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Characterization of Kaolin Nanoparticles for Biodiesel Catalyst

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Abstract: Currently, the use of nanotechnology is increasing rapidly in all aspects of life. This study uses natural materials, namely kaolin. Kaolin has a composition of (Al2O3.2SiO2.2H2O), rich in kaolinite minerals because it is composed of kaolinite (85-95%). Kaolin particles are in the form of hexagonal sheets consisting of layers of tetrahedral silica and octahedral alumina. This content makes kaolin suitable for use as a heterogeneous catalyst in the manufacture of biodiesel production. The manufacture of kaolin catalysts is carried out by a calcination process at high temperatures to form metakaolin. In order for the reaction time to be faster and produce higher conversion, the particle size is reduced to nanoparticle size. The nanoparticle process is carried out with energy using Ball Milling High Energy Milling Ellipse 3D Motion. After ball milling the kaolin sample before and after the calcination process were analyzed using a particle size analyzer (PSA), then used as a kaolin catalyst and the optimum value of the % yield of biodiesel was tested by X-ray diffraction (XRD) and Scanning electron microscopy- Energy dispersive X-ray Spectroscopy (SEM-EDX) to see the chemical composition and particle size for all specimens. Based on XRD data and morphological analysis using SEM, the calcination process causes changes in the structure of kaolin from pseudohexagonal layered to an amorphous phase. rom this analysis, new developments in nano materials can be achieved and can be utilized especially for biodiesel production. The smaller the catalyst particles, the faster they will react during the production process biodiesel. Biodiesel uses used cooking oil as raw material with methanol. The %FFA content in used cooking oil is more than 5% so that in this biodiesel process it uses esterification and transesterification processes. The optimum %yield of biodiesel is 98.48%.

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I. INTRODUCTION

Development of particle size from macron to micron appeared in the early 1960s and smaller particle-based technology has developed a lot and provided benefits to humans. Since then, humans have tried to improve and enhance technology by creating materials that are less than micro or called nano size. The idea came from Sobolev to reduce and create smaller sizes. From the nano size it was introduced to the world until it developed until now (Fadzil & Nurhasri, 2017).

The catalyst used comes from kaolin, kaolin is a type of *clay* mineral, which have composition ($Al_2O_3.2SiO_2.2H_2O$). Kaolin is composed of kaolinite (85-95%) (Purbasari & Walmiki, 2021). Kaolin is also an aluminosilicate containing silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), a small amount of iron (Fe_2O_3) and titanium (TiO_2). Kaolin particles are in the form of hexagonal sheets consisting of layers of tetrahedral silica and octahedral alumina (Sunardi et al., 1999). Each of these layers is joined through oxygen atoms alternately into one unit through hydrogen bonds between oxygen from silica and hydroxyl oxygen from alumina (Fadzil & Nurhasri, 2017). This content makes kaolin suitable for use as a heterogeneous catalyst in the manufacture of biodiesel production (Dang et al., 2013). The manufacture of kaolin catalysts is carried out by a calcination process at high temperatures to form metakaolin (Satriyo, 2014). The purpose of developing kaolin in calcination for heterogeneous catalysts. The performance of this heterogeneous catalytic activity can be improved by engineering particle size through a nanotechnology approach (Wahyuningsih et al., 2018). Innovation of heterogeneous catalysts on a nanoparticle scale is expected to reduce production costs and accelerate the reaction. Engineering the catalyst particle size in the nanometer range can be done with a top-down approach. Top-down contruction using ball mill (Dwi, 2020). The selected ball milling technology uses High Energy Milling Ellipse 3D Motion (HEM-E3D) ball milling technology. High Energy Milling Ellipse 3D Motion Ball Milling Technology is a rotary motion technology that utilizes the impact energy between the crushing ball and the chamber wall. With this tool, it can easily make kaolin particles in the form of nanoparticles. Furthermore, the use of nanoparticle-sized

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catalysts produces a more reactive surface and a lower reaction temperature (Yuni et al., 2016). Therefore, in this study, the aim is to analyze the structural properties of nanoparticles before and after calcination. In addition, after calcination of kaolin that has undergone atomization treatment, its characteristics were evaluated by X-ray diffraction (XRD) and scanning electron microscopy (SEM) testing to see the chemical composition and particle size for all specimens. Previously, particle size analysis (PSA) was a tool for testing the size distribution of nanometer-sized particles. Furthermore, the calcined kaolin nanoparticle catalyst was then tested for catalytic activity in biodiesel production using waste cooking oil as the raw material.

Biodiesel is a mixture of long-chain fatty acid methyl esters, which are mostly produced through the esterification of free fatty acids or transesterification of animal fats, vegetable oils, even used cooking oil with short-chain alcohols, usually using methanol or ethanol in the presence of a suitable catalyst (Dang et al., 2013). Methyl or ethyl ester is a relatively stable compound, liquid at room temperature (melting point 4°C to 18°C), non-corrosive, and has a low boiling point. The process of making biodiesel generally involves two reactions namely esterification and transesterification reactions. Both use methanol as a reactant. Esterification is usually carried out before transesterification if the raw material used contains high free fatty acids with %FFA above 5% (Jaya et al., 2022). The esterification process is used to removes free fatty acids and produces, additional esters. For to encourage the reaction to proceed to perfect conversion at low temperatures, the methanol reactant must be added in greater quantities and the product water bound to the reaction is removed from the reaction phase, namely into the oil phase (Ibrahim et al., 2020). Retested %FFA from the esterification reaction. If the %FFA produced is less than 5% then process to the transesterification reaction. The transesterification reaction is a process in which triglycerides in vegetable oils or animal fats reaction with short-chain alcohols such as methanol or ethanol to produce biodiesel methyl esters and glycerol as by-product. Here, thedecomposition of triglyceride coumpounds and the migration of alkyl groups between esters coumpounds occur. The ester produced from this transesterification reaction is called biodiesel (Muhammad et al., 2017). The triglyceride reaction does not occur all at once, but through steps that convert triglycerides into diglycerides, diglycerides into monoglycerides, and monoglycerides into alkyl esters. The results of kaolin nanoparticle catalysts in biodiesel production are seen from the yield of biodiesel produced.

II. RESEARCH METHODOLOGY

A. Research Design

The research design data was analyzed using the Response Surface Methodology (RSM) application by selecting Standard Design 3**(K-p) and Box-Behnken Designs, Number of factor dipilih 3, Number of blocks 1, Number of runs 27 and Number of center Point 3 to determine the optimal conditions obtained 30 experimental data. This optimization process includes three stage, namely conducting experiments that statistically designed, estimating coefficients in mathematical models and predict the response by checking the suitability of the model (Box and Hunter, 1957; Box et al., 1978). The research design was used for the biodiesel production process with kaolin nanoparticle catalysts. To determine the optimum conditions resulting from the %yield of biodiesel. The independent variables selected include the collision time on the HEM-E3D tool, the reaction time in the transesterifocation process and the molar ratio of methanol.

B. Tools and Materials

The tools used include: Ball Miling High Energy Milling Ellipse 3D Motion (HEM 3D Motion), Furnace, Strier and hotplate, oven, PSA Horiba SZ 100z, X-ray Diffraction Shimadzu XRD-7000, JEOL Benchtop Scanning Electron Microscopy JCM 7000, and chemical glassware.

Materials used include: used cooking oil comes from a food stall in the Ngesrep Tembalang area of Semarang City, kaolin comes from Tanjong Pandan Belitung with an initial size of 325 mesh, 98% methanol, isopropanol, pp indicator, H2SO4, NaOH 0,1 N, aquadest, and alumunium foil.

C. Preparation of Kaolin Nanoparticle Catalyst

Kaolin was prepared by calcination at 600°C for 6 hours. But before calcination, kaolin is made into nanoparticle particle size using high energy milling ellipse 3D motion ball milling technology. How HEM 3D Motion works is the first step to clean the ball jar with a cloth soaked in alcohol. Then insert up to 50 grams of large or small ball jar and 5 grams of kaolin sample with a weight ratio of ball jar to sample of 10:1. Put it in a clean tube and close the tube tightly. Attach the tube to the high energy milling ellipse 3D motion ball milling technology milling using the L key until tight. After the tube is securely installed in the machine, the ball mill is turned on and set the time to 60 minutes, the OFF time to 10 minutes, and also set the milling time to 1 hour, 3 hours and 5 hours. Milling by grinding the material from powder form into a smaller size causes a material to experience particle size breakdown (Nugraha et.al., 2015). The kaolin results from the high energy milling ellipse 3D motion ball milling tool are then calcined with a furnace. Kaolin nanoparticles were taken ±4 grams for thermal activation by calcination process at 600°C for 6 hours with a temperature gradient of 10°C per minute from room temperature to calcination temperature. After calcination, kaolin particles were measured with particle size analyzer (PSA). The kaolin result after calcination process is called metakaolin. The result of kaolin nanoparticle catalyst is used as a catalyst in biodiesel production.

D. Preparation of Raw Materials for Used Cooking Oil

100 ml of used cooking oil is put into a threenecked flask, and 100 ml of distilled water was added with a ratio of 1:1. Then heat at 100°C for 3 hours until the water becomes half the original volume. The oil-water mixture was separated in a separating funnel for 1 hour. After 1 hour, two phases were formed, the upper phase is oil and the lower phase is water. The oil phase is taken and the water phase at the bottom is removed. The pretreated oil *is* filtered from impurities and reheated to 100°C so that the oil is anhydrous. The processed

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used cooking oil is analyzed for free fatty acids contained in it. The free fatty acid content is tested using the acid-base titration method according to the standard AOCS (American Oil Chemistry Society).

E. Biodiesel Production with Kaolin Nanoparticle Catalyst

Biodiesel production uses used cooking oil as the raw material. The %FFA result of used cooking oil as the raw material is 16.69% more than 5% so that the biodiesel production process goes through two reaction processes, namely esterification reaction and transesterification reaction. Because the raw material for biodiesel used is used cooking oil, the FFA content of used cooking oil is above 5%, so it needs to go through an esterification process first. So if the FFA content exceeds 5%, an esterification process is needed before the transesterification process in biodiesel production. The biodiesel production process begins with the esterification process by mixing 44.4 ml of methanol with 100 ml of used cooking oil. 2.5 mL of H_2SO_4 catalyst was then added to the mixture of methanol and used cooking oil. This stirring was carried out at a stirring speed of 700 rpm and a working temperature of 65°C for 60 minutes. In addition, the products of this reaction were separated from the catalyst using a separating funnel to form three layers. The methyl ester layer in the middle is removed by adding hot water at a temperature of 60-80°C added to the methyl ester layer, then the bottom layer of water is removed. Then discard the bottom water until the washing is repeated 2-3 times with hot water of 60°C-80°C until the water becomes clear. The methyl ester results from the esterification process are then re-tested for %FFA content. Until the %FFA value is less than 5% then it can continued to the transesterification process. In addition, the transesterification process takes place where the methyl ester obtained from the esterification process is processed and added with 3% kaolin nanoparticle catalyst with a molar variation of methanol: methyl ester from the esterification process, namely (25:1); (30:1); and (35:1) each placed in a three-necked flask and reacted on a hot plate stirrer at a temperature of 65 °C during time variations, namely 30 minutes, 60 minutes, and 90 minutes. After the process is complete, pour it into a separatory funnel and let it stand for 1 hour. The product of this reaction is thn separated from the catalyst using a separatory funnel to form two layers. The bottom glycerol layer is removed with hot water at a temperature of 60°C to 80°C added to the top methyl ester layer. The water layer at the bottom and the remaining glycerin are removed. Repeat washing 2-3 times with warm water at 60°C-80°C until the water is clear. Methyl ester produced from the transesterification process is heated at 120°C for 30 minutes to reduce water content.

III. RESULTS AND DISCUSSION

A. Particle size analyzer PSA Characteristics

Particle size analysis was tested with a particle size analyzer (PSA) where the particles in the sample can be measured and the distribution of representative samples. Determination of particle size and distribution using a particle size analyzer can be done using the method dynamic lights scattering (DLS) (Pasi et al., 2020). Particles can be measuredin the range of 0.3 nm to more than 3 mm, the size distribution of the resulting kaolin nanoparticles is in the nanometer range. Based on the results in table 3.1 where with variations in milling time, namely 1 hour, 3 hours and 5 hours with the high energy milling ellips 3D (HEM-E3D) technique, the results of the particle size analyzer (PSA) measurements are as follows:

Table 1 Particle Size Analyzer Results Kaolin Catalyst

No.	Sampel	Ukuran S.D (nm)	Rata-Rata Ukuran/Mean (nm)	Z average (nm)
1	Waktu Milling 1 hours	280,0	1478,1	988,8
2	Waktu Milling 3 hours	258,0	1405,7	984,1
3	Waktu Milling 5 hours	195,4	1034,1	651,6

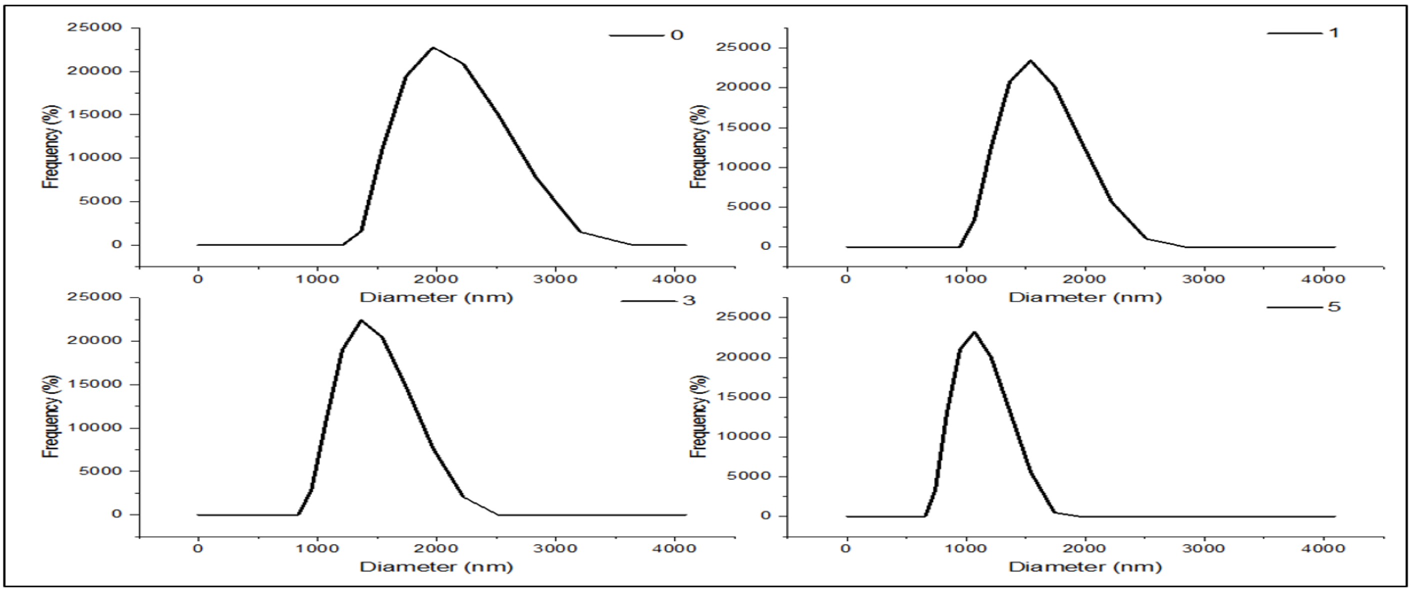

Fig 1 Graph of PSA Results Against Milling Time

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The size of kaolin particles decreases with increasing milling time in high energy milling ball milling technology. The results of figure 3.1 The graph decreases with increasing time. At 5 hours of milling, the particle size results produced Particle size analyzer Z average 651.6 nm, with an average particle size of 1034.1 nm and SD size 195.4 nm. This size is still large, this is likely because small particles stick together very high. Therefore, the resulting particle size is larger than expected due to agglomeration. Agglomeration describes the tendency of particles small in suspension to combine into larger aggregates. The aggregation process stops when secondary particle stability is achieved. This agglomeration process increases the size of the resulting particles (secondary particles). The size of these secondary particles is read by the Particle size analyzer. The standard deviation indicates the accuracy of the method being investigated. A lower standard deviation value indicates higher precision. Standard deviation is also related to the particle size distribution, the smaller the standard deviation value, the narrower the particle size distribution (Rasy et al., 2019). From the best particle size results, the longer the collision time, the smaller the particles produced, namely at a collision time of 5 hours.

B. Results of Analysis %yield Biodiesel with Kaolin Nanoparticle Catalyst

Characteristics of biodiesel using kaolin nanoparticle catalyst can be done by analyzing the %yield biodiesel. There are %yield biodiesel from 30 experiments using response surface methodology where the independent variables entered are milling time, reaction time and methanol molar ratio. The optimum condition was obtained in the sample 27, which produced biodiesel of %yield biodiesel 98,48%. The independent variables in optimum conditions are collision time on the HEM-E3D device at 5 hours, then the transesterification reaction time in the range of 80-90 minutes and the molar ratio of methanol and waste cooking oil at a ratio of (35:1). From these data, a response fitted surface methodology graph can be made in Figure 3.2.

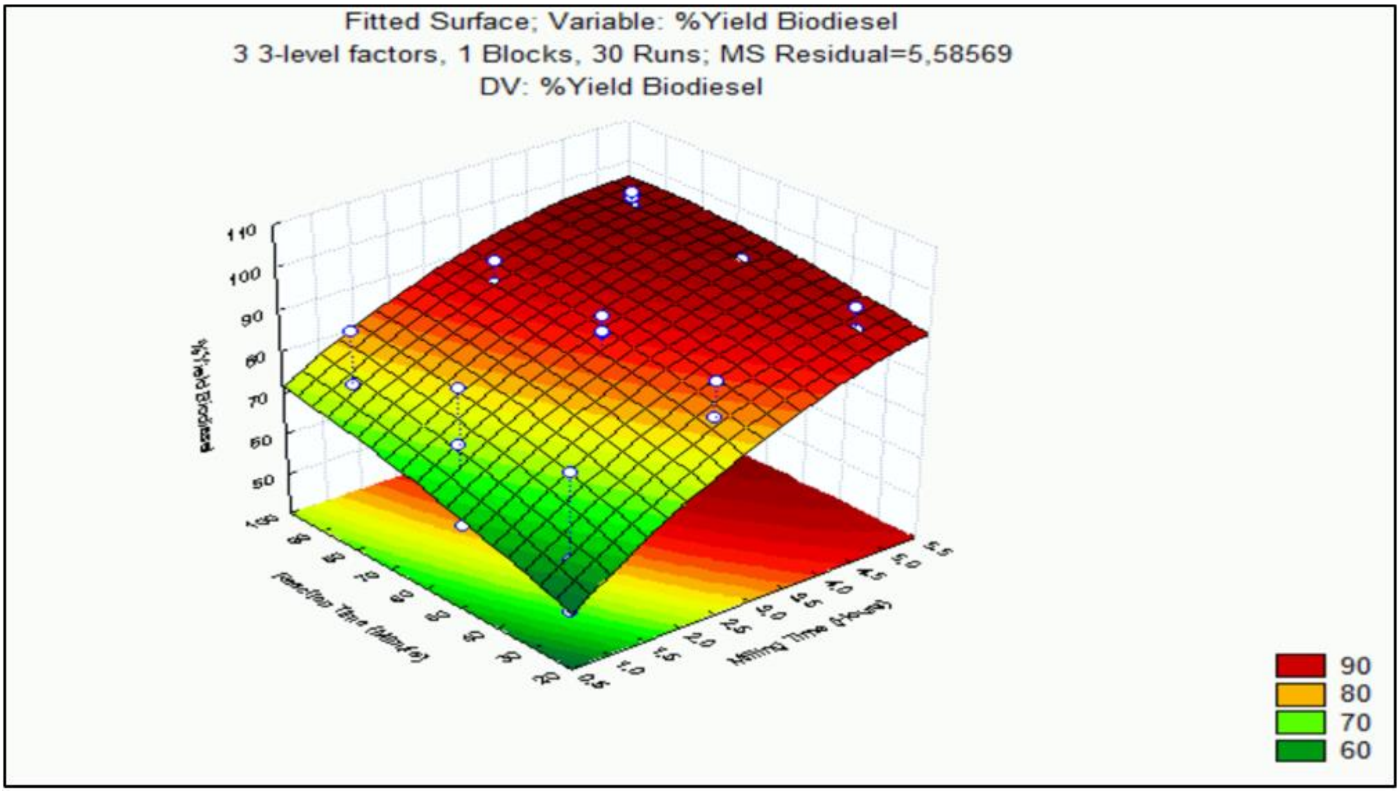

Fig 2 Graph Response Fitted Surface Methodology.

The accuracy of this model can be seen from the value of the coefficient of determination (R^2). Valur R^2 the provides a measure of how much of the variability in observed response values can be explained by the experimental variables and their interactions. From the R^2 value it can be concluded that the value that estimated by the model approaching the value obtained from the results of the experiment. The R^2 value always between 0 and 1. The closer value R^2 is to 1, the better model is at predicting the response (Yulianto, 2018). From the results of this study, the coefficient of determination value (R2 = 0.97752) shows that 97.75% of the variability in the response can be explained by the regression model that matches the experimental response. In Figure 3.2, the response graph shows the relationship between biodiesel yield, milling time (hours) and reaction time (minutes). By applying multiple regression analysis to the experimental data, a second-order polynomial equation was obtained to represent the acquisition of yield content biodiesel as follows :

$$Y=-96{,}6435-0{,}9778X_1^2+31{,}7682X_1-0{,}0012\ X_2^2+0{,}7946\ X_2-0{,}0290\ X_1X_2$$

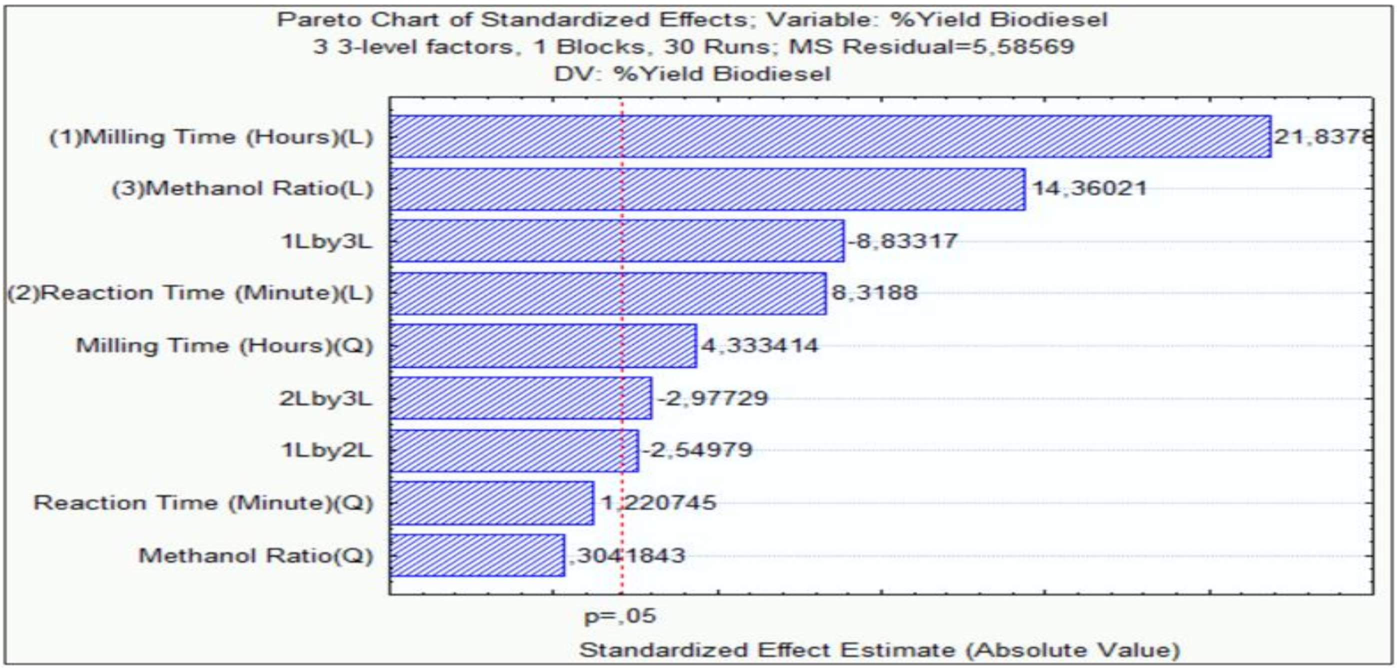

Fig 3 Pareto Diagram of the effect independent variables on %Yield Biodiesel

From the pareto block diagram in figure 3.3, it can be seen that the most influential independent variable in the biodiesel process from used cooking oil with kaolin nanoparticle catalyst is milling time with a standard effect of 21,837, meaning that the length of milling time linearly greatly affects the %yield biodiesel. The longer the time, the greater the % yield of biodiesel produced, followed by the effect of the methanol ratio which has a standard effect value of 14,360, meaning that the proportion of methanol added to oil has a strong effect, because the addition of methanol is the main reactant in the transesterification reaction process and The transesterification reaction time in the biodiesel production process has a standard effect value of 8,318, meaning that the reaction time has a significant effect but not as strong as the milling time or methanol ratio. Furthermore, interactions such as 1Lby3L appear to give meaning that the milling time and methanol ratio began to have an effect too, although not as strong as the single effect or linear effect. This shows that the more methanol added will shift the equilibrium in the biodiesel process so that the response of the % yield of biodiesel produced will be greater.

C. SEM-EDX Characteristics Kaolin Nanoparticle Catalyst

Scanning Electron Microscope (SEM) is a tool used to determine the morphology or microstructure of the surface of a material. Microstructure analysis is carried out to see the size and shape of the resulting particles. The SEM instrument is also equipped with EDX (Energy Dispersive X-Ray Analyzer) analysis where the characteristic X-rays emitted are the result of electron collisions on the atoms of the sample material(Susila Arita et al., 2020). The discussion will also cover the behavior of kaolin samples before and after calcination. Morphological analysis of kaolin before and after calcination in the form of SEM photos is presented in Figure 3.4.a which shows that the dominant content of the typical kaolin morphology is in the form of a group of layered hexagonal sheets with heterogeneous sizes. The results of Figures 3.4.a and 3.4.b show the morphology of porous silica with a magnification of 500x. The structure of kaolin has a size of 5 µm with the number of sheets per layer of around 10-50 pieces (Murray, 2000). Figure 3.3.b is produced from the best biodiesel production results, namely at 5 hours of milling time of kaolin after the calcination process, showing changes in the morphology of kaolin with the appearance of the destruction of pseudohexagonal sheets of the initial kaolin. Based on the morphology shown, the particles tend to form agglomerates.

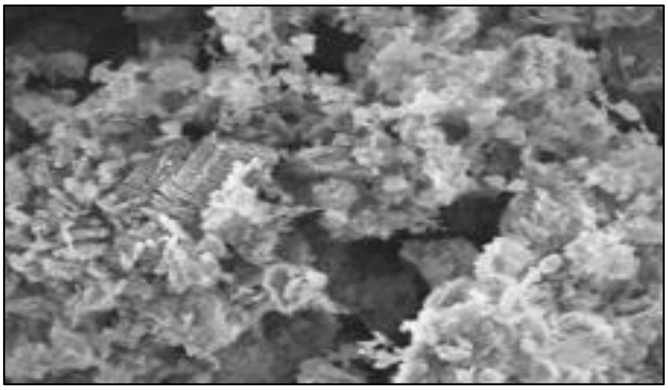
Fig 4 A. Kaolin before the calcination process

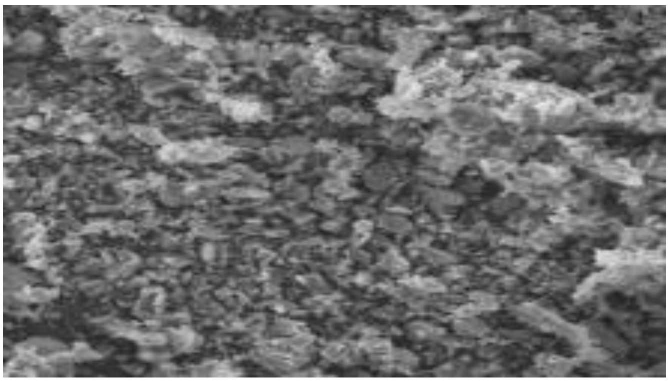
Fig 5 B. Kaolin after the calcination process

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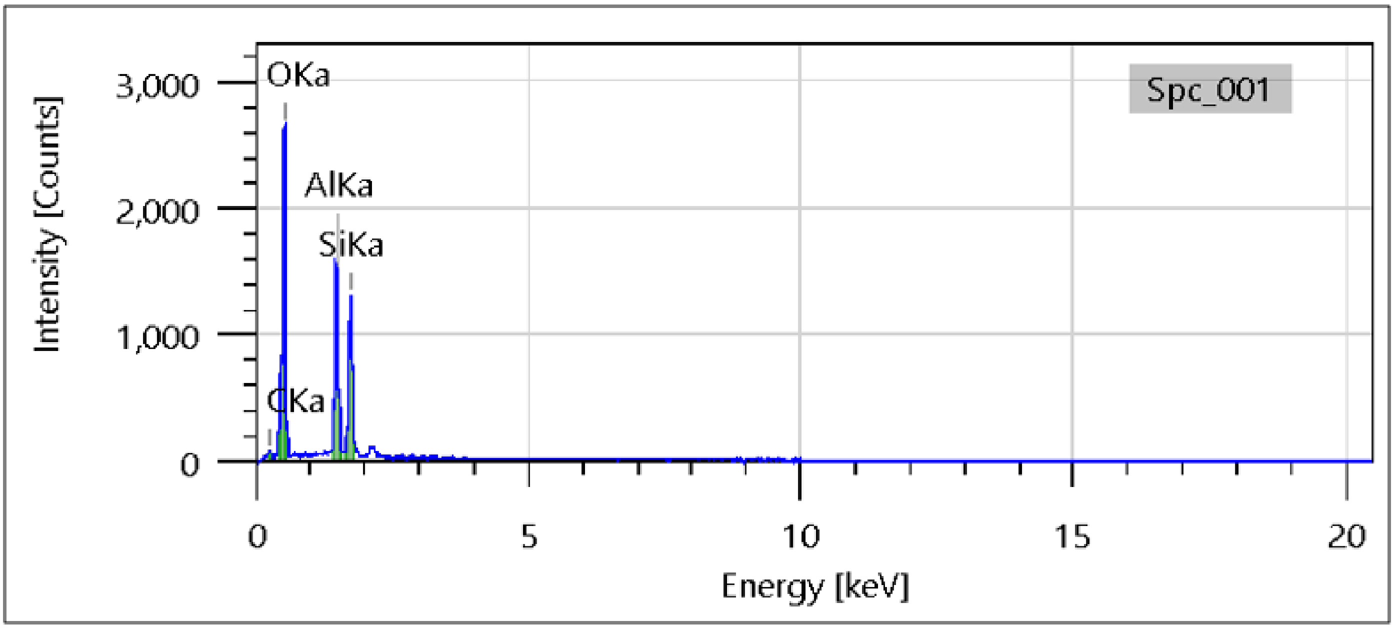

Fig 6 A Spektra EDX of Kaolin Before the Calcination Process

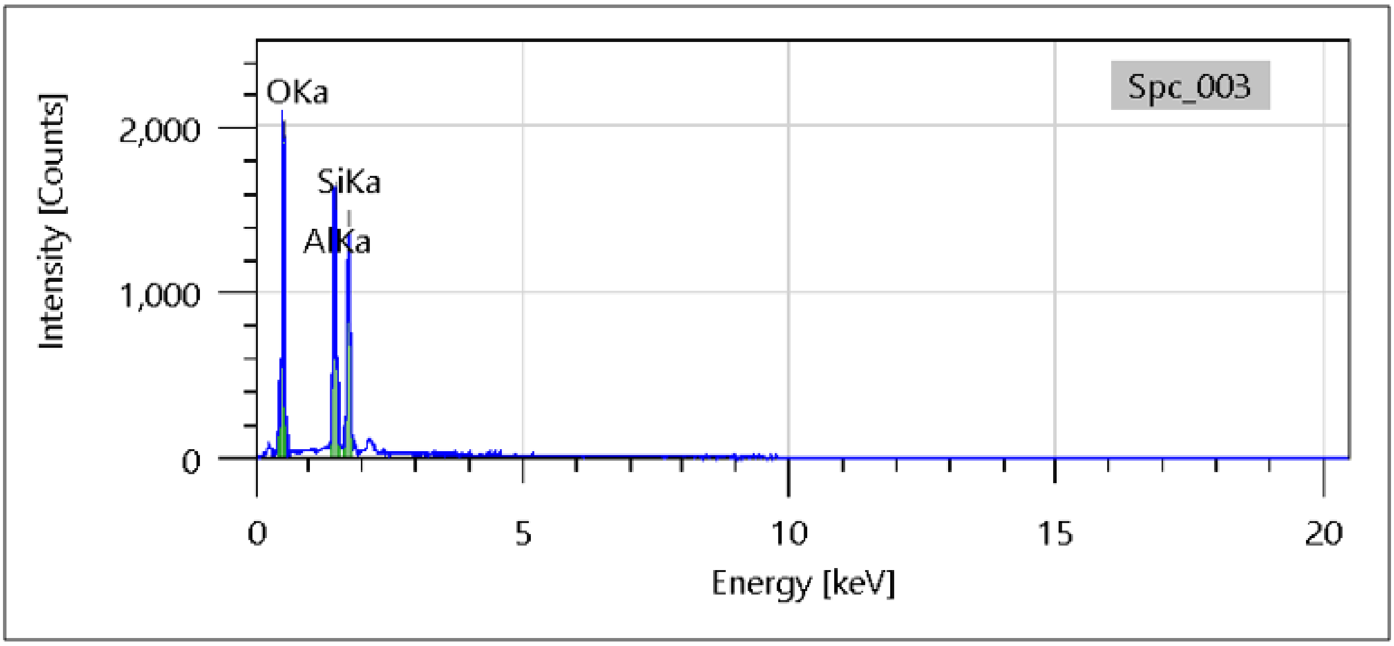

Fig 7 B Spektra EDX of Kaolin After Calcination Process

The graph in figure 3.5.a of the EDX spectra of kaolin before the calcination process show a high peak containing the composition of the elements C (3.07%), O (52.34%), Al (22.13%), and Si (22.46%). While the results of figure 3.5.b of the EDX spectra of Kaolin after calcination (Metakaolin) which show a high peak containing the composition of the elements O (46.20%), Al (26.39%), and Si (27.41%). Judging from the results of the composition of the elements formed before and after the calcination process, there was an increase in the the calcination process, there was an increase in the elements Al and Si. While the composition of the O element decreased. There was a decrease in the O element due to the removal of the OH group in kaolin and the formation of metakaolin with an amorphous phase following the following reaction. Calcination is heating at a certain temperature to remove certain compositions and remove water molecules (dehydroxylation). At low temperatures <100ºC, water absorption occurs in the pores on the kaolin surface. At a temperature of 100-400ºC loss related to the dehydration process, which is the result of rearrangement of the octahedral layers, occurs first in the OH groups on the surface and at a temperature of 400-800ºC, the dehydroxylation process occurs (removal of OH groups in kaolin and the formation of metakaolin with an amorphous phase (Alkan, 2005).

D. XRD Characteristics of Kaolin Nanoparticle Catalyst

Kaolin analysis using XRD aims to determine the crystallinity and minera; properties of kaolin. XRD analysis is carried out in an angle range (2θ) between 20-60 degrees with Cu radiation of 30.0 kv and current of 30 mA. The resulting data is pure kaolin before and after calcination (selected from the optimum results in biodiesel production)

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in the form of distance between planes, intensity and angle size (2θ). The results of kaolin analysis using XRD are shown in figure 3.6.a and figure 3.6.b.

The XRD date of kaolin from figure 3.6.a. shows pure kaolin before calcination. XRD analysis shows that the highest peaks of kaolinite characteristics are shown at angles 2θ = 25,2523° (3,52397); 38,7872° (2,31979); dan 20,7079 (4,28591) with relative intensities of respectively 100%; 39% and 29%. While the XRD date of figure 3.6.b. shows nanoparticle kaolin after calcination. calcination (metakaolin) the highest peaks for nanoparticle kaolin absorption are shown at angles 2θ = 27,2903° (3,26526); 26,8962° (3,31220); dan 26,5235° (3,35789) with relative intensities of respectively 100%; 55% and 55%. The results of XRD analysis also show that the crystallinity is pure kaolin of 56% and metakaolin 70%. The calcination process causes an increase in the crysallinity of kaolin. The crystallinity of a material indicates the strength of the bonds between atoms. Therefore, the higher the crystallinity, the stronger the bonds between atoms and vice versa(Yarangsee et al., 2021).

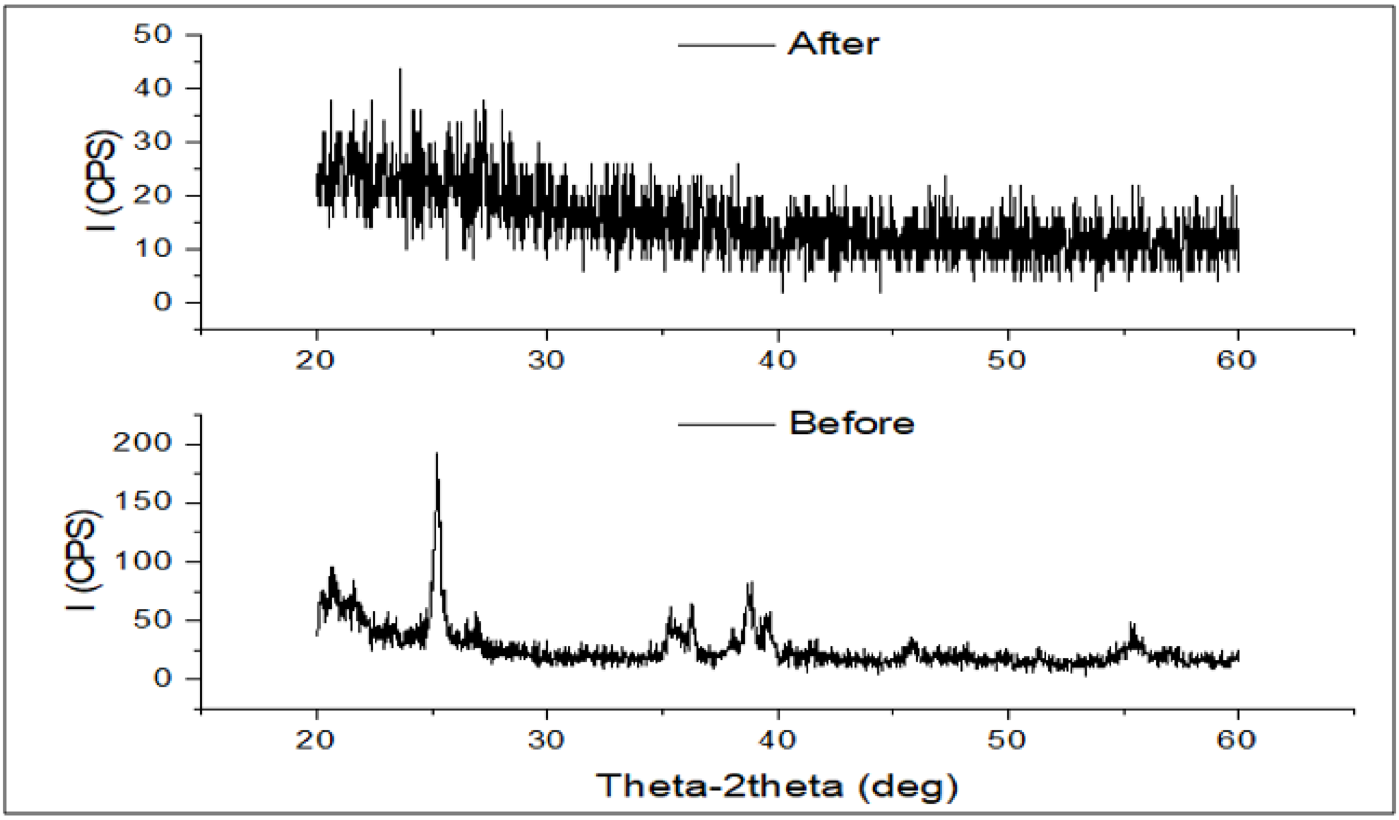

Fig 8 A XRD of Pure Kaolin, b. XRD of Kaolin after calcination process

IV. CONCLUSION

➢ *The Conclusions of this Study are as follows:*

- Ball milling type High Energy Milling Ellipse 3D Motion (HEM-E3D) can make particles smaller. Shown in the results of the Particle size analyzer (PSA) test at the longer milling time, the smaller the particle size produced. Selected at a collision time of 5 hours.
- The optimum value of %yield biodiesel which was produced in the response surface methodology design of 98,48% with the independent variable at optimum conditions, namely the collision time on the HEM-E3D device at 5 hours, then the transesterification reaction time at a time range of 80-90 minutes and the molar ratio of methanol and waste cooking oil at a ratio of (35:1).
- The calcination process on kaolin can form metakaolin where in the SEM-EDX results after calcination the content of the resulting elemental composition increases in Si and Al. While the composition of the O element decreases.
- The calcination process causes an increase in the crystallinity of kaolin. The crystallinity of a material indicates the strength of the bonds between atoms. As shown by the results of the XRD test where the crystallinity before and after calcination is high, namely pure kaolin of 56% and metakaolin 70%.
- Catalyst with small particle sizes can indeed increase the %yield value produced from the biodiesel process. The addition of more methanol can also increase the %yield of biodiesel.

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