



## Original Research Article

### Development of Zeolite from Locally Sourced Clay for the Cracking of Waste Engine Oil

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<http://doi.org/10.5281/zenodo.12600371>

#### ARTICLE INFORMATION

##### Article history:

Received 05 May 2024

Revised 07 Jun. 2024

Accepted 13 Jun. 2024

Available online 30 Jun. 2024

##### Keywords:

Waste Engine Oil

Zeolite

Catalyst

Clay

Cracking

#### ABSTRACT

The current global economic realities have necessitated the transformation of used motor oil into valuable hydrocarbons, lowering dependency on virgin resources, fostering a circular economy, and supporting economic sustainability. This research therefore focuses on the production of a catalyst that is sustainable in the re-engineering of waste engine oil. The waste engine oil used was collected locally in Benin City, Edo State, Nigeria and the clay used for the production of zeolite was obtained from Ibusa, Delta State, Nigeria. The Zeolite was prepared by calcining clay at 900°C. It was then de-aluminated using sulfuric acid. The elemental composition was analysed of the Zeolite with an X-ray fluorescence (XRF) which showed that the prevalent elements were silica and alumina with concentrations of 71.74% and 13.91% respectively. X-ray diffraction (XRD) was used to determine the catalytic characteristics of the zeolite obtained from neutralized de-aluminated clay. Four compounds identified as the catalyst were orthoclase, quartz, albite, and muscovite, with figures of merit of 1.70, 0.83, 2.18, and 2.81, respectively. The effect of the catalyst load on the rate of conversion was depicted in a plot of the various variations of the catalyst against their respective cracking rates. The result showed that at 5%, 10%, 15%, 20%, and 25% of catalyst load, the cracking rates were 0.022 min, 0.012 min, 0.108 min, 0.054 min, and 0.106 min, respectively. This paper gave a comprehensive understanding of zeolite synthesis and its effectiveness in the cracking process of used engine oil is presented and discussed.

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## 1. INTRODUCTION

The development of machines and vehicles that require fuel as a result of human evolution, population growth, and technological advancement has led to an increased demand for energy sources (Armaroli and Balzani, 2007). This has prompted scientists and engineers to explore alternative and renewable energy options to reduce reliance on fossil fuels (Holechek *et al.*, 2022). Additionally, the negative environmental

impact of burning fuel has raised concerns about air pollution and climate change, further driving the need for sustainable energy solutions (Omer, 2014; Perera, 2018). For example, used oil from internal combustion engines generally accumulates a variety of contaminants which increase the oil's toxicity (Aljabiri, 2018). Since waste engine oil contains dangerous substances like heavy metals, organic contaminants, and carcinogens, it must be properly disposed of to prevent environmental risks. In conversion and upgrading processes, catalysts are crucial components because they make it possible to produce valuable products without being consumed in the process. The latter are substances that can be used in chemical reactions, some of which are commercially available (Pawar, 2020).

Zeolite is a common catalyst used in the catalytic cracking process in the modern petroleum industry (Olaremu *et al.*, 2021). It is a hydrated aluminosilicate known to have generated higher profits with lower capital expenditures. For the refiner, zeolites were and remain the best deal ever (Zafari and Kharat, 2013). According to Vreeswijk and Weckhuysen (2022), zeolite materials are used in common products and biomedical applications, and to address a variety of climate change issues as long-term alternatives to, for example, the depletion of crude oil and the recycling of plastic as catalyst materials. Numerous types of zeolites are developed and used for industrial applications, from their early use in petroleum refineries to more recent uses in environmentally friendly, sustainable chemical processes (Niwa *et al.*, 2010).

The objectives of waste engine oil processing are to upgrade petroleum-based wastes by refining and recycling them to create products like heavy oil and gasoline that can be used again and again. Cracking, distillation, and pyrolysis are acceptable procedures (Balat, 2008). The cracking process was used to convert waste-used engine oil into valuable chemicals and fuel. Waste engine oils can be cracked at high temperatures to produce olefins-rich oils. The most common type of cracking is catalytic cracking, otherwise known as Fluid Catalytic Cracking. Fluid catalytic cracking (FCC) is a central technology in modern refining (Nemati Kharat and Zafari, 2013). Olaremu *et al.* (2021) also stated that catalytic cracking incorporates the use of a catalyst to facilitate high thermal efficiency at a shorter time under low temperature and pressure. The intrinsic characteristics of zeolite, which are also in charge of determining its general behaviour, combine to give rise to the catalytic properties of zeolites. The unique properties of zeolites make them attractive in a variety of industrial applications. Their secret lies in their porous structure, the wide variety of their three-dimensional channel system, and the diversity of their pore size (Vajtai, 2013). Other advantages of catalytic cracking over other types include high product selectivity, high yields of gasoline, high octane number, and high aromatic yield, among others.

According to Gnanavel *et al.*, (2010), it is necessary to consider crucial catalyst factors like texture and chemical surface to connect the catalyst's catalytic activity to its physical characteristics. As a result, the characterization of catalysts becomes crucial to any research on catalysis. The characterization techniques employed were X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier Transform Infra-Red (FTIR) spectroscopy and Gas Chromatography-Mass Spectrometry (GCMS). By utilizing these characterization techniques, an optimized zeolite catalyst that can be used effectively for the cracking of waste engine oil production can be developed.

## 2. MATERIALS AND METHODS

### 2.1. Materials

The main materials utilized during the course of this work are waste engine oil collected from a local mechanic workshop at Benin City, clay (Used in the production of zeolite) obtained from Ibusa in Oshimili North Local Government Area (Figure 1), Asaba, Delta State, Nigeria (6°11'45"N 6°37'38"E). All reagents used in this work were of analytical grade and obtained from Luco Scientific Chemical Laboratory, Benin City, Edo State, Nigeria.

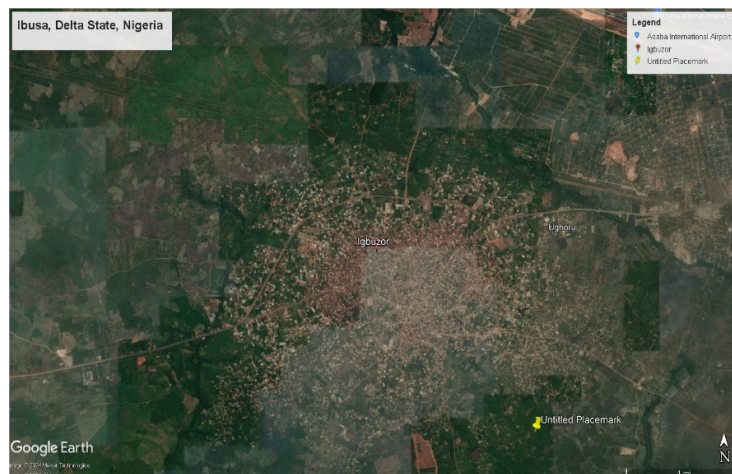


Figure 1: Geographical location of the sampling site in Ibusa, Delta State, Nigeria (6°11'45"N 6°37'38"E)

## 2.2. Sample Preparation and Pretreatment

The clay sample was obtained and dried, hence, there was no need for sun-drying it. It was then crushed in a crucible into powdery form and sieved using a 2 mm mesh screen to obtain a homogeneous particle size. The clay sample was also subjected to a calcination process for 2 hours at a temperature of 900 °C in a furnace. A small quantity of the clay was set aside for XRF analysis for its elemental composition before the calcination process. It has been reported that the calcination of clay takes place at high temperatures in line with the work of Mgbemere *et al.*, (2018). By doing this, the ions in the clay were activated and became more reactive. The calcined clay was added to the prepared 600 ml of H<sub>2</sub>SO<sub>4</sub> solution to de-aluminate the clay. Acid treatment has been found to improve zeolite reactivity via de-alumination and simultaneously increase its specific surface area (Chen and Srubar, 2020). The clay was repeatedly washed with ionized water and filtered till it was neutral (pH of about 7) to remove any unspent acid. A hydrothermal treatment was performed with the aid of an autoclave at a set temperature of 130 °C for 2 hours on a mixture of the washed clay and 2.5M of NaOH. This was done to neutralize any acid still present in the clay. The mixture was filtered and washed with ionized water till it was neutral and dried in an oven at a set temperature of 150 °C. The zeolite obtained was then characterized using X-ray Diffraction.

## 2.3. Cracking Process

The standard mass of waste engine oil used was 50g. This said mass was measured with the aid of a weighing balance and poured into a distillation flask at a temperature of 38.5 °C. The flask containing the oil was placed on a heating mantle which was preheated for about 2 minutes before adding the catalyst. The 50g of oil was varied with 5%, 10%, 15%, 20% and 25% of the catalyst. For each of the variations, the temperature and time readings were noted down for the first drop, 10ml, 15ml and 20ml of the cracked product. FTIR and GCMS analyses were conducted on the cracked product.

## 2.4. Determination of Density and Viscosity

Before the commencement of the cracking process, the density of the waste engine oil was determined using a pycnometer. The empty density bottle was placed on a weighing balance to get its weight. Then, the weight of the density bottle containing the waste engine oil was also recorded. The values obtained were used in the calculation of the density. The viscosity determination of the waste engine oil was carried out with the aid of a viscometer and was carried out following the Esteves *et al.*, (2016) method. The density and viscosity of the cracked product were also determined. The values obtained were recorded.

### 3. RESULTS AND DISCUSSION

#### 3.1. X-ray Fluorescence Analysis

The elemental analysis of the raw and calcined Ibusa clay is presented in Table 1. The result showed that SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> were the major elements present with concentrations of 71.74% and 13.91% respectively which is in line with the work of Omisanya *et al.*, (2012) which also had them as the elements in abundance. The clay was also found to contain other oxides of iron, magnesium, manganese, calcium, potassium, sulphur, and titanium among others which were in minimal amounts. These oxides were also present in trace amounts as reflected in the works of Folorunso *et al.*, (2014) and Adeoye *et al.*, (2017). A key indicator of the presence and quality of kaolinite in a clay mineral is its percentage composition of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> (Adeoti *et al.*, 2019). From a previous study, the small amount of K<sub>2</sub>O may indicate a small amount of illite clay mineral (Schackow *et al.*, 2020). The molar ratio of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> for the clay was 5.16, which was higher than the theoretical molar ratio (range of 1-2) from the previous study of Adeoye *et al.*, (2017). Molar ratios higher than the theoretical value suggest the presence of quartz and other silicates in the samples which is also attributed to the work of Schackow *et al.*, (2020). This high quartz content was revealed from the result obtained from the XRD analysis.

Table 1: XRF analysis of the raw calcined clay

Elements	Concentration (%)
Fe <sub>2</sub> O <sub>3</sub>	1.082
SiO <sub>2</sub>	71.742
Al <sub>2</sub> O <sub>3</sub>	13.909
MgO	3.690
P <sub>2</sub> O <sub>5</sub>	0.322
SO <sub>3</sub>	0.130
TiO <sub>2</sub>	1.105
MnO	0.076
CaO	0.132
K <sub>2</sub> O	0.115
ZrO <sub>2</sub>	0.173
Nb <sub>2</sub> O <sub>5</sub>	0.022
CuO	0.003
ZnO	0.005
V <sub>2</sub> O <sub>5</sub>	0.009
Cr <sub>2</sub> O <sub>3</sub>	0.008
SrO	0.071
Cl	0.004
CeO <sub>2</sub>	0.040
Rb <sub>2</sub> O	0.005
Rh <sub>2</sub> O <sub>3</sub>	0.014

#### 3.2. X-ray Diffraction Analysis

The XRD patterns of the produced zeolite are shown in Figure 2. The XRD mineralogical data indicates the presence of some mineral phases. The limited number of minerals measured could be a result of the equipment detection limit, which could mean that low concentrations are hard to pick up by the equipment or could be due to the dominance of these mineral phases in the samples (Anumata *et al.*, 2021). Following the XRD data, the major minerals present in the zeolite are Quartz, Albite, Muscovite and Orthoclase with weight percentages of 46.6%, 4%, 19% and 29.7% respectively. These various compositions are shown in Figure 3. The qualitative analysis result is presented in Table 2. Albite, Muscovite and Orthoclase are types of aluminosilicates. Due to the sodium hydroxide used in the production process of the zeolite, albite was formed. The Quartz formed only shows excess silica present. Muscovite is the only hydrated aluminosilicate present due to the presence of hydrogen. All the elements needed for zeolite to be formed are present in

Muscovite. It shows the presence of zeolite. Muscovite also has the highest figure of merit compared to the others. This figure of merit shows how accurate the predictions are.

#### Qualitative Analysis Results

Phase name	Formula	Figure of merit	Phase ref. detail	Space Group	DB Card Number
Quartz	Si O <sub>2</sub>	0.832	S/(PDF-4 Minerals 2024)	154 : P3221	00-001-0649
Albite	Na Al Si <sub>3</sub> O <sub>8</sub>	2.181	S/(PDF-4 Minerals 2024)	2 : P-1	00-003-0508
Muscovite	H <sub>2</sub> K Al <sub>3</sub> Si <sub>3</sub> O <sub>12</sub>	2.871	S/(PDF-4 Minerals 2024)	15 : C12/c1	00-002-0055
Orthoclase	Al <sub>2</sub> O <sub>3</sub> · K <sub>2</sub> O · 6 Si O <sub>2</sub>	1.696	Import(PDF-4 Minerals 2024)	12 : C12/m1	00-002-0534

#### Phase Data View

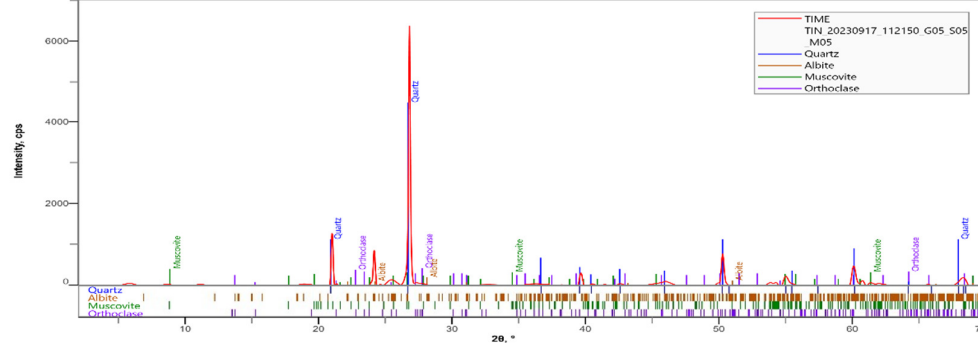


Figure 2: X-ray Diffraction patterns

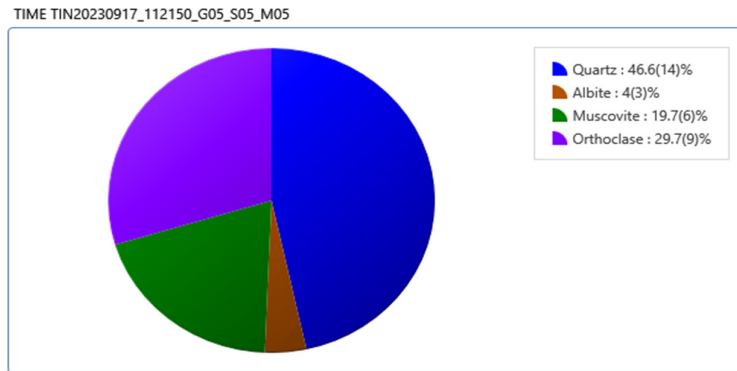


Figure 3: X-ray diffractometer analysis

Table 2: Qualitative analysis results

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Muscovite	H <sub>2</sub> K Al <sub>3</sub> Si <sub>3</sub> O <sub>12</sub>	2.871
Orthoclase	Al <sub>2</sub> O <sub>3</sub> K <sub>2</sub> O <sub>6</sub> Si O <sub>2</sub>	1.696

### 3.3: Fourier Transform Infrared Spectroscopy (FTIR) Analysis

This analysis was carried out to help know the functional groups present in the cracked product based on the peak values in the region of Infrared radiation either directly or by inference. The result of the FTIR analysis conducted is presented in Figure 4. According to Anumata *et al.*, (2021), the associated changes in the spatial arrangement of the groups involved are reflected in the infrared spectrum as additional bands and added complexity. The selected peak positions for this study are those with bands at 2956.55 cm<sup>-1</sup>, 2925.36 cm<sup>-1</sup>, 2855.61 cm<sup>-1</sup>, and 3422.30 cm<sup>-1</sup>. The three stretching peaks of 2956.55 cm<sup>-1</sup>, 2925.36 cm<sup>-1</sup>, and 2855.61 cm<sup>-1</sup> reflect the presence of alkane (C-H) as opined by Nandiyanto *et al.* (2019). The frequency 3422.30 cm<sup>-1</sup> indicates the presence of either C-H, C-N or O-H wide branched broad band which is consistent with the findings of Omoruwou *et al.* (2017). According to the Infrared Spectroscopy Absorption Table (*Infrared*

*Spectroscopy Absorption Table*, 2022), it can be deduced that the C-H functional group is predominantly present as explained in Table 3.

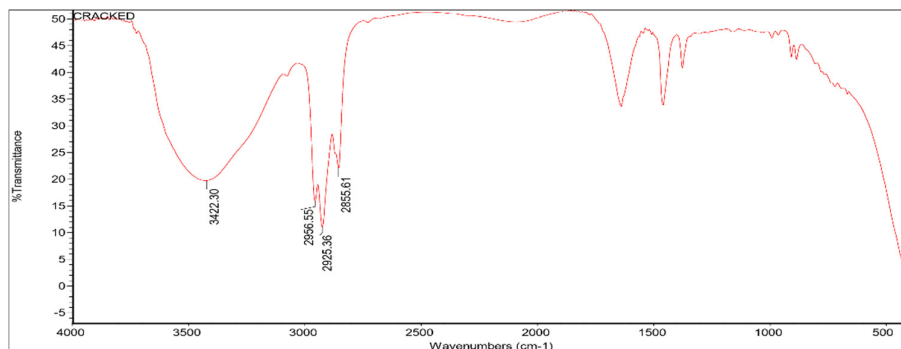


Figure 4: FTIR spectrum

Table 3: Detailed description of functional group present in the cracked product

Wavenumbers (cm <sup>-1</sup> )	Bond	Strength of bond	Nature of bond	Group
2956.55	C-H	Medium	Stretching	Alkane
2925.36	C-H	Medium	Stretching	Alkane
2855.61	C-H	Medium	Stretching	Alkane
3422.30	O-H	Medium	Wide Branded	Alcohol

### 3.4. Gas Chromatography-Mass Spectrometry (GCMS) Analysis

The components present in the cracked product were identified by GCMS. GCMS tells the compound and the exact mass produced. The chromatogram is shown in Figure 5. The active principles with the retention time (RT) and concentration are shown in Table 4 with undecane having the highest concentration. The result revealed that all compounds present in the cracked product are alkanes which is in line with the functional group present as determined by the FTIR analysis.

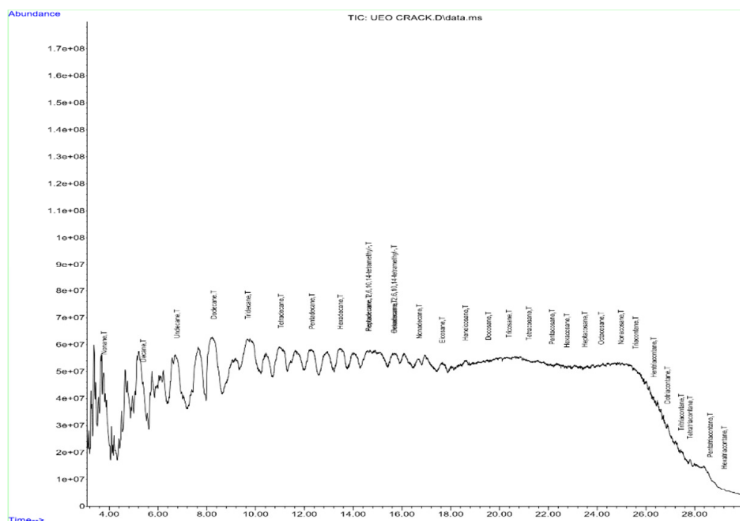


Figure 5: GCMS chromatogram

Table 4: Description of compounds present in the cracked product

Compounds	Molecular formula	Concentration (mg/l)	Retention time
Undecane	C <sub>11</sub> H <sub>42</sub>	1106.30	6.772
Tetradecane	C <sub>14</sub> H <sub>30</sub>	1080.30	11.035
Tetracosane	C <sub>24</sub> H <sub>50</sub>	1.32	21.203

### 3.5. Examining Density and Viscosity Changes in Waste Engine Oil Cracking

The density of the waste engine oil was obtained to be 0.9072 g/ml and viscosity to be 14.8 mPa.s. From previous work, this density obtained is in line with the range which is 0.700-0.950 g/ml at 15 °C (Elkhaleefa, 2016). Additionally, the density and viscosity of the cracked product were obtained to be 0.8416 and 1.74 mPa.s respectively which corroborates the work of Arpa *et al.*, (2010). The change in density and viscosity indicates a transformation in the composition of the waste engine oil during the cracking process. The decrease in density from 0.9072 g/ml to 0.8416 g/ml suggests a reduction in molecular weight, while the decrease in viscosity indicates a shift towards a less viscous and potentially more desirable product. These changes may be attributed to the breakdown of larger hydrocarbon molecules into smaller, lighter compounds during the cracking process.

### 3.6. Effect of Catalyst Load on Rate of Conversion

Before the addition of zeolite to the cracking process, Table 5 shows the cracking results obtained. From the plot, it can be inferred that as time increases, the values of  $-\ln(1 - X_A)$  also increase thereby giving a direct variation. The equation of the line is  $y = 0.0217x - 0.4861$ . Also, the slope of the graph gives the value of the rate constant,  $k$ . Hence, the value of  $k$  is  $0.0217 \text{ min}^{-1}$ . A plot of  $-\ln(1 - X_A)$  against time was obtained as shown in Figure 6. At 10%, the time reached to obtain the various volumes of the cracked product increased but there is also a direct relationship between time and  $-\ln(1 - X_A)$ . The plot is shown in Figure 7. From the plot, the equation of the line is  $y = 0.012x - 0.1814$ . Hence,  $k$  is  $0.012 \text{ min}^{-1}$ . The plot of  $-\ln(1 - X_A)$  against time is as shown in Figure 8. The equation of the line is  $y = 0.0108x - 0.1946$ . From the plot,  $k$  is  $0.0108 \text{ min}^{-1}$ , which is the slope.

Table 5: 0% Zeolite of 50 g of waste engine oil

Volume (ml)	Time (minutes)
10	48.67
15	63.32
20	77.91

Table 6: 5% zeolite of 50 g of waste engine oil

Volume (ml)	Time (minutes)	Mass of cracked (g) $C_A$	$X_A$	$-\ln(1 - X_A)$
10	30.80	8.416	0.16832	0.18430753
15	36.18	12.624	0.25248	0.29099422
20	41.23	16.832	0.33664	0.41043745

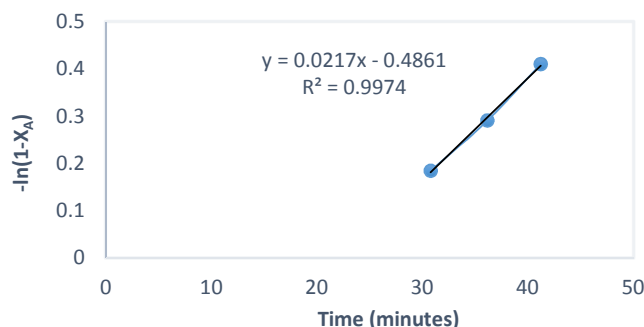
Figure 6: A plot of  $-\ln(1 - X_A)$  against time with 5% zeolite

Table 7: 10% Zeolite of 50g of waste engine oil

Volume (ml)	Time (minutes)	Mass of cracked (g) $C_A$	$X_A$	$-\ln(1 - X_A)$
10	30.45	8.416	0.16832	0.18430753
15	39.53	12.624	0.25248	0.29099422
20	49.32	16.832	0.33664	0.41043745

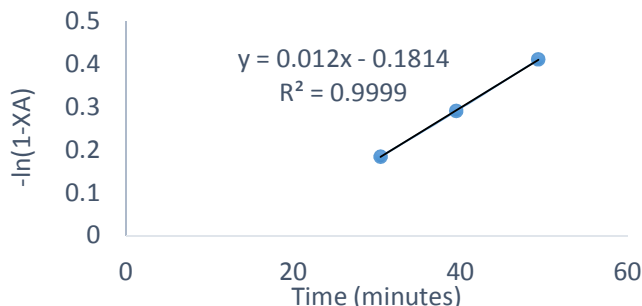
Figure 7: A plot of  $-\ln(1 - X_A)$  against time with 10% zeolite

Table 8: 15% Zeolite of 50g of waste engine oil

Volume (ml)	Time (minutes)	Mass of cracked (g) $C_A$	$X_A$	$-\ln(1 - X_A)$
10	34.40	8.416	0.16832	0.18430753
15	46.02	12.624	0.25248	0.29099422
20	55.10	16.832	0.33664	0.41043745

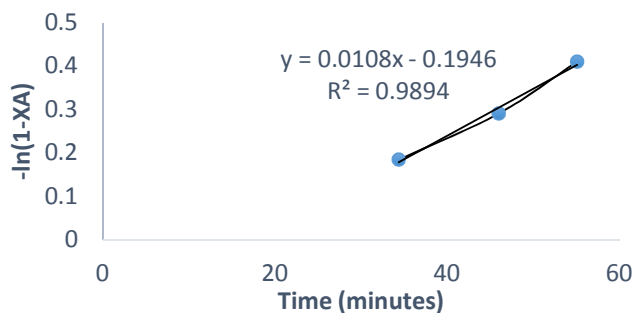
Figure 8: A plot of  $-\ln(1 - X_A)$  against time with 15% zeolite

Table 9: 20% Zeolite of 50g of waste engine oil

Volume (ml)	Time (minutes)	Mass of cracked (g) $C_A$	$X_A$	$-\ln(1 - X_A)$
10	24.98	8.416	0.16832	0.18430753
15	27.40	12.624	0.25248	0.29099422
20	29.15	16.832	0.33664	0.41043745

From the plot of  $-\ln(1 - X_A)$  against time with the addition of 20% zeolite (Figure 9), the equation of the line gave  $y = 0.0536x - 1.1615$  and the  $k$  value was obtained to be  $0.0536 \text{ min}^{-1}$ . From the plot, the equation of the line is  $y = 0.106x - 1.925$  and the value of  $k$  was  $0.106 \text{ min}^{-1}$ . The data obtained from the determination of the cracking rate is presented in Table 11. The cracking rates were obtained from plots of  $-\ln(1 - X_A)$  against the time of the various cracking processes which took place (with the respective catalyst variations). The bar chart (Figure 11) illustrates the plot of Table 11. From the plot, the rate of conversion did not consistently rise with increased catalyst load. The 5% to 15% range showed a gradual decline, reaching its lowest at 15%. The sudden spike at 20% and the subsequent sharp increase at 25% showed that the conversion rate increased with an increase in catalyst load. This demonstrates the potential for the zeolite to effectively convert the waste engine oil.



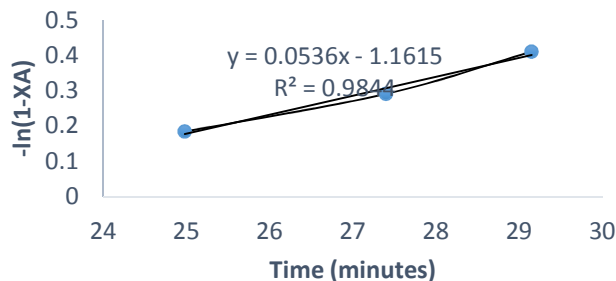
Figure 9: A plot of  $-\ln(1 - X_A)$  against time with 20% zeolite

Table 10: 25% Zeolite of 50g of waste engine oil

Volume (ml)	Time (minutes)	Mass of cracked (g) $C_A$	$X_A$	$-\ln(1 - X_A)$
10	19.82	8.416	0.16832	0.18430753
15	21.12	12.624	0.25248	0.29099422
20	21.90	16.832	0.33664	0.41043745

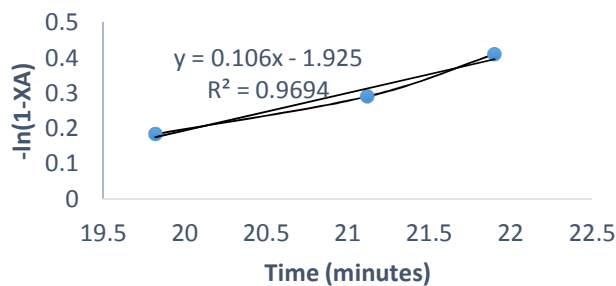
Figure 10: A plot of  $-\ln(1 - X_A)$  against time with 25% zeolite

Table 11: Cracking rates for various catalyst load

Catalyst load (wt%)	Cracking rate constant $k$ ( $\text{min}^{-1}$ )
5	0.0217
10	0.0120
15	0.0108
20	0.0536
25	0.1060

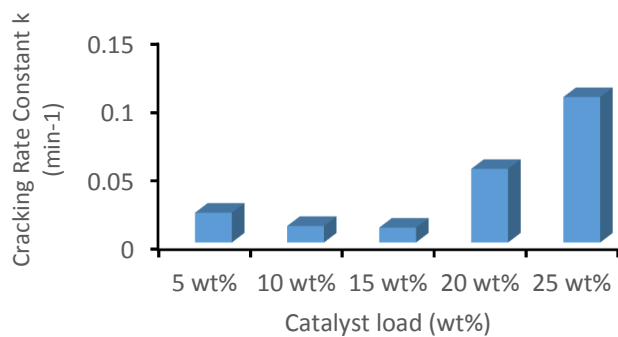


Figure 11: A plot of cracking rate constant against catalyst load

#### 4. CONCLUSION

The feedstock of this study is a readily available material in our environment and currently poses a threat to the environment due to indiscriminate disposal by most artisans. Also, clay is readily available and sustainable as it is a natural resource. The high concentration of silica and alumina in the clay which was revealed from the XRF result indicated the quality of kaolinite in the clay thereby making it suitable for the catalyst production. Four compounds were found in the catalyst, according to the XRD result: orthoclase, quartz, albite, and muscovite, with figures of merit of 1.696, 0.832, 2.181, and 2.871, respectively. Zeolite was formed due to the presence of muscovite which was the only hydrated aluminosilicate present and its high figure of merit. The catalyst was found to be effective because the rate of cracking increased when the zeolite was added. This indicated that the developed catalyst could be used to produce valuable products from waste engine oil. The characterization of the cracked product provided insights into the nature and quality of the catalytic conversion. The FTIR result showed that the C-H functional group was present in the cracked product, which confirmed that the product is an alkane, which is also in line with the GCMS result showing that only alkanes were present.

#### 5. ACKNOWLEDGMENT

The authors wish to acknowledge the assistance and contributions of the laboratory staff of the Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Nigeria, and LUCO Scientific, Benin City, Edo State, Nigeria, for various analytical work carried out in their laboratory. Their expertise and dedication were invaluable in ensuring the success of this research project. Their commitment to excellence greatly enhanced the quality of our findings and conclusions.

#### 6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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