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Application of Zeolite in the Catalytic Cracking of Waste Vegetable Oil for the Production of highly Volatile Liquid Fuel

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ABSTRACT: *In this study, zeolite was used as a catalyst in a catalytic cracking reaction of waste cooking oil, which generates compositions similar to gasoline. This reaction was catalyzed by batch distillation of WCO in a hydrothermal reactor with zeolite catalyst synthesized using local clay sediment. The catalytic cracking was carried out under ambient conditions using 100-ml distillation apparatuses placed on a heating mantle and 50g of oil-added variable catalyst loading from 2 to 10 Wt% with zeolite as catalyst. The heating rate, residence time, and catalyst loading influenced the product yield. The physicochemical properties of the product, such as the density, the flash point, and the kinematic volumetric viscosity, were examined for fuel suitability. Chemical gas chromatography-mass spectrometry (GC-MS) determined the chemical composition of the compounds present in the cracked product. The overall study showed that the product's optimum zeolite loading, cracking temperature, and cracking rate were 8 wt%, 380 °C, and 0.0048 min^{-1}, respectively. The study on zeolite catalytic cracking revealed that local clay synthesizes the zeolite catalyser, which is effective in WCO catalytic cracking for liquid fuel formation.*

KEYWORDS: catalyst, acid, biofuel, zeolite, triglycerides.

INTRODUCTION

Due to rising demand, environmental concerns, depletion of petroleum supplies, and the quality of crude oil declining, the production of renewable and environmentally friendly biofuels has gained popularity (Biswas and Sharma, 2013). Most people agree that plant oils are the most promising raw material for producing these renewable fuels, often known as biofuels (Zaher *et al.*, 2017). These vegetable oils' high viscosity, volatility, and tendency to deposit carbon in different internal combustion engine components are the main disadvantages of using them directly as liquid fuels. Bio-oils also have other undesirable qualities that make them incompatible with a range of equipment, most notably internal combustion engines. These include being intensely oxygenated, acidic, corrosive to metallic materials, chemically and thermodynamically unstable, and incompatible with fossil fuels (Yigezu & Muthukumar, 2014). The need for petroleum fuel has increased significantly over the past few years on a global scale, not just in wealthy nations but also in developing nations like Indonesia. According to the 2018 Indonesian Energy Outlook, without new resource discoveries, coal will run out in 68 years, natural gas in 42 years, and petroleum in 9 years. This is based on the 2018 R/P (reserve/production) ratio. Both in Indonesia and internationally, efforts are underway to develop biofuel as an alternative energy source. The term "biofuel" refers to a variety of biomass-based products, including solids like biochar, liquids like ethanol and biodiesel, and gases like biogas, biohydrogen, and biosynthetic gas (Genene Demisu, 2021). In Indonesia, leftover cooking oil and microalgae are two possible feedstocks for biodiesel. Because palm and sunflower oils are the most commonly used in food processing and cooking, much research on vegetable oil cracking has concentrated on these types of oils. Exploring the cracking of inedible vegetable oils, however, is becoming more popular. Plants such as jatropha, castor, and pongamia produce these inedible oils, which have shown promise as substitute sources of biodiesel and other industrial products. Researchers want to harness the full potential of these non-edible oils and support a more diversified and sustainable vegetable oil market by comprehending their breaking behavior. However, the desire to use inedible vegetable oils as food commodities necessitates cracking them in order to create biofuels (Melero et al., 2010). Cracking non-edible vegetable oils is a viable way to address the growing demand for biofuels without sacrificing food resource availability. For example, waste vegetable oil is abundant and less expensive than edible oils, making it a good candidate for conversion into biofuels. The cracking process breaks down the complex molecular structure of these oils into simpler hydrocarbons, which can subsequently serve as fuel or feedstock for a variety of industrial uses. To maximize the production of biofuels and optimize the conversion process, it is imperative to comprehend the cracking behavior of non-edible oils. The goal of this study was to evaluate the catalytic cracking of waste vegetable oil for biofuel production using zeolite catalyst extracted from nearby sediments. To fulfill the study's declared purpose, we employed the following goals.

i. Collection and pretreatment of the materials used.
ii. Synthesizing zeolite catalyst from local clay sediment and to determine the physical and chemical properties.
iii. Thermal and catalytic cracking of waste vegetable oil using zeolite catalyst in a laboratory-scale distillation experiment.
iv. characterization of physicochemical properties of the cracked oil

METHODOLOGY

Materials and method

The experiment used leftover cooking oil from a nearby restaurant in the city of Warri as the feedstock. In the Uzebba hamlet in Owan Local Government Area, Edo State, clay sediment was used to create the catalyst. Table 1 lists the equipment utilized in this investigation.

Table 1: Equipment used for the study

Property	Equipment
Kinematic Viscosity	NDJ-5SViscometer and a density bottle
Stirrer	MS300ConstantTemperatureMagneticStirrer
Drying	VacuthermVT6025Air-DryVacuumOven
Calcination	UniscopeSM9080MuffleFurnace
Flash Point	Pensky-Martens Closed Cup Flash Point Tester -Automatic NPM 440
Density	SVM300050milliliter Density Bottles
Water Bath	HH-4WaterBath
GCMS	GC-MSQP2010S SHIMADZU with AGILENT HP1MS Column, and Helium as the gas carrier.
Dynamic Viscosity	NDJ-5SViscometer
Calorific Value	C2000OxygenBombCalorimeter

Experimental procedure

To ascertain the acidity and the percentage composition of free fatty acids, used cooking oil waste was filtered and subjected to acid value analysis. Two stages of pre-treatment were carried out: dehydration and filtration.

1. Filtration***:*** this process consisted of removing the solid dirt content from the waste vegetable oil using filter paper for 24 hours.
2. Dehydration***:*** this process removes traces of water content present in the oil by drying it in an air-dry oven at 105°C in an atmospheric pressure distillation process.

Preparation and Characterization of Zeolite Catalyst

Pre-treatment of clay

A similar procedure to that described by Maciver et al. (2020) was used to pretreat the kaolin sample. A 100-µm mesh sieve and a mechanical grinder were used to crush and finely screen the raw rocky clay sample, which was anticipated to include silica and alumina. The residual volatile substance was removed from the clay powder by oven-drying it for a night at a temperature of roughly 120°C, which caused the powder to decompose and destroy the structure.

Clay Calcination and Dealumination

The process of calcining pre-treated clay involved heating 200g of the sample, which was deposited in 100 ml crucibles. The crucibles were placed in a muffle furnace and heated to about 900°C for four hours (Hartati et al., 2019). The process of calcination aids in bringing the clay's chemical constituents back to life. About 120g of calcined clay was dealuminated by mixing with 250 ml of a 60 wt% H_2SO_4 solution in a 1000 ml round-bottom flask. The mixture was then stirred with the help of a magnetic stirrer to form a homogenous mixture. The mixture was then allowed to react for about 10 minutes without external heating before being heated externally for 30 minutes. Ajayi *et al*. (2018) reported that the reaction's temperature increased from approximately 55 °C to 120 °C. Following the cessation of the reaction, the collected material was filtered, cleaned with deionized water until it reached a neutral pH, oven-dried for approximately four hours, and packaged for analysis.

RESULTS AND DISCUSSION

Physical and Chemical Analysis of Waste Cooking Oil

According to studies, high FFA lowers catalyst effectiveness and lowers production yield; hence, the recommended amount of FFA should not exceed 2 wt%. The results of the waste cooking oil analysis are displayed in Table 2. The amount of FFA in the oil was found to be 7.602%. In contrast, Bharti et al. (2019) stated that heterogeneous catalysts have proven to catalyze oil with a higher FFA content of about 6–15% without any pre-treatment of the oil. The iodine value of 108.42 I2g/100g was slightly high due to the high fatty acid composition (Zamberi and Ani, 2016).

Table 2: Physical and Chemical Properties of Waste Cooking Oil

Properties(Unit)	Waste Cooking Oil
Density (g/cm^3)	0.9362
Viscosity (mPa.s)	11.5
Moisture content (wt%)	0.85
Acid value(mg KOH/g)	15.207
FFA content (%)	7.602
Saponification value (mg KOH/g)	188.652
Iodine content (I2g/100g)	108.42
Peroxide value (mgEq/kg)	9.4

Properties of Zeolite Catalyst

The physical parameters of the zeolite catalyst are summarized in Table 3. It was noticed that the qualities resulted in considerable activity in their actions. The catalyst SEM pictures (Figure 4.1) (Buasri et al., 2013) corroborate this idea. The characteristics of the biodiesel made with a catalyst obtained from eggs are displayed in Table 3 under ideal circumstances for every variable.

Table 3: Properties of Zeolite Catalyst

Zeolite	**Value**
Surface Area	86.10 m^2/g
Bulk Density	1.285 g/cm^3
Particle Size	<100μm

A high surface area is desirable for better diffusion of reactant and product molecules, according to Jain and Sharma (2010); this observation is consistent with that of Ordóñez and Díaz (2009), who found that zeolite catalysts with slightly higher pore volumes (0.76 cm3/g) allowed reactants to diffuse easily into the interior of the catalyst (Bharti et al. 2019).

Surface Morphology of Zeolite Catalyst

Fig. 1 presents a micrograph of the catalyst from the SEM images at 1500 x magnifications. The SEM micrograph showed that the particles are twisted and rod like shape and possibly must be referenced. It is not spherical. It is worth mentioning that these particles' surfaces were rough and angular; the characteristics of the morphologies facilitated differentiating the crystal from the amorphous.

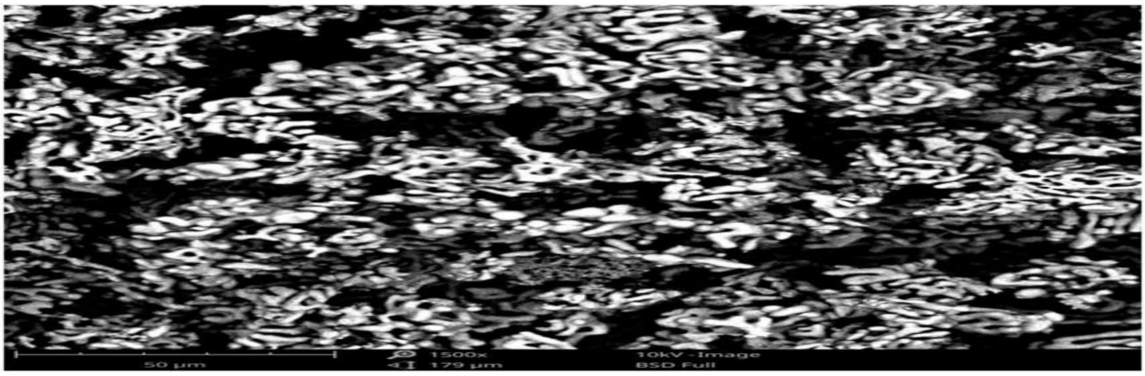
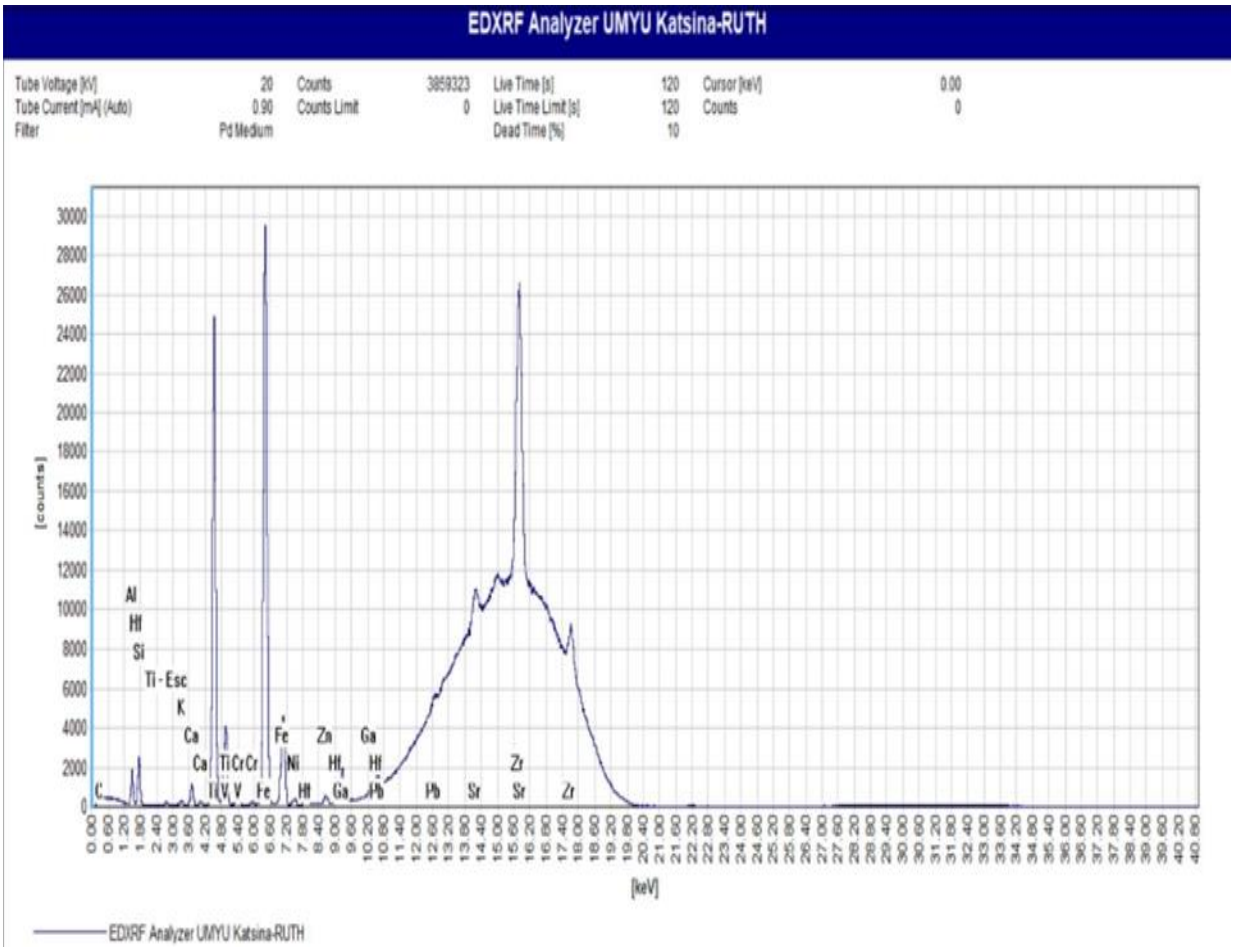


Fig. 1. Surface morphology of zeolite catalyst by Scanning Electron Microscope.

Elemental Composition of Calcined clay

Using an energy dispersive spectrometer (EDS)-equipped X-ray fluorescent (XRF) system, the elemental composition of the zeolite catalyst was determined. According to Fig. 2, the results revealed that the most prevalent element was silicon (Si) at 1.54% with a peak of 6 cps/mA, followed by aluminum (Al) at 1.3031% with peaks of 27211 cps/mA. Sodium (Na) was present at 0.4393%, with a peak of 251 cps/mA.



EDXRF Analyzer UMYU Katsina-RUTH

Tube Voltage [KV] 20
Tube Current [mA] (Auto) 0.90
Filter Pd Medium
Counts 3859323
Counts Limit 0
Live Time [s] 120
Live Time Limit [s] 120
Dead Time [%] 10
Cursor [keV] 0.00
Counts 0

Fig.2: EDXRF Spectrum of Zeolite Catalyst

Table 4: Elemental content of zeolite catalyst

Element	Concentration	Peak(cps/mA)
	Si	6
Al	1.3031 %	27211
Na	0.4393 %	251
Nb	0.296 %	29
Bi	0.175 %	2
Fe	0.11774 %	1997
Ni	0.01438 %	26
Zn	0.000861 %	39
Mg	0.03617 %	227
S	0.02723 %	406
Ca	0.07709 %	430
K	0.01755 %	97
Mn	0.001573 %	88
Rb	0.000253 %	1
Sr	0.00383 %	28
W	0.0202 %	13
Ta	0.00439 %	6
Pb	0.390 %	4

Crystallinity of Mineral Constituents of Zeolite Catalyst

An X-ray diffractometer was used to identify the solid mineral component of the zeolite catalyst. The results show the typical reflections of two types of crystal phases: the hexagonal crystalline phase in albite (NaAlSi3O8) and the cubic crystalline phase in quartz (SiO_2). Alpha = beta = gamma = 90°for lime and alpha = beta = 90^o, gamma = 120^o for orthoclase are the crystallographic parameters that verified the existence of the distinct crystalline phases.

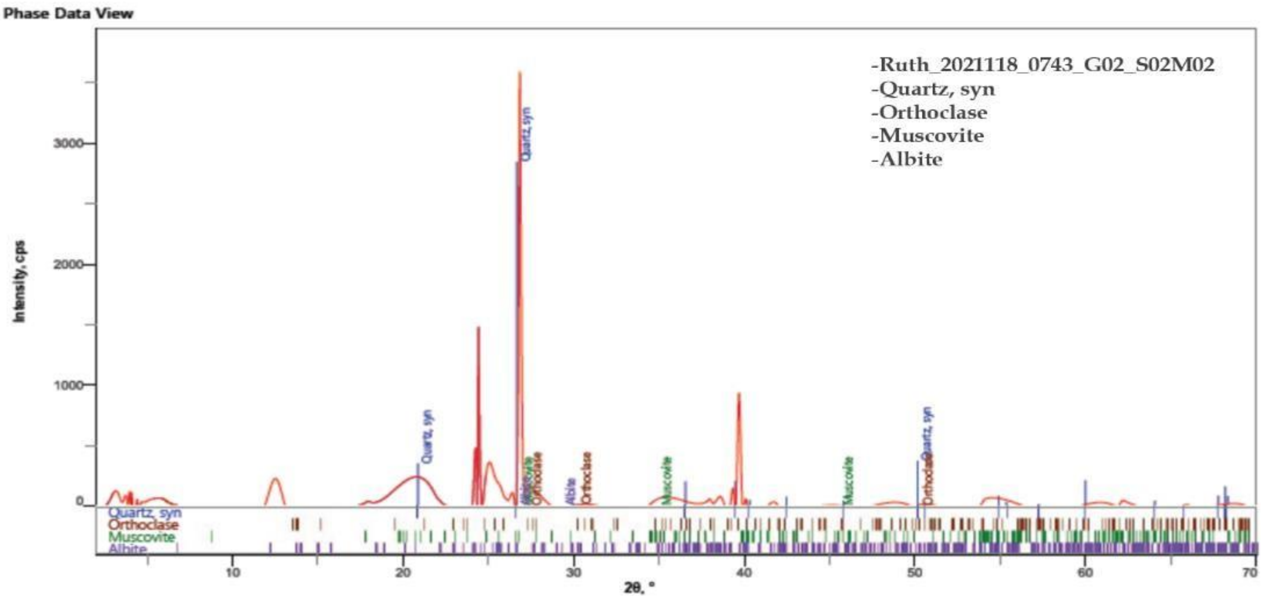


Fig. 3: XRD Analysis of Zeolite Catalyst

Table 5: Solid Mineral Constituents of Synthesized Zeolite

Compound Phase Name	Chemical Formula
Quartz	SiO_2
Orthoclase	$Al_2O_3.K_2O.6SiO_2$
Muscovite	$KAl_2(Si_3Al)O_{10}(OH,F)_2$
Albite	$NaAlSi_3O_8$

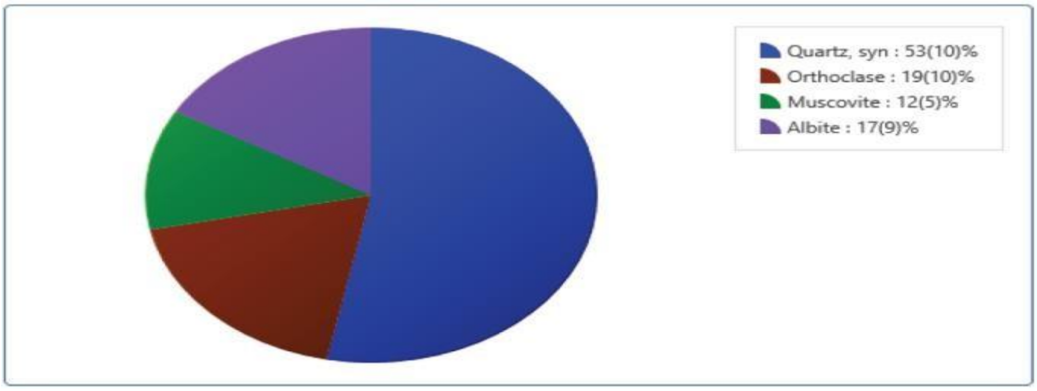


Fig. 4. Weight Fraction, wt%

Functional Groups of Calcined Zeolite

The surface functional groups that contain oxygen have a significant impact on the surface characteristics and catalytic behavior of heterogeneous catalysts (Dawood and Sen, 2014). These groups can be introduced through oxidation or formed during the activation process. Figure 4 displays the FTIR spectra obtained for the prepared zeolite catalyst. The sample displayed three major absorption bands in the 1600–1400 cm-^{1} range.

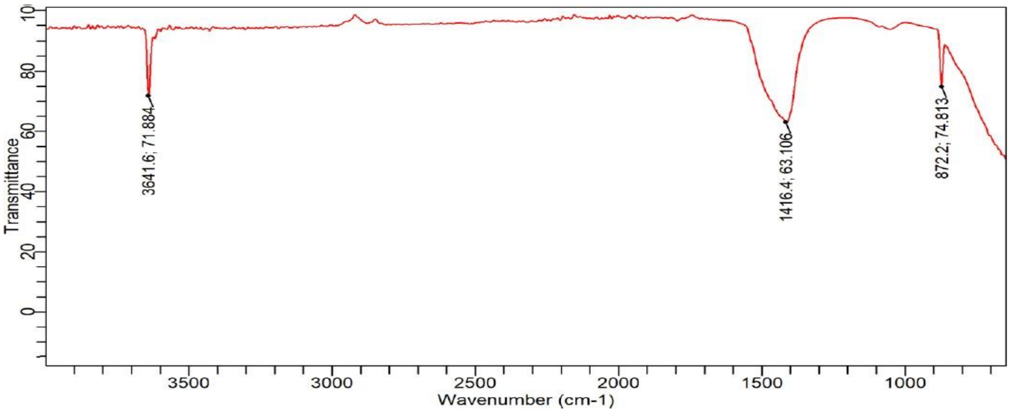


Fig. 5: FTIR spectrum analysis calcined zeolite catalyst

From Table 6, the strong band around 1416 cm1 indicates the stretching vibrations of carbonate ion (CO2) bonds, which are often found in clay deposits. Similarly, the weak peak seen at 3641 cm1 has been assigned to stretching vibrations of heterocyclicamine (N-H) bonds. The summary of the results is presented in Table 6.

Table 6: Summary of Spectrum peaks of Zeolite

Peak Number	Wave number (cm^{-1})	Intensity	Functional group	Comment
1	872.19705	74.81318	Epoxyandoxirants (C – O–)	Weak Stretch
2	1416.38838	63.10627	Carbonateion ($CO^{□2}_{3}$)	Strong stretch
3	3641.60907	71.88433	Heterocyclicamine (N – H)	Weak stretch

Results of Cracking Process

The result in Fig. 5 demonstrated the efficiency of the catalyst in the thermal breakdown of the triglyceride in leftover vegetable oil. The highest catalytic efficiency was achieved between 6 and 8 weight percent of catalyst loading. Up to 8 wt%, the conversion rate of triglyceride to biofuel rose with increasing catalyst loading; beyond that, the performance began to deteriorate. This implies that there is a spectrum of catalyst loadings that are ideal for the thermal breakdown process. To fully comprehend the underlying principles and maximize catalyst loading for industrial-scale applications, more research is required

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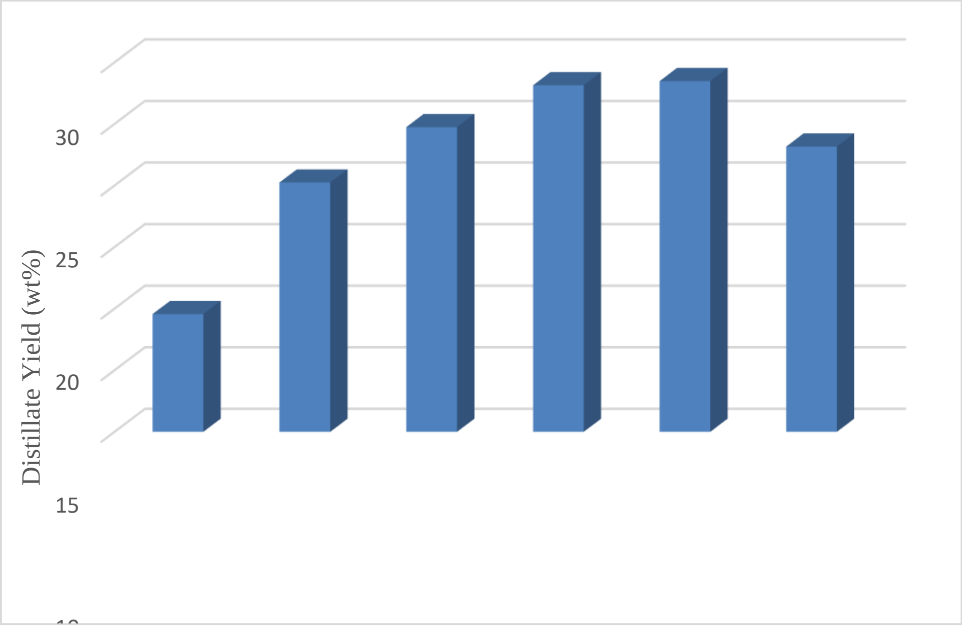

Fig.6: Crack product distillate recoveries with catalyst loading

Results of Kinetic Modelling of Cracking Process

A first order of reaction was assessed using integral method of analysis to predict the cracking rate. In the first order prediction, from Equation1, a plot of$-\ln(1-X_A)$ vstas was made, with slope k representing the rate constant. The overall rate equation (K) can be found using equation 1 for first order reaction

$$kt = -\ln(1-X_A) \qquad 1$$

Where: XA is taken as conversion of triglycerides at various catalysts loading.

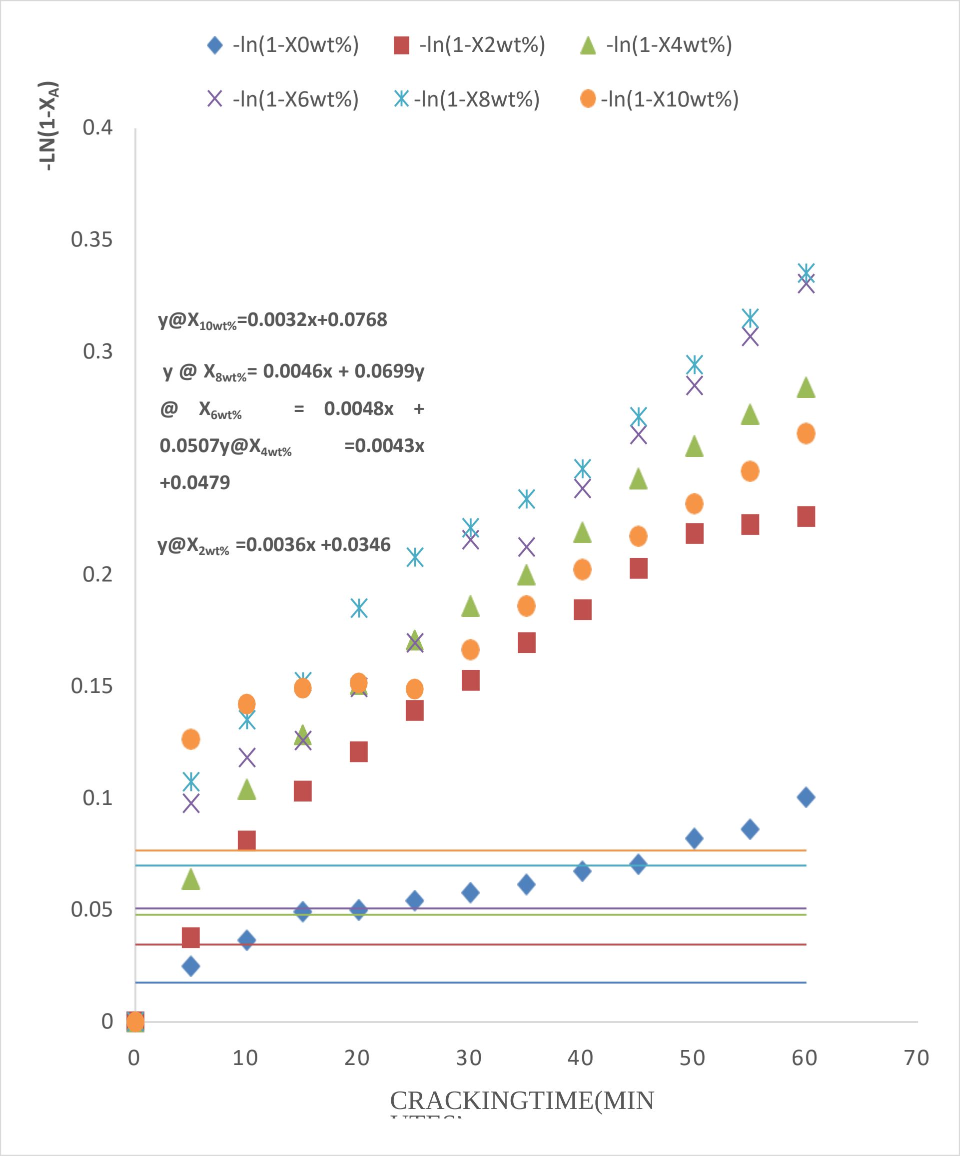

Fig. 7: First order kinetic plots of thermal decomposition of triglycerides in the presence of zeolite catalyst.

Results of first-order rate kinetics are shown in Fig. 6 for the conversion of triglycerides into liquid fuel under different catalyst loading scenarios. The graph unequivocally demonstrates that raising the catalyst loading causes triglycerides to convert into liquid fuel at a faster pace. This suggests that the catalyst is critical to the reaction's facilitation and efficiency enhancement. A further increase in catalyst loading may not considerably improve the conversion rate beyond a certain point, according to the data, which also points to an optimal catalyst loading where the conversion rate reaches its maximum.

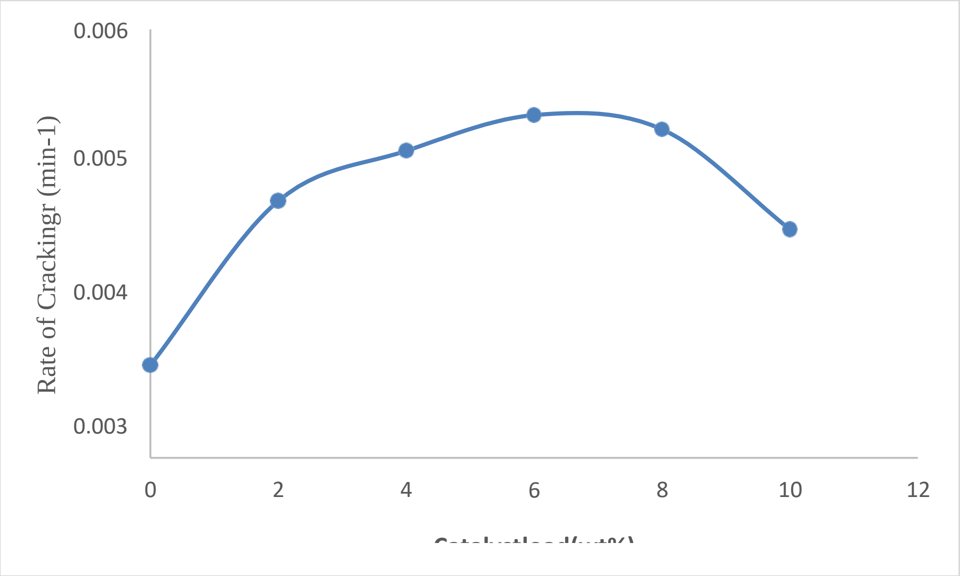

Fig. 8: Effects of catalyst loading rate of conversions of triglycerides into volatile liquid fuels

The study revealed that the rate of triglyceride conversion increased as catalyst loading increased (Fig. 4.7), reaching its peak rate at 6 wt% of catalyst. The rate of triglyceride conversion exhibited declining returns and only moderate gains beyond the optimum rate of catalyst loading. This indicates that the optimal amount of catalyst to get the best conversion efficiency is 6 weight percent. Increases in catalyst loading may potentially cause unintended side reactions or product deterioration, which would lower the process's overall efficiency.

Result of GCMS Analysis of Cracked Product

The chromatogram from the GC-MS analysis of the biofuel sample produced under ideal conditions for waste cooking oil thermal cracking in the presence of zeolite catalyst is displayed in Fig 9 displaying the numerous peaks. A particular, identifiable compound is represented by each peak. The chemicals found in the cracked distillate product are listed in Table 7 along with their respective concentrations.

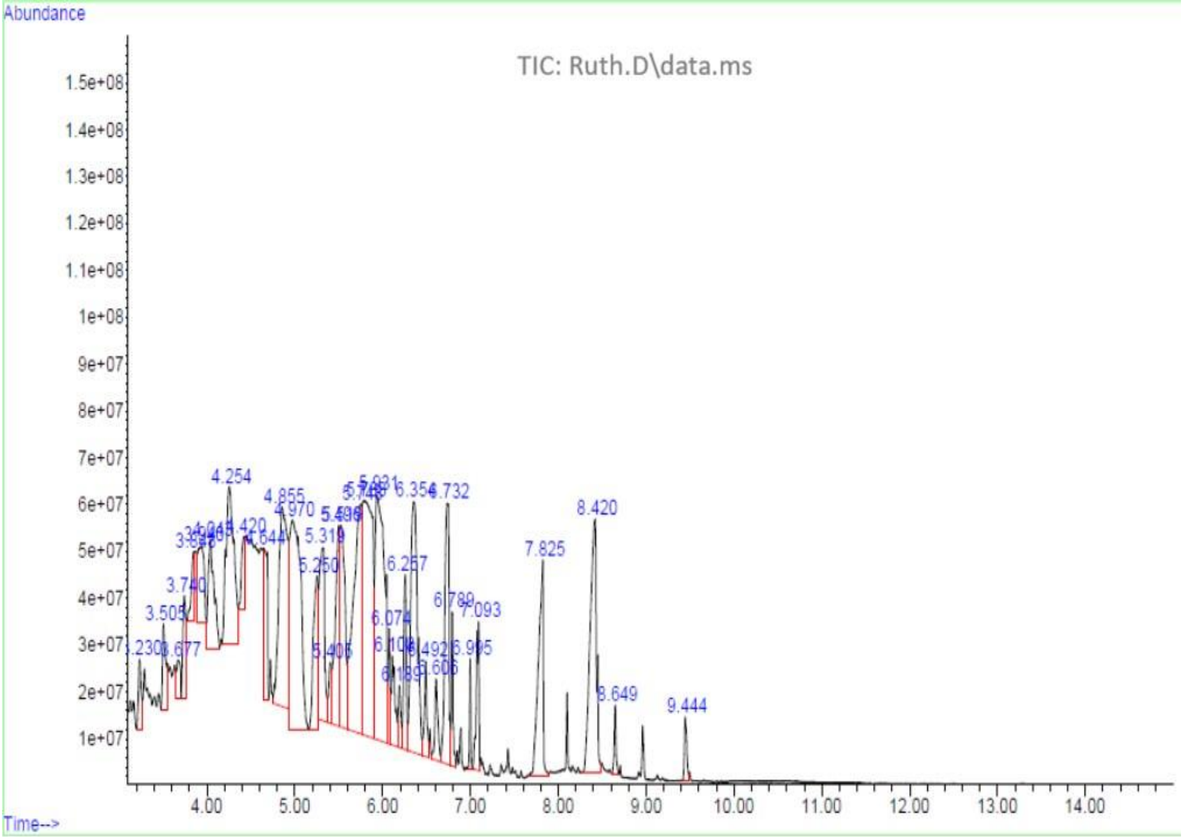

Fig. 9: 4.8 Chromatogram of Cracked Waste Cooking Oil

Table 7: Compounds present in cracked product

Retention Time (minute)	Compound Identified	Concentration (%)
3.230	4,6-Decadiene	0.75
3.505	Naphthalene,1,2,3,4,4a,8a-hexahydro-	1.02
3.677	Hexanoic acid	0.54
4.043	5-undecene	3.77
4.254	1-cyclopentane,1(Methylenecyclopropyl)-	4.81
4.420	4-dodecene	0.92
4.644	Heptanoic acid	2.18
4.855	Cyclododecene	6.55
4.970	Octanoic acid	8.58
5.250	5-undecyne	2.77
5.319	Nonanoic acid	5.319
5.405	Bicyclo[3.3.1]nonan-3-one,7-methylene	0.60
5.496	5-tetradecene	6.4
5.788	n-Decanoic acid	16.12
6.074	E-6-Octadecene-1-olacetate	0.72
6.108	2-Decylfuran	1.18
6.189	7-tetradecyne	0.54
6.257	Succinicacid,tridec-2-yn-1-yltranshexa-3-en-1-ylester	1.72
6.354	7-hexadecene	6.33
6.492	Bicycle[3.3.0]octan-2-one, 7methyl1-ene-6-methyl	0.90
6.606	1-methylbicyclo[3.2.1]octane	0.90
6.732	8-Heptadecene	4.15
6.789	Nonadecane	0.96
6.995	1-methylbicyclo[3.2.1]octane	0.48
7.093	9-octadecene	1.44
7.825	n-Hexadecanoic acid	3.81
8.420	Oleicacid	5.98
8.649	11-hexadecene-1-ol	0.48
9.444	11-hexacosyne	0.56

Table 7 indicates that the predominant chemical found in the sample's fractured triglyceride was n-decanoic acid, which was present at a concentration of 16.2%. Octanoic acid, on the other hand, was present at a value of 8.54%. Hydrocarbons comprising 5-undecene, hexacosyne, and heptadecene with concentrations of 2.77%, 4.15%, and 6.33, respectively, were among the other chemicals found in the cracked triglyceride. The residual substances were responsible for the 4.67% of the triglyceride that is still fractured. These findings suggest that a combination of hydrocarbons and carboxylic acids made up the majority of the fractured triglyceride.

Results of Cracked WCO

Table 8 shows that waste cooking oil can be thermally and catalytically cracked to create biofuel with attributes that are within the range of the ASTM diesel fuel specification. This suggests that waste cooking oil-derived biofuels could be a good substitute for conventional diesel fuel. Waste oil is efficiently converted by the thermal and catalytic cracking processes into a fuel that satisfies the requirements for use in diesel engines. This presents a viable way to lessen reliance on fossil fuels and lessen the negative environmental effects.

Table 8: Physical properties of cracked WCO

Property	Cracked WCO product	Diesel standard
Density(g/cm^3)	0.84	0.85
Dynamic viscosity(mPa.s)	4.40	15.62
Kinematic viscosity (mm^2/s)	4.77	17.04
Pour point (^{o}C)	0.69	-9.54
Flash point (%)	44.5	63.4

CONCLUSION

Catalytic cracking is a viable method for producing biofuel using and processing WCO. Zeolite was used as a catalyst to completely crack WCO without any prior pre-treatment. Based on the collective cracking reactions, the subsequent deductions are made.

1. The hydrothermal synthesis method can be used to produce a zeolite Y catalyst from local clay sediment.
1. Zeolite Y catalyst is very active in the catalyst cracking of waste cooking oil for the production of volatile liquid fuel.
2. Increasing the catalyst concentration to about 10 wt% increases the rate at which crack product distillate forms.
3. The comprehensive analysis of catalytic cracking demonstrated the catalyst's recovery. The study's findings revealed that WCO can act as a potential catalyst for biofuel production via catalytic cracking

Recommendation

The present study examines the catalytic cracking process of waste cooking oil (WCO) to produce biofuel. The findings show that the zeolite catalyst is effective in breaking down triglyceride compounds. Nevertheless, further research is suggested to investigate the use of locally abundant materials that do not require complicated preparation procedures. Investigating the use of locally abundant materials can make the process of producing biofuel from waste cooking oil more economical and ecologically friendly. This research may lead to the development of a sustainable and effective method for converting WCO into biofuel on a larger scale. Additional research into different catalysts and reaction conditions may yield important insights into refining the procedure and facilitating its use by communities and industries around the globe.

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