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<http://fupre.edu.ng/journal>**RHS-doped FAU Zeolite-Y Catalyst Synthesis for Transesterification of Jatropha oil****OSHOMOGHO, F. O.^{1,*} , OSSAI, J. E.² **^{1,2} *Department of Chemical Engineering, University of Benin***ARTICLE INFO***Received: 21/01/2025
Accepted: 04/04/2025***Keywords***Biodiesel, Catalyst,
Clay, Silica, Zeolite***ABSTRACT**

This study investigated the synthesis of faujasite zeolite catalysts from aluminosilicate-containing natural sediments modified by biowaste silica in the catalysis of transesterification of jatropha oil. Zeolite was synthesized by the hydrothermal method with NaOH at 3.07g/mol, SiO₂ at 10.50g/mol, and Al₂O₃ at 2.61g/mol were the optimum amount required in the synthesis of a zeolite-Y in an autoclave. Transesterification reaction was conducted on constant temperature magnetic stirrer fitted with a reflux condenser. Functional group study of synthesized zeolite revealed that silicone (Si – O – Si) was highly prevalent at 1095.8cm⁻¹ in IR spectrum. XRD and EDS examination showed a faujasite zeolite-Y with Si at 85.60g and Al at 17.50g with Si/Al ratio of 4.891g/mol. The synthesized zeolite catalysis in transesterification reaction yielded 91.63% biodiesel which had 0.17% FFA. Biodiesel characteristics met ASTM standard having kinematic viscosity of 4.97mm²/s and flashpoint of 113°C. The fatty acid profile showed that fatty acid constituent was mostly linoleic acid methyl ester (30.29%) and hexadecanoic acid (20.81%), from GCMS analysis. The synthesized faujasite zeolite-Y, with its large mesoporous structure, exhibits remarkable catalytic activity in the transesterification of jatropha oil to produce biodiesel.

1. INTRODUCTION

Zeolite catalyst synthesis has been produced from kaolinic clay materials because of the basic constituents of kaolin (Murray 2002). Different types of zeolite have been exploited for applications in various catalytic functions as a result of different thermal or chemical treatments of feedstock clay materials because of the type and extent of impurities contained which mostly tend to be chemically bonded and the physical and chemical properties of the products (Kumar et al. 2013). Different types of zeolites have been exploited for applications in various

industrial functions as a result of different thermal or chemical treatments of feedstock. Clay materials because of the type and extent of impurities contained which mostly tend to be chemically bonded and the physical and chemical properties of the products (Kumar et al. 2013).

Zeolites have an advantage among all solid heterogeneous catalyst for industrial applications because of their uniform pore structure whose surface can be hydrophobic thereby preventing deactivation of active sites of the catalyst by polar solvents like water or alcohol. Zeolite can be synthesized

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to overcome several limitations that often arise by diffusion to enhance optimal productions in transesterification reactions. Zeolite catalyst has a high catalytic potential because of its reactivity. Catalysts play a significant role in the transesterification reaction. Catalyst types and concentrations are very important for achieving an optimal process (Liu *et al.*, 2018). Catalysts are usually used in the production of biodiesel to improve the reaction rate and yield. Catalytic activity is a function of its specific surface area, base strength, and base site concentration. Zeolite catalyst have several qualities such as not deactivated by water, stability, not activated at low temperature and have high selectivity (Rizwanul *et al.*, 2020). This study therefore synthesizes a faujasite type of zeolite-Y catalyst for the transesterification of high free fatty acid-containing jatropha oil, a non-edible seed oil.

2. METHOD

2.1 Clay Preparation and Dealumination

Clay will be obtained by calcination of pre-treated kaolin in a furnace at about 600 to 900°C for about 4 to 5 hours (Hartati *et al.* 2019). In order to adjust the mole ratio of Si/Al, about 120gm of metakaolin will be dealuminated by mixing with 500ml 60wt% H₂SO₄ solution in a 1000ml round bottom flask and stirred to form a homogeneous mixture with the aid of a magnetic stirrer and left to react without external heating for about 10 minutes, before heating externally for 5 minutes. The temperature of the reaction will be noted to start from about 55°C and progressed to about 120°C (Ajayi *et al.* 2010). The reaction will be stopped, and the collected solid will be washed with deionized water until a neutral pH, filtered, and oven dried at 120°C for about 4hrs and packaged for analysis.

2.2 Synthesis of Zeolite from Local Clay Sediment

An adequate volume of a standard NaOH solution was prepared and gradually added to specific mass of metakaolin and rice husk silica mixture to obtain an aluminosilicate gel according to the work done by Pandiangan *et al* (2019). The gel will be continuously stirred at room temperature for 120minutes with a magnetic stirrer and allowed to age for 24 hours. A hydrothermal treatment would be performed on the mixture at 120°C for about 6 hours with the aid of autoclave after which the mixture would be subjected to drying in the oven for about 4 hours at 105°C to remove any moisture content. The dried sample would then be calcined in the muffle furnace at temperature between 600 to 900°C. The calcine sample will be cooled to ambient temperature in a desiccator and then analyzed for type of faujasite zeolite formed as well as testing for its catalytic activities in the transesterification of jatropha seed oil.

2.3 Silica Doped Zeolite Preparation Method

Silica was extracted from rice husk using the acid crystallization method according to Oshomogho *et al.*, (2023). 5g of silica was added to synthesized 50g zeolite containing 100ml of de-ionized water. The mixture was thoroughly blended, oven dried and calcined in a muffle furnace at 650°C for 60minutes and cooled in a desiccator.

2.4 Methods of Physical and Chemical Characterization of Zeolite

Characterization of synthesized zeolite samples will be conducted with Fourier transform infra-red spectroscopy (FTIR), X-ray fluorescence equipped with energy dispersive spectrometer (XRF-EDS), X-ray diffractometer (XRD), and Scanning electron microscope (SEM) to determine the functional groups, elemental and oxides composition, type and crystallinity of

synthesized samples as well as surface morphology respectively.

2.5 Application of Modified Faujasite Zeolite Catalyst in Biodiesel Production

Jatropha oil, an inedible seed oil, was added to a 1000ml 2-neck flask equipped with a Braham condenser and placed on a constant temperature magnetic stirrer to achieve a homogenous mixture at a constant mixing speed of 500rpm. The reaction flask was attached to a Braham condenser to condense any evaporating methanol and then return to the flask. For the duration of the 60minutes of reaction, at constant temperature. The experiment was repeated at a variety of temperatures.

2.6 Esterification and Transesterification Reaction

Exactly 50g of oil was measured into a flat bottom flask and set to preheat to the required reaction temperature with constant stirring at about 300rpm. The catalytic reaction of jatropha oil with methanol was with a fixed methanol to oil molar ratio of 7:1 (i.e. 25wt%). Catalyst loading of 1.0 to 5.0wt%, reaction temperature of 40 to 80°C and reaction time of 30 90minutes at a constant mixing speed. The reaction was allowed to stand for about 2 hours to attain complete esterification and transesterification of the fatty acids component of the triglycerides into fatty acids methyl esters (FAME). Finally, catalyst and glycerol part were separated from the biodiesel mixture by centrifugation and a separating funnel for 24 hours.

2.7 Crude Biodiesel Purification Technique

The crude biodiesel was separated from the feedstock and washed with 28% (by volume) distilled water to remove impurities. To prevent the formation of an emulsion, crude biodiesel was combined with distilled water

and gently stirred, allowing droplets of water to slowly percolate through the ester (Atadashi et al. 2013). In order to ensure that all contaminants were removed, this procedure was repeated until clear wash water was produced. The American Society for Testing and Materials (ASTM) required this level of purification to ensure compliance with its international standard specification. Biodiesel quality can be diminished and engine performance impacted by impurities including residual methanol, glycerol, catalyst, glycerides, and FFA (Atadashi et al. 2013).

2.8 Product Estimation

The yield and conversion were calculated as follows:

$$\text{Yield}(\%) = \frac{\text{Weight}_{\text{Biodiesel}}}{\text{Weight}_{\text{Oil}}} \times 100$$

(1)

$$\text{Conversion}(\%) = \frac{\text{Weight}_{\text{Triglyceride}} - \text{Weight}_{\text{Biodiesel}}}{\text{Weight}_{\text{Triglyceride}}} \times 100$$

(2)

Where 50g is the initial mass of oil, which was calculated using the weight of triglyceride. Clean biodiesel resulting from the transesterification reaction was weighed to determine the yield of each run.

3. RESULTS

3.1 Results of FTIR Analysis of Synthesized Zeolite

Figure 1 illustrates the locations of the peaks in the infrared spectrum of the synthetic crystalline zeolite sample. The structure of the zeolite does not affect the location of the internal TO₄ (T = Si or Al) tetrahedral bending peak, which may be found at 693.3 cm⁻¹. The zeolite-specific double ring

external linkage is the cause of the peak that can be found at 565 cm^{-1} . The peak that occurs at 685 cm^{-1} is attributed to external linkage symmetrical stretching, whereas the peak that occurs at 775 cm^{-1} is attributed to internal tetrahedral symmetrical stretching. The peaks at 1010 cm^{-1} and 1080 cm^{-1} have been assigned to internal tetrahedral asymmetrical stretching and external linkage

asymmetrical stretching, respectively. The peak near 3400 cm^{-1} has been assigned to the hydroxyl groups of zeolites. In general, the FTIR spectra of nanocrystalline zeolite Y that has been artificially manufactured closely matches the FTIR absorption peaks of naturally occurring microcrystalline zeolite Y. (Taufiqurrahmi *et al.*, 2011).

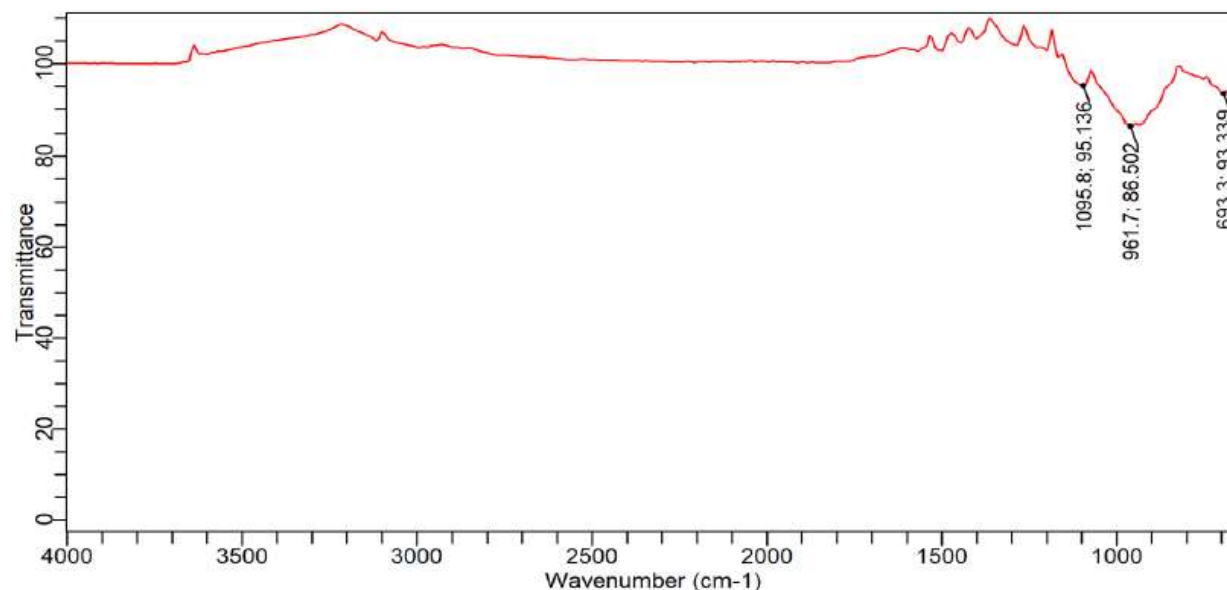


Figure 1. FTIR Spectrum of synthesized zeolite

This analysis showed what kinds of functional groups present on the catalyst's surface and how those groups interacted with one another. The FTIR spectrum of the impregnated catalyst showed wavelengths of 1095.84 cm^{-1} , 961.65 cm^{-1} and 693.28 cm^{-1} respectively.

3.2 Results of EDS Analysis of Modified Faujasite Zeolite

Energy dispersive X-ray spectrometer was used to determine the Si/Al ratio in the

modified zeolite catalyst. Figure 2 revealed that Si was present at 85.60wt% and Al at 17.50wt% making a ratio of 4.891. This is a faujasite type of zeolite and a zeolite Y which usually have ratio $\text{Si}/\text{Al} > 2$ (O. A. Ajayi *et al.* 2018). Stability of zeolite is enhanced with an increase in the Si/Al ratio (García-Martínez *et al.* 2012; Mgbemere *et al.*, 2019).

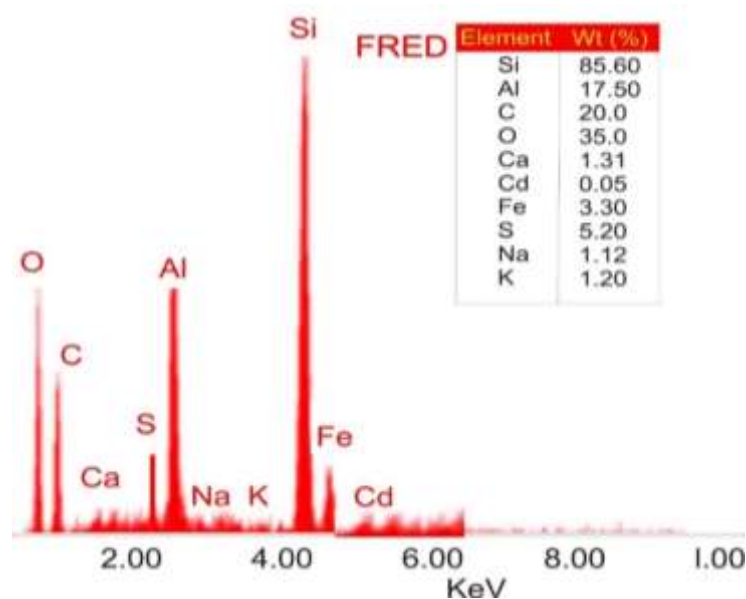


Figure 2. EDS spectrum of modified faujasite zeolite

3.3 Results of TGA and DTA Analysis of Modified Zeolite

Figure 3 shows that desorption of loosely bound sorbate (nitrogen) started very mildly at 96°C with about 2% of mass lost. Stability and sorbate retention was retained as temperature increased to 237°C with only 11% mass of sorbate lost. Sorbate retention stability started to reduce at about 280°C at which about 58% of sorbed nitrogen desorbed and at 550°C only about 10% was retained at which stability of sorbate on the zeolite surface was lost.

The zeolite sample's thermogram revealed two response behaviours. The first is an

endothermic reaction at low temperatures, whereas the second is an exothermic reaction at high temperatures. To adsorb the inert gas (nitrogen), the operational index, such as desorption temperature, adsorption time, adsorption surface area, and so on, was utilized.

The sharp exotherm (onset 1584.40°C, peak 1599.64°C K) is assigned to the formation of separate silicate and aluminate molecules. The endothermic loss of water molecules occurs at 1352.64°C, and the endotherm at 1665.35°C corresponds to the breaking of to compound. The thermogram of the sample exhibits an endotherm (1665°C) for melting.

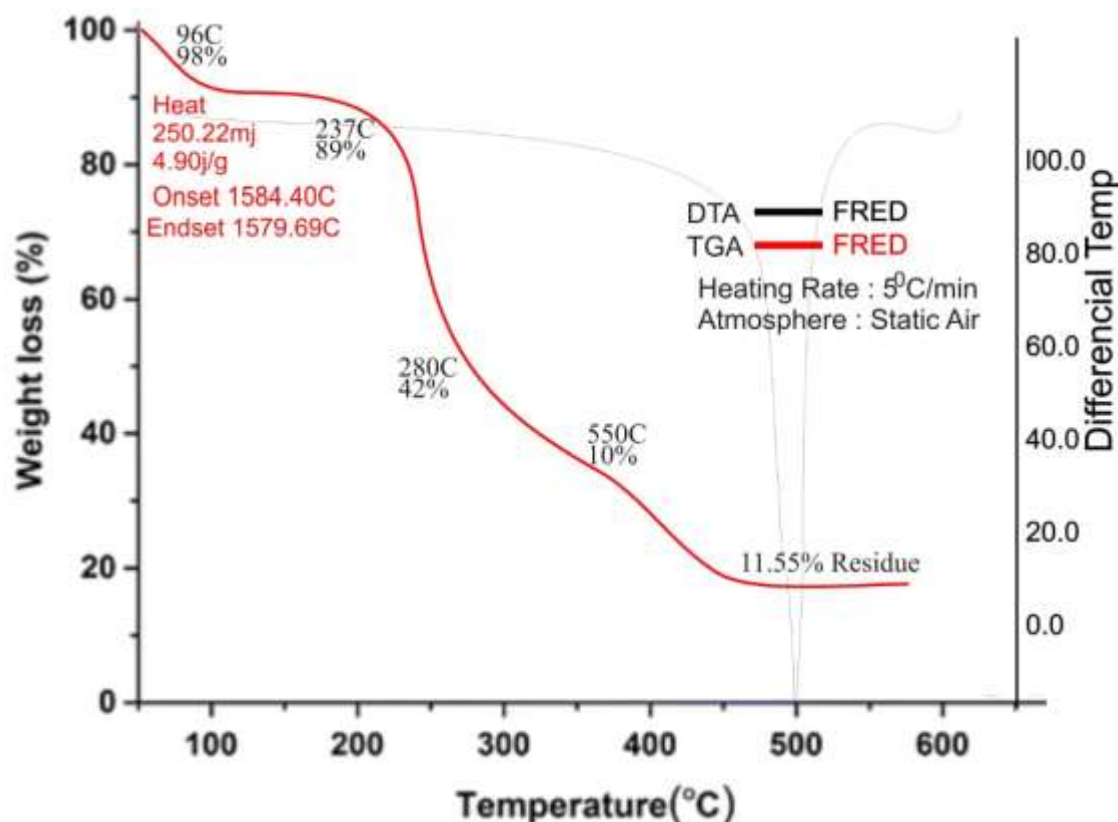


Figure 3. TGA and DTA Thermogram of modified faujasite zeolite

3.4 Result of Biodiesel Synthesis from *Jatropha* Oil using Zeolite Catalyst

In the production of biodiesel from *jatropha* oil, the catalytic activities of synthesized zeolite via hydrothermal reaction between silica from rice husk ash and alumina from local clay were examined on the transesterification of *jatropha* oil and methanol and the results obtained showed

high conversion of the feedstock into fatty acid methyl ester. Some parameters of biodiesel after purification with water washing were tested using the American Society for Testing and Materials standard (ASTM) for pure biodiesel (B100). Table 4.17 revealed that the results demonstrate that all of the measured values were within the test limit range.

Table 4.17 Physicochemical Properties of *Jatropha* Biodiesel

Properties Measured	Values Obtained	Standard ASTM Values
Biodiesel yield (%)	89.74	NA
Density @ 30°C (g/ml)	0.87	0.88
Kinematic viscosity @ 30°C (mm ² /s)	3.97	1.9 – 6.0
Acid value (mgKOH/g)	0.341	<0.5
Free Fatty Acid (%)	0.1705	NA
Flash Point (°C)	113	100 – 170
Calorific Value (MJ/kg)	40.41	35>

Note: NA = Not Available.

The specific gravity is directly proportional to density; therefore, the specific gravity varied the same way as the density with the lowest density, 0.88g/cm^3 (Table 4.17). The viscosity is also directly proportional to the density, so viscosity varied the same way as density. The kinematic viscosity was $4.97\text{mm}^2/\text{s}$ as shown in Table 4.17. According to Prah, (2010), the ASTM standard range for biodiesel viscosity at 40°C is between 1.9 to $6\text{mm}^2/\text{s}$, this implies that the viscosity of biodiesel obtained in this experiment is within the standard range of biodiesel viscosity. It is important for the density, specific gravity and viscosity to comply with the specification of the biodiesel standards because these parameters affect engine performance to a great extent. The values obtained for the biodiesel samples in experiment are within the accepted range of biodiesel standards. As shown in Table 4.17,

the flash point for biodiesel sample produced in this experiment is 113°C which is within the range of the standard specification for biodiesel flash point, $100 - 170^\circ\text{C}$ (Sahoo, 2020). This implies that the biodiesel is safe for handling and transport purposes.

3.5 Results of GCMS Analysis of *Jatropha* Biodiesel Catalysed by Zeolite

Figure 4. shows the various peaks on the chromatogram obtained from the GC-MS analysis of the biodiesel sample produced at optimum conditions for *Jatropha* biodiesel. Each peak represents a specific compound which has been identified from the library software (NIST 14 mass spectral database). Tables 4 summarizes the compositions and concentrations of fatty acid methyl ester present in *jatropha* and castor biodiesel respectively.

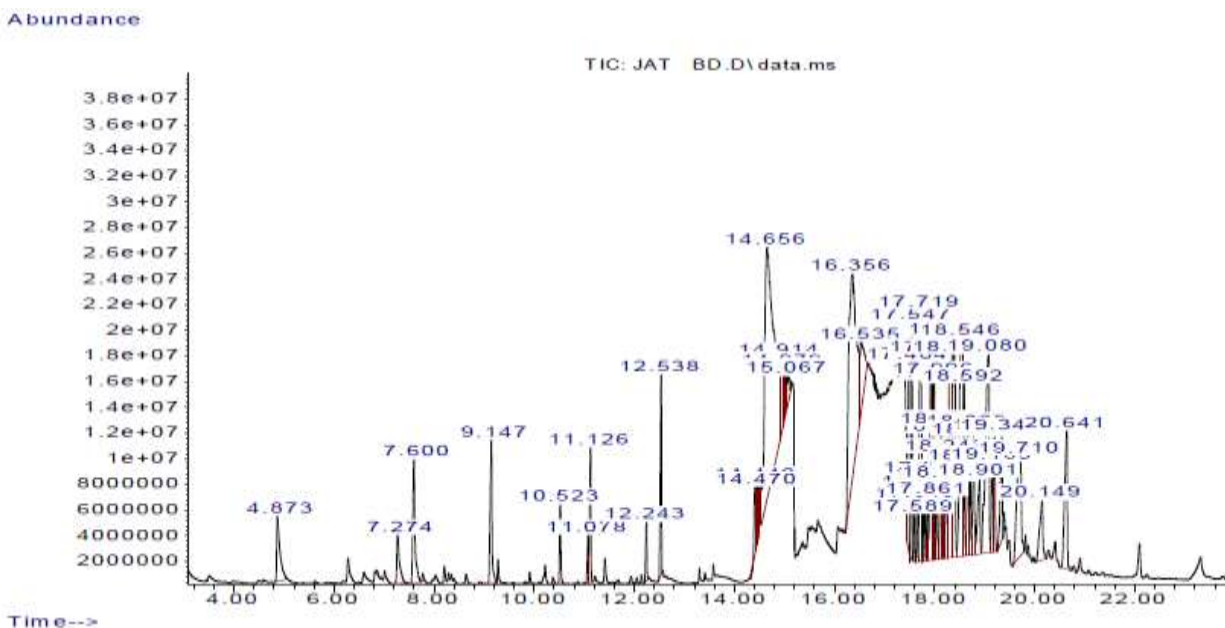


Figure 4. Chromatogram of *jatropha* biodiesel produced at 60°C reaction temperature, 90minutes reaction time, 9:1 methanol to oil mole ration.

The major fatty acid methyl ester present in the *jatropha* biodiesel sample were Octadecadienoic acid, methyl ester which is a linoleic acid with a concentration of

30.29% followed by hexadecanoic acid, methyl ester with a concentration of 20.81%.

3.6 Effect of Catalyst Loading

As shown in Figure 5, low yield (38.73 – 85.47%) was obtained with low zeolite catalyst loading (1 – 4wt%), which was insufficient to catalyse the reaction to completion in the synthesis of fatty acid methyl ester. This observation corroborates what Hussein et al. (2021) reported, that heterogeneous catalyst in biodiesel production should range from 1.0 – 10.0wt% for higher yield.

Higher biodiesel yield (90.18%) was observed at an optimum point of 5.0wt% catalyst loading. Beyond this range (6 -10 wt%), a decrease in biodiesel yield was observed. This observation can be attributed to the non-availability of active sites on the

catalyst for transesterification reaction (Kim et al., 2019). Reaction rates increased as reactants occupied more catalytic sites until saturation was reached. The impact of mass transfer limitation became more significant at higher catalyst loading, thus limiting reactants' accessibility to active sites (Thangaraj et al., 2019). The transesterification reaction is strongly dependent on the weight of catalyst which consequently affects the yield. An adequate increase in catalyst concentration results in an increase in the number of its active sites, thereby increasing the yield of methyl ester (Aftab et al., 2020). Excessive catalyst loading leads to high slurry viscosity and consequent poor reaction mixtures (Liu et al., 2018).

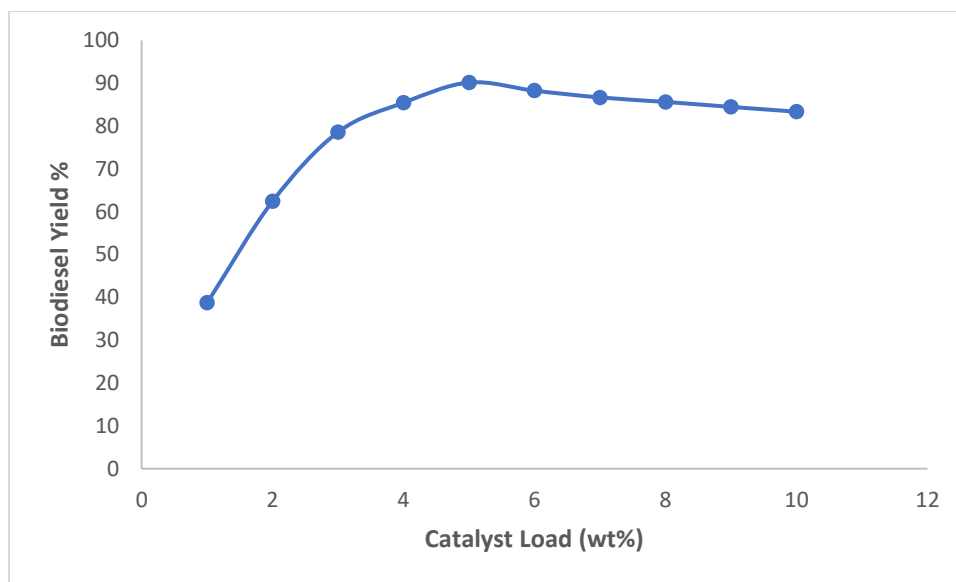


Figure 5. Effect of catalyst loading at 60°C reaction temperature and 9:1 methanol to oil mole ratio.

Effect of Temperature

Temperature had an impact on the mass transfer of the reaction: the optimum temperature was found to be 60°C with a yield of 89.93% under optimal conditions (catalyst loading 5.0 wt%, methanol -to- oil ratio 6:1) (Figure 6). As the reaction temperature increased to 60°C, there was also

an increase in the reaction rate due to higher energy input and reduced mass transfer resistance (Lin and Hsiao, 2010). Increasing the temperature towards 70°C and beyond resulted in a decrease in the production due to the fact that any reaction occurring beyond the methanol boiling point (65°C) resulted in its intense continuous vaporisation. Hence it remained in the gas phase in the reflux,

causing a reduction of methanol in the reaction media (Dewanto et al., 2017).

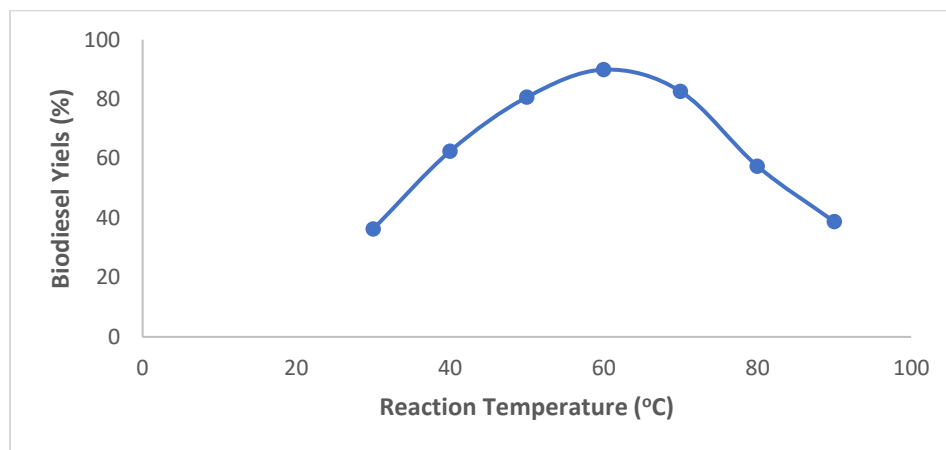


Figure 6. Effect of Reaction Temperature at 5wt% catalyst load and 9:1 methanol to oil mole ratio.

3.7 Effect of Methanol to Oil Molar Ratio

The optimum biodiesel yield (91.63%) was observed at a methanol-to-oil molar ratio of 9:1. It can be observed from Figure 7, that a decrease in molar ratio below the optimum point (9:1) had a negative impact on the biodiesel yield. The yield decreased drastically to 35.31%, at an methanol -to- oil ratio of 4:1. This is due to the dilution effect of insufficient methanol and higher molar ratio will result in dilution effect and subsequent interference between the high

molar ratio of methanol/oil and the catalyst (Jamil et al., 2021).

There was a subsequent increase in solubility and a decrease in the separation of glycerine and methyl ester (Gondra, 2010; Wong et al., 2020). Zamberi and Ani, (2016) showed that the most suitable molar ratio was found to be within 9:1, in the production of biodiesel using zeolite as heterogeneous catalyst. The overloading of methanol would inactivate the catalyst and consequently favour the backward reaction of transesterification process (Aransiola et al., 2013; Tiwari et al., 2006).

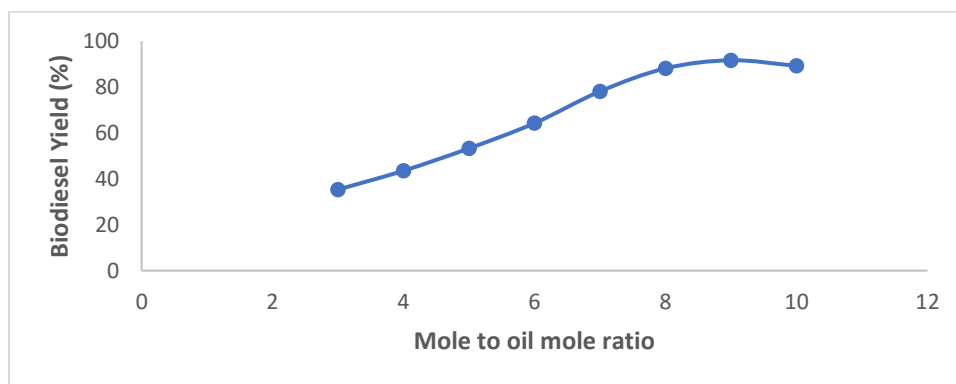


Figure 7. Effect of methanol to oil ratio on the yield of biodiesel at 5wt% catalyst load and 60°C reaction temperature

3.8 Reaction Time

The effect of reaction time on the conversion of palm oil to biodiesel was investigated. Reaction time is one of the key parameters during the transesterification carried out in glass reactor using zeolite catalyst. Figure 8 shows an increase in the yield with time from 30 to 120 minutes with a catalyst amount of

5wt% relative to oil and a methanol/oil molar ratio of 9:1. The maximum yields of 91.45% was obtained in 80 minutes resident time of reaction. In the initial stages of the transesterification reaction, production of biodiesel was rapid, and the rate diminished and finally got to a steady state at a reaction time 80 minutes and beyond. The uptake value increases as the contact time increases (Zamberi and Ani, 2016).

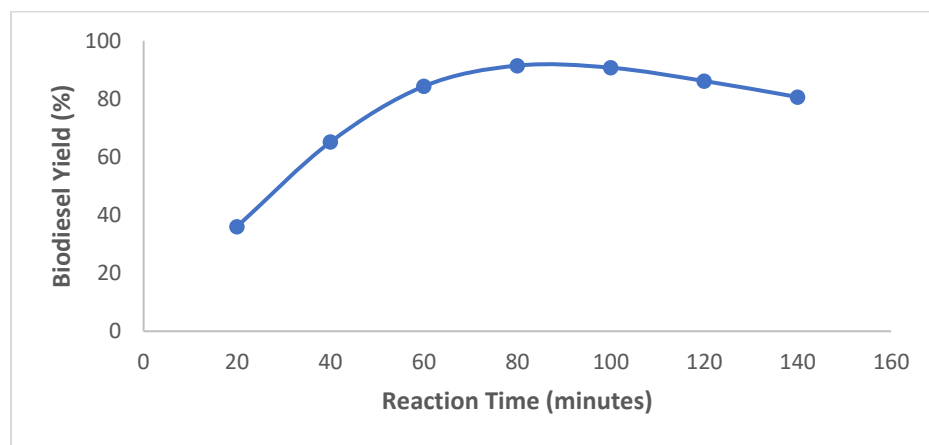


Figure 8. Effect of reaction time on biodiesel

4. CONCLUSION

Aluminosilicate containing clay minerals are the major ingredient for zeolite synthesis. Faujasite Zeolite Y has a potential as a bi-functional catalyst for biodiesel production. Zeolite has an Si presence of about 85.60wt% and Al at 17.50wt% with a ratio of 4.891 silicon to aluminum. Silica doped zeolite catalyst is most effective in the transesterification of jatropha oil at about 5wt% of catalyst loading. High biodiesel yield of 91.63wt% can be obtained with methanol-to-oil molar ratio of 9:1.

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