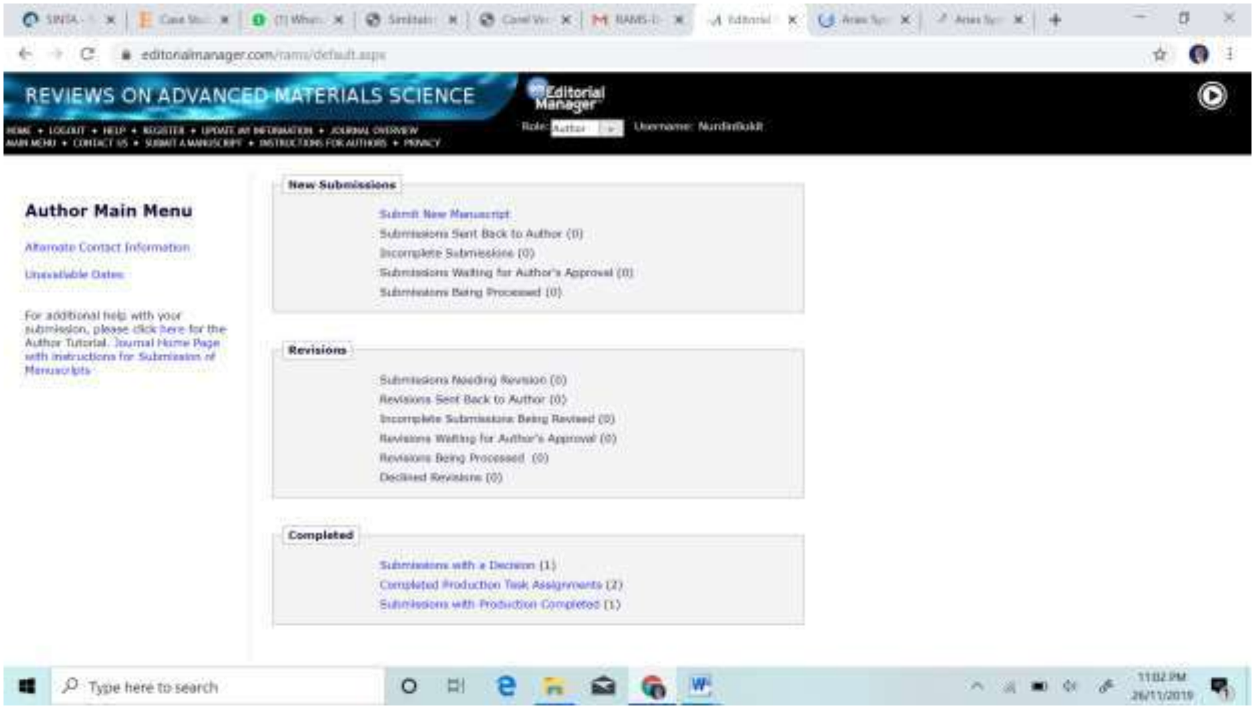
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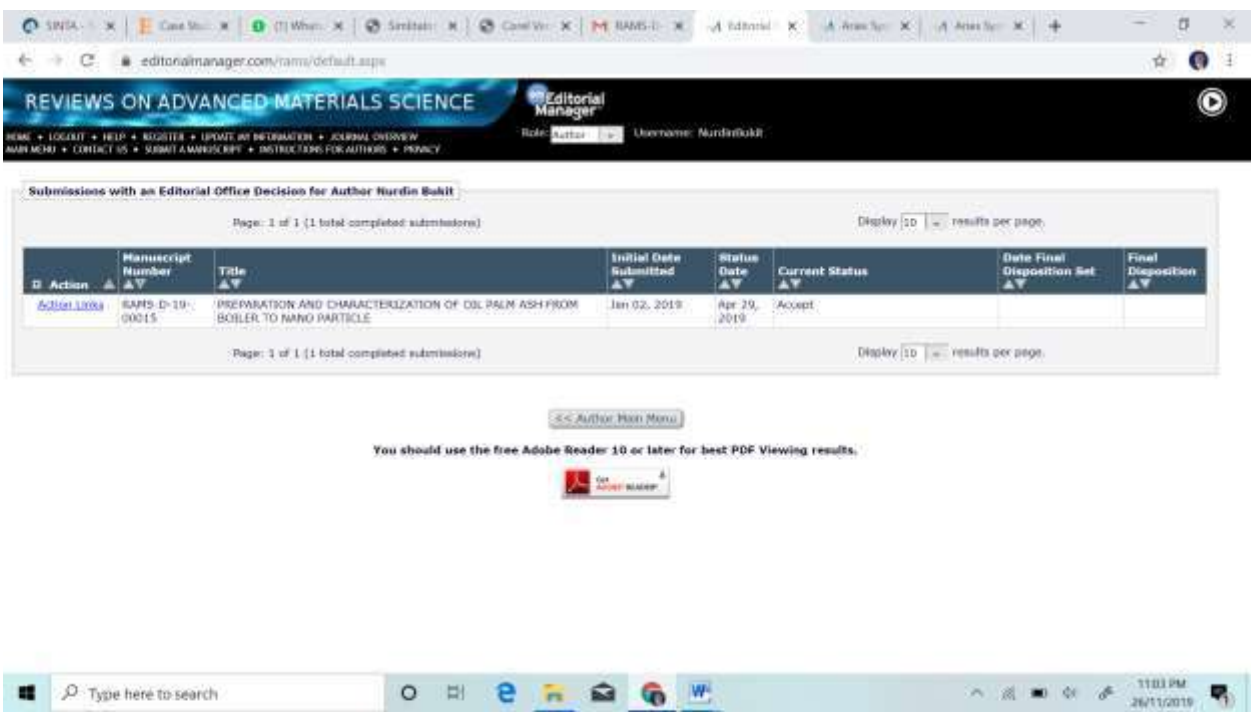
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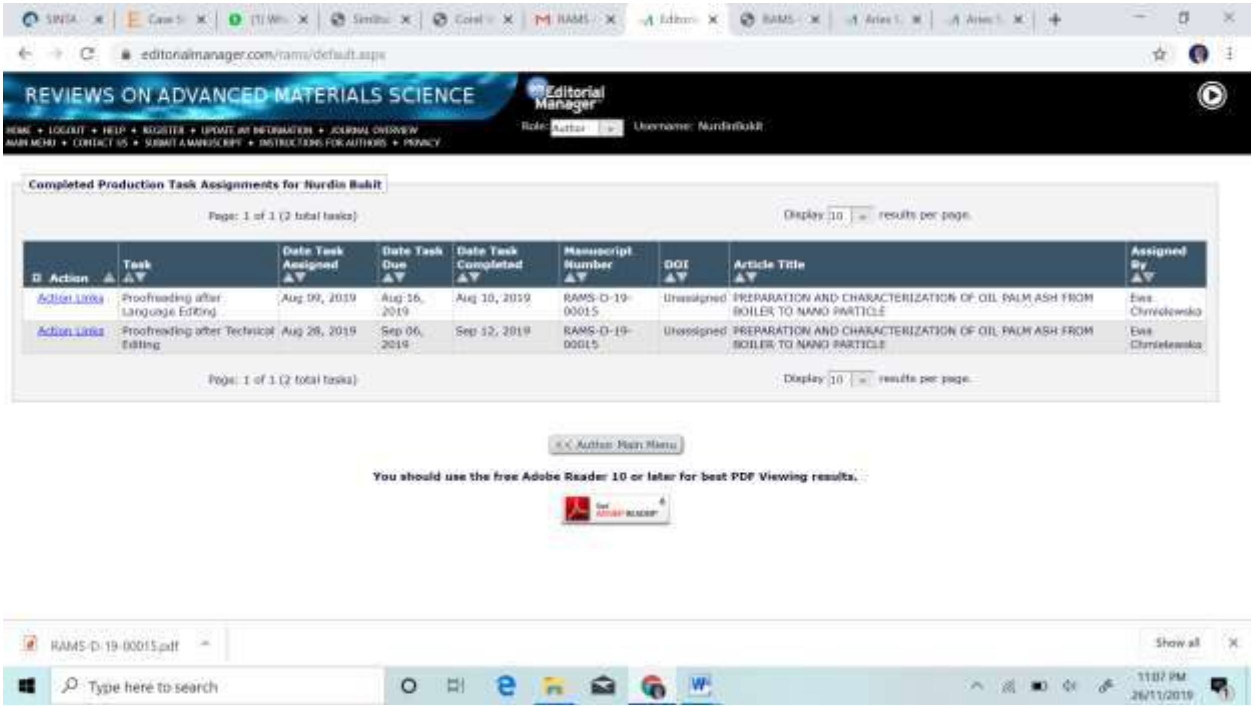

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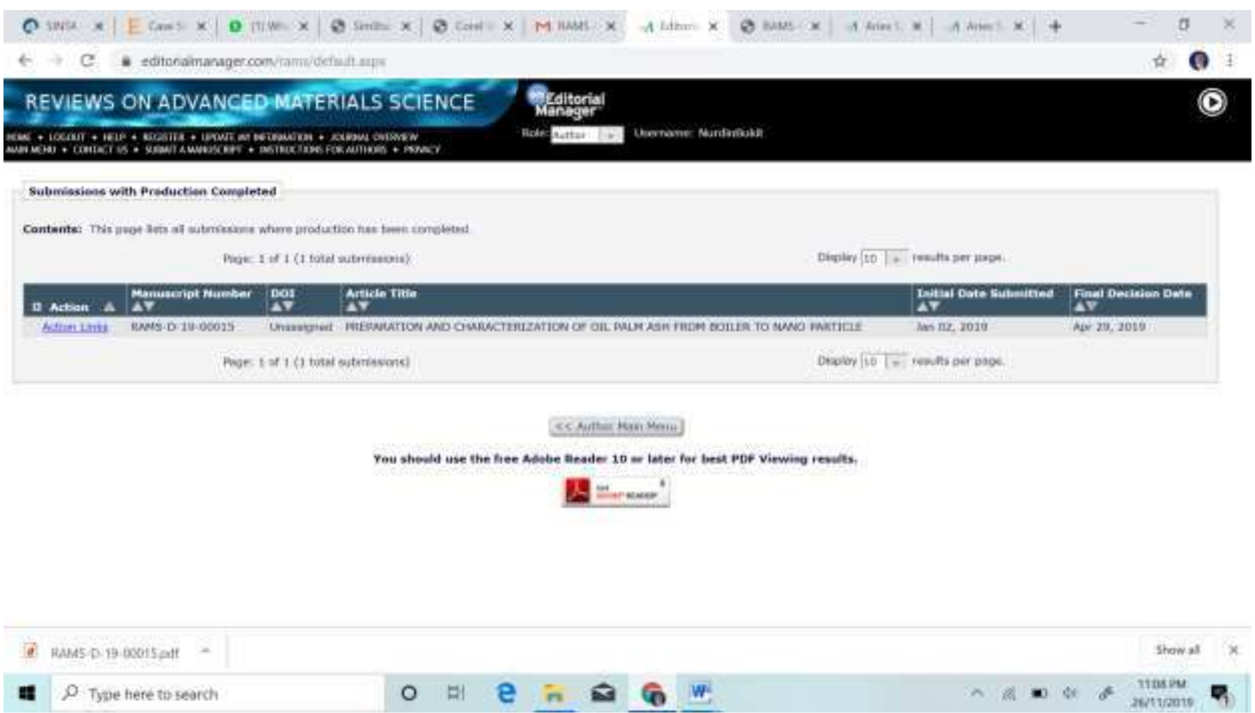

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Research Article

N. Bukit*, E. M. Ginting, E. A. Hutagalung, E. Sidebang, E. Frida, and B. F. Bukit

Preparation and characterization of oil palm ash from boiler to nanoparticle

https://doi.org/10.1515/rams-2019-0023
Received Jan 02, 2019; accepted Apr 29, 2019

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 (SiO2) 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 compress precipitation method has been widely carried out, among others [17–22].

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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, $2\theta = 5°$ 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

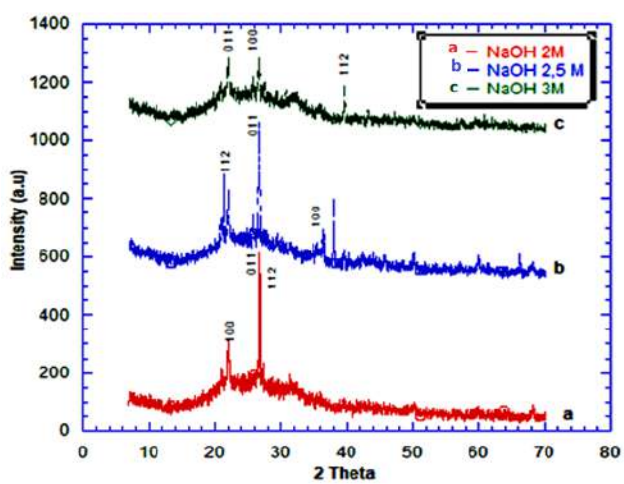

Figure 1: OPBA diffraction pattern with variation of NaOH solution

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

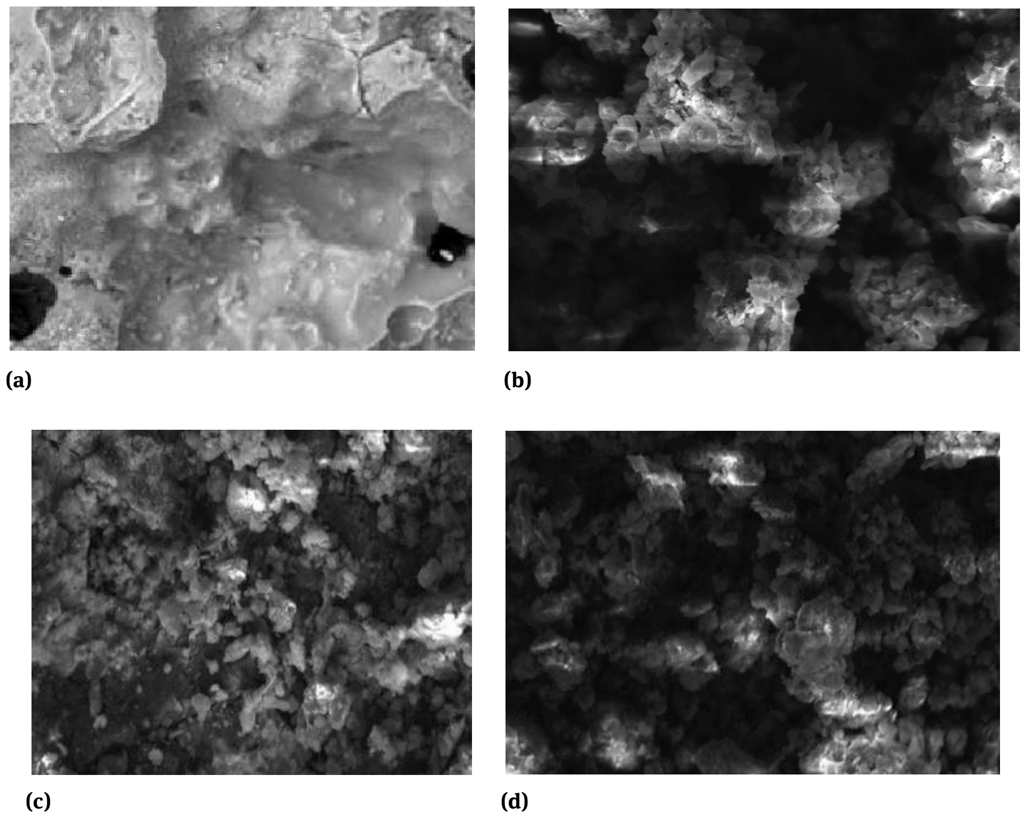

Figure 2: Morphology a. Pure OPBA ,b .OPBA with NaOH 2M c .OPBA with NaOH 2,5M d.OPBA with NaOH 3M

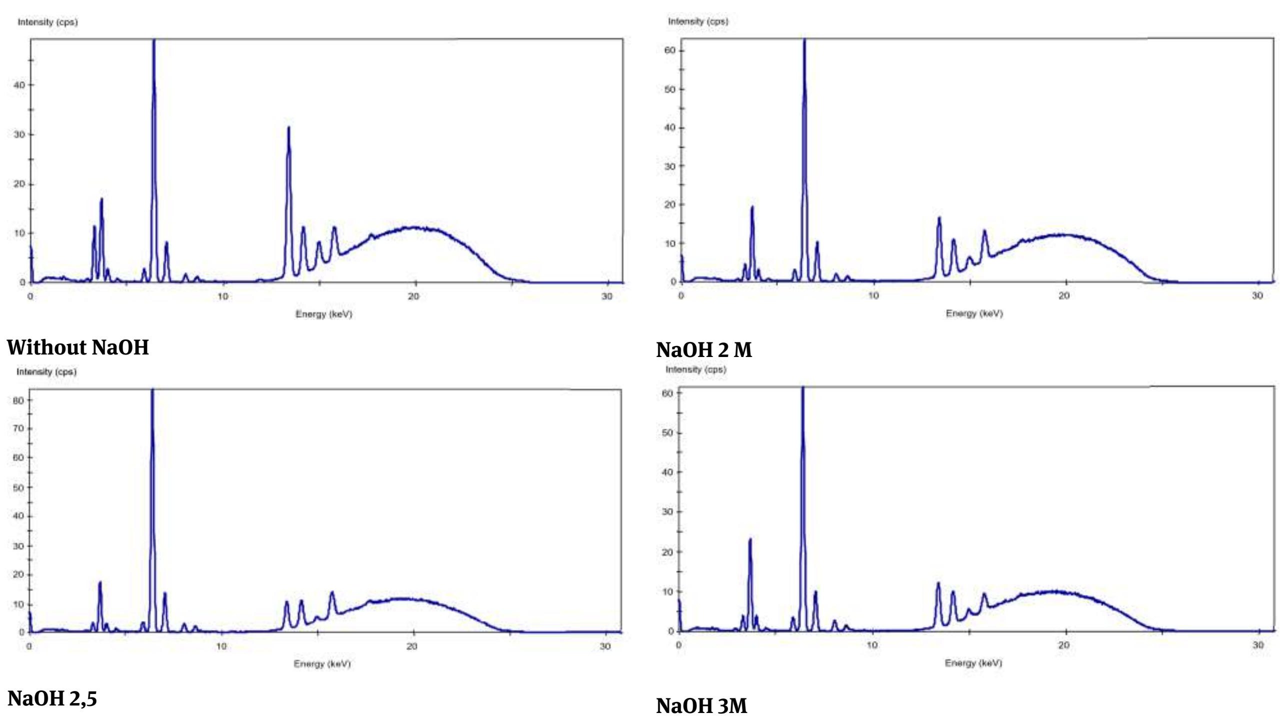

Figure 3: OPBA nanoparticle XRF analysis

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.*, 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}.

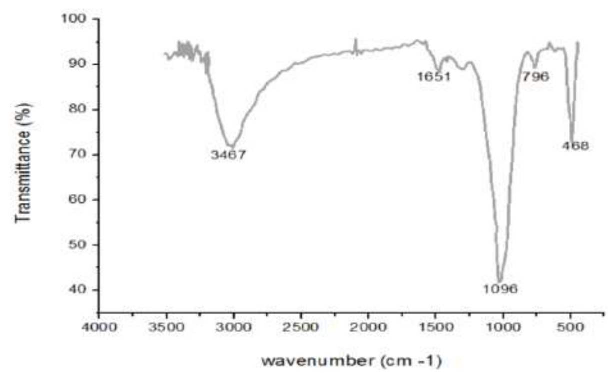

Figure 4: FTIR characterization graph

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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