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Synthesis and Characterization of Li-Zeolite Catalyst for Biodiesel Production from Castor oil

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ABSTRACT

This study used lithium-modified Zeolite as a heterogeneous catalyst to make biodiesel from non-edible castor oil. The catalyst was wet impregnated with natural Zeolite and characterized by XRD, FTIR, and SEM. Castor oil was extracted and injected into a GC-MS unit for analysis, and established procedures were used to evaluate chemical properties. The catalyst's performance in the transesterification reaction was examined to determine optimal process conditions and reusability. A catalyst characterization study found that the lithium-modified zeolite catalyst selectively produces methyl esters from castor oil with 4% FFA in a batch process. The maximum biodiesel yield was 92% at 60°C, 60 min, 6:1 methanol-to-oil molar ratio, and 1.5% catalyst loading. The catalyst reusability test showed 12.8% degradation after four cycles. Under ideal conditions, biodiesel had a flashpoint of 62°C, kinematic viscosity of 1.24 cSt, and density of 0.81 g/cm^3. Fuel properties were compared to ASTM standards to verify compliance. Castor oil is a promising biodiesel source. Zeolite modified with lithium is an effective heterogeneous catalytic transesterification catalyst for methyl ester under milder reaction conditions.

Keywords: Castor oil, Biodiesel, Natural zeolite, Heterogeneous catalyst, Transesterification.

INTRODUCTION

Using a stable heterogeneous catalyst can make synthesizing fatty acid methyl esters (FAMEs) through the transesterification of vegetable oils more cost-effective and feasible. This solid catalyst can be easily extracted from the reaction mixture and reactivated in batch and continuous operations. Additionally, heterogeneous catalysts offer environmental benefits and are non-corrosive. They decrease the necessity of neutralizing the base catalyst using acids and eliminating water in

the industrial manufacturing of biodiesel, thereby reducing production expenses. However, there are several disadvantages associated with the use of homogeneous catalyzed transesterification for synthesizing biodiesel from vegetable oils. For example, using primary alcohols may lead to oil saponification. In contrast, acid-catalyzed transesterification necessitates a significant quantity of catalysts and a high ratio of alcohol to free fatty acid, leading to a slower reaction.[1]

Heterogeneous catalysts may be tailored to incorporate the necessary catalytic characteristics, hence addressing catalyst recovery challenges and enabling the implementation of continuous production processes. These catalysts facilitate the gradual conversion of triglycerides into esters, resulting in the production of biodiesel in a very cost-effective manner.[1–3] When alkali metal salts are impregnated and subsequently decomposed within zeolite cages, they form alkali metal oxide clusters that increase the fundamental properties of these materials.[4] The zeolites faujasite NaX and titanosilicate structure-10 were used in the transesterification process. The catalysts are mostly composed of alkali-cation-exchange materials. Through the process of performing ion exchange with more electropositive metals like potassium and cesium, the alkalinity of the zeolites NaX and ETS-10 was increased.[5] Natural zeolites in Sudan are found in the Nubian Sandstone Formation, located in the northern part of the country, and the Red Sea Hills region and AlGadarf state. Smaller deposits of zeolites have also been reported in other parts of Sudan, including the Darfur region.[6]

Vegetable oils are not suited for fuel due to the presence of phospholipids, free fatty acids, sterols, water, and other impurities. However, Transesterification, microemulsion, dilution, and pyrolysis are some of the minor chemical processes that are necessary for the oil.[7] In the transesterification process, a catalyst, often an alcohol base, is used to react with vegetable oil or animal fat and form the conforming alkyl esters of the fatty acid combination present in the original oil or fat.[8] Methanol is widely utilized as an alcohol mostly because of its cost-effectiveness and low water content. An enough amount of alcohol is required to tip the equilibrium of the reaction towards the creation of the desired product. The transesterification technique has been widely utilized to lower the high viscosity of triglycerides.[7] The transesterification reaction produces several benefits, such as a significant drop in viscosity by a factor of eight and a reduction in molecular weight by one-third. Additionally, it exhibits increased volatility and reduced carbon deposits when compared to vegetable oils. The resultant biodiesel possesses the characteristics of diesel fuel.[9]

Biodiesel, which satisfies the ASTM D6751 criteria, has demonstrated great potential as a sustainable and renewable energy source. More precisely, biodiesel made mostly from renewable vegetable oils or animal fats in the form of mono alkyl esters has demonstrated potential.[10] There are many benefits, such as savings from currency value, safety reasons, regional development, reduction of greenhouse gas emissions, nontoxicity, biodegradability, and non-aromatic compounds. It's considered safe storage because of its high flash point.[11] Biodiesel may be derived from a wide range of feedstocks, encompassing popular vegetable oils like sunflower, peanut, coconut mustard, soybean, cottonseed, rapeseed canola, palm, and olive oil. Additionally, biodiesel can also be created from unconventional sources such as algae, fungus, bacteria, molds, yeast, animal fats, and waste oils. Modifications to the production process may be required based on the source and quality of the feedstock.[12,13] The octane number, cloud point, and stability of the finished biodiesel are all dictated by the feedstock that is utilized. Between 70% and 95% of the raw materials utilized in biodiesel synthesis worldwide come from food oils.[14] Biodiesel obtained from these oils possesses characteristics that make it a viable substitute for conventional diesel fuel. However, this could create competition with the vegetable oil market, leading to higher costs for oils and biodiesels. The biodiesel source should fulfill two requirements as much as possible: lower production costs and a large manufacturing scale.[15]

Ricinus communis, commonly known as castor beans, is a significant non-edible oilseed plant from the Euphorbiaceae family. It is typically cultivated in arid and semiarid regions. Castor oil has been investigated for biodiesel production in recent years. Heterogeneous catalysts offer the advantages of straightforward separation, regeneration, and multiple reuses throughout the transesterification process. This characteristic

contributes to their reputation as environmentally friendly solutions. Numerous heterogeneous essential catalysts, including alkaline earth oxides, have been extensively investigated for their potential use in biodiesel synthesis.[16,17]

Biodiesel has disadvantages such as high feedstock and production costs, poor thermal performance at temperatures below 50°C, and an increase in NOx emissions by 27%.[18] Extensive research is now being conducted to create technologies that will aid in the reduction of emissions and the establishment of more economically efficient procedures. Amalia and her co-author examined the performance of Indonesian KOH/zeolite under optimal conditions, varying the contact time between 55-65°C for 5-7 h, along with catalyst concentrations ranging from 50-70%. The highest yield was obtained at a contact time of 7 h, a temperature of 55°C, and a catalyst concentration of 70%.[5]

On the other hand, Nallusamy *et al*., examined the effectiveness of Pine oil as biodiesel, and their results were compared with standard biodiesel fuel, The results indicate a slight improvement in brake thermal efficiency and a reduction in brake-specific fuel consumption for all pine-blended fuels compared to regular diesel fuel.[19]

The fundamental objective of this study is to find a heterogeneous catalyst that can be used to make biodiesel from castor oil using natural zeolite that has been treated with lithium. Additionally, the objective of this research is to determine how different process parameters affect the final biodiesel product.

EXPERIMENTAL

Chemicals and Materials

Methanol (MeOH 99.9%, obtained from Resach-lab Fine Chem Industries, Mumbai, India), and natural Zeolite collected from Wad-Kolly (AlGadaref state, East Sudan), n-hexane, and lithium hydroxide were purchased from DUSKAN Co. Ltd.

Castor oil extraction and characterization

Castor oil was extracted from castor seeds (obtained from a local market in Omdurman, Sudan) with n-hexane using a Soxhlet extractor apparatus according to the method described by Du L *et al.,*[20] However, to remove the gum in the oil, the oil was treated with sulfuric acid in an aqueous solution. 21 Gas Chromatography–Mass Spectrometry (GC-MS) was employed to examine the chemical characteristics of crude castor oil. The Shimadzu QP2010ultra GCFIDMS, which is equipped with a flame ionization detector, was utilized to analyze the fatty acid as methyl ester with a split ratio of 50. Helium was a carrier gas with a flow rate of 1.61 mL per minute. The identification of castor oil components involved comparing retention times and mass fragmentation patterns from the National Institute of Standards and Technology (NIST) library. The information regarding the fatty acid data was documented as the proportional fraction of the entire area that represents all of the peaks that have been detected.

Preparation of Li Zeolite catalyst

10 g of natural Zeolite from AlGadaref state, located in East Sudan, was collected and analyzed by XRD. The analysis revealed that the type of Zeolite is stilbite 1. Before modification, the Zeolite was activated using hydrochloric acid and treated with sodium hydroxide. In a separate process, approximately 15 g of lithium hydroxide were accurately weighed and combined with natural Zeolite. The mixture was then transferred into a 250-mL round-bottom flask and connected to a reflux condenser. To this setup, 100 mL of distilled water was added, and the resulting mixture was heated to 60°C and maintained at that temperature for a duration of 12 h using a reflux process. After that, the slurry was allowed to reach 25°C, using centrifugation, the solid components were separated from the liquid phase, and finally, by using 300 mL distilled water each component was washed and rinsed. The isolated solid material was then calcinated within a furnace, which was maintained at 450°C for seven hours, according to the previous method.[22]

Characterization of Li Zeolite catalyst

X-ray diffraction was used to determine the overall structure, content, and level of crystallinity of the lithium-modified zeolite catalyst. Scanning

electron microscopy micrographs were obtained utilizing a TESCAN VEGA3 instrument. The detection of elemental analyses was performed using Energy Dispersive Spectroscopy with an Oxford EDS detector. Shimadzu, Japan, successfully obtained the FTIR spectrum of the catalyst with a high level of detail, achieving a resolution of 2 cm^{-1} within the range of 500-4000 cm^{-1}.

Transesterification of the Castor oil (Biodiesel production)

The conversion of castor oil into biodiesel was achieved by introducing a blend of methanol and LiOH/zeolite catalyst into the oil at a temperature of 60°C. Various oil-to-methanol molar ratios were used to carry out the transesterification process (ranging from 1:3 to 1:12), various reaction periods (30, 60, 90, and 120 min), a range of temperatures (55 to 70°C), and different concentrations of catalyst (1, 1.5, 2, and 2.5% (w/v%). A three-hour purification procedure including methanol washing and subsequent drying at 300°C was used to evaluate the catalyst's reusability. This procedure effectively eliminated any impurities present.

Characterization of the biodiesel

The final products were isolated via vacuum filtration and transferred to a separation funnel. Following a thorough washing with distilled water and petroleum ether, the top layer, which was composed of methyl ester, was dehydrated with anhydrous sodium sulfate. Subsequently, the biodiesel was analyzed by measuring its viscosity, density, octane number, cloud and pour points, distillation characteristics, flash and combustion points, fatty acid composition, and higher heating value following ASTM standard methods (ASTM D4052, ASTM D97, ASTM D93, ASTM D445, and ASTM D2500.

RESULTS AND DISCUSSION

Properties of the Extracted Castor Oil

The seeds were used to extract castor oil, resulting in a yield of 31%. This results in the range of extracted oil found in the literature.[1] These results suggest that the oil holds potential as a viable biodiesel feedstock. Table 1 shows the oil's physicochemical properties and fatty acid composition. The acid content in the oil was 8%w, and free fatty acids were 4%w. This number of fatty acids leads to the formation of soap that can lower the yields of esters.[5] Viscosity is a crucial characteristic that determines the quality of fuel oil. The castor oil sample has a kinematic viscosity of 171.50 mm^2/s at a temperature of 313 K. Vegetable oil has a high viscosity, but the maximum allowable viscosity for diesel fuel according to the ASTM is between 10 and 20,2.7 mm^2/s. Due to its high molecular mass, castor oil has a very thick viscosity. Hydroxyamide and Ricinoleic acid in castor oil are the reasons for this high viscosity and density. The viscosity obtained is lower than the findings reported in the prior study.[10] The water content is a crucial physicochemical aspect that can significantly impact the transesterification process and ultimately affect biodiesel yield. High water content in the feedstock can prompt an unintended oil hydrolysis, forming free fatty acids. Subsequently, in the presence of a base catalyst, these free fatty acids can lead to a saponification reaction, generating soap[23]. Hence, to prevent such unfavorable reactions, the feedstock utilized in biodiesel production must remain either devoid of water or contain a water content below 0.3 wt%.[6,24] There was very little moisture in the castor oil that was utilized for this research about 0.0109%, making it appropriate for the transesterification process. Castor oil is predominantly composed of monounsaturated ricinoleic acid, which accounts for 71% of its total fatty acid composition. Other minor acids are also present in castor oil, as shown in Table 1 and Table 2. However, compared to the results reported in earlier studies by Lixiong Du *et al.*,[20] The castor oil used in this investigation has a reduced concentration of ricinoleic acid, measuring 71.8%, in comparison to the 88.7% reported by Lixiong Du *et al.*,[20] Additionally, it displays elevated amounts of stearic acid, measuring 3.27% as opposed to 2.1%. However, the amount of palmitic acid is nearly the same.

Ricinoleic acid $C_{18}H_{34}O_3$, a monounsaturated fat, is the primary component of castor oil features. In addition to having high density, viscosity, and hygroscopicity, it contains several other physically

and chemically distinctive characteristics. To determine whether oil is suitable as a raw material for biodiesel, it is necessary to analyze its composition. This is because the oil content directly influences the properties of the biodiesel produced. The distinctive composition of castor oil is attributed to its high ricinoleic acid concentration.[14]

Table 1: Physiochemical properties of castor oil

Property	Method	Value
Specific gravity at 15°C	ASTM D79	0.96
Viscosity at 40°C	ASTM D445	171.5
Water content, % (w/w)		0.0109
Acid value		8
FFA content, % (w/w)		4
Flash point, °C	ASTM D93	208
Pour point, °C	ASTM D97	-14
Cloud point, °C	ASTM D2500	-10

Table 2: Fatty acid composition of castor oil

Fatty acid common name	Molecular formula	w/w %
Stearic	$C_{18}H_{36}O_2$	3.27
Palmitic	$C_{16}H_{32}O_2$	2.73
Palmitoleic	$C_{16}H30O_2$	1.21
Elaidic	$C_{18}H_{34}O_2$	9.93
Ricinoleic	$C_{18}H_{34}O_3$	71.80
Linoleic	$C_{18}H_{32}O_2$	11.5

Characterization of the catalyst

XRD results

The lithium-modified Zeolite exhibited a change in crystallinity level to 11%, accompanied by a rise in amorphous content to 89%. The reduction in crystallinity seen during the catalyst manufacture is attributed to the alkali activation of LiOH, which leads to the cessation of Si-O bonds and the commencement of nucleation.[25] The matched phase analysis, depicted in Fig. 1 through XRD patterns, highlighted that the natural Zeolite belongs to the stilbite type. However, the XRD pattern indicated the existence of 12 different matching peaks that are exclusive to lithium oxide hydrated H_3LiO_2, making up 86% of the phases. Having a unit cell with dimensions (a = 7 Å, b = 8 Å, c = 3 Å, and β = 110°), and other morphology exhibited a monoclinic crystal structure and were calculated to have a density of 1.56 g/cm³. Table 3 provides a more detailed breakdown of the remaining phases, which consist of 11.4% monoclinic zabuyelite, 2.6% monoclinic $C_{25}H_{21}Li_6N_3O_{14}$, and carbonated lithium oxide.

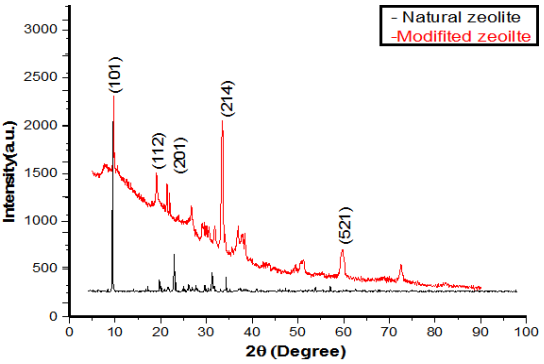

Fig. 1. XRD pattern of natural zeolite and Li-Zeolite catalyst

Table 3: Modified natural zeolite catalyst XRD data

Peaks	2-theta	Miler index	Height	d-space	FWHM	D(nm)
1	9.7	(101)	1443	9.09	0.0811	-
2	19	(112)	898.4	4.64	0.5	237.5
3	21.3	(201)	814	4.05	0.187	107.34
4	33..4	(214)	1263.2	2.67	0.452	208.2
5	59.9	(521)	352	1.54	1.112	92.4

SEM results

Characteristics of the surface morphology of the pure zeolite and the catalyst can be seen in Fig. 2(a) and (b), respectively. Clusters can be observed on the surface of Zeolite, as illustrated in (a), which signifies the crystalline nature of the parent zeolite. In addition, the absence of these clusters in (b) indicates structural damage, transitioning the Zeolite from a crystalline to an amorphous state. Consequently, this alteration leads to the heightened alkalinity of lithium hydroxide. This shift diminishes the original Zeolite's crystallinity, primarily attributed to lithium's complete coverage of pores and surfaces. This results in the formation of particles, and the crystal appears to be agglomerated after modification.

Fourier transform infrared

Lithium-modified zeolite catalyst functional groups were investigated using FTIR spectroscopy. The zeolitic octahedral metal oxide is shown by the distinctive signal of NH_4 bending vibrations at 1400 cm^{-1} in the spectrum seen in Fig. 3. The presence of a distinct peak at 1639.5 cm^{-1} indicates that the zeolite sample has not been adequately dehydrated and indicates that the water molecule deformation mode is present. Furthermore, the broad band observed at 3400 cm^{-1} refers to the OH-stretching of H_2O inside the zeolite channels.[7]

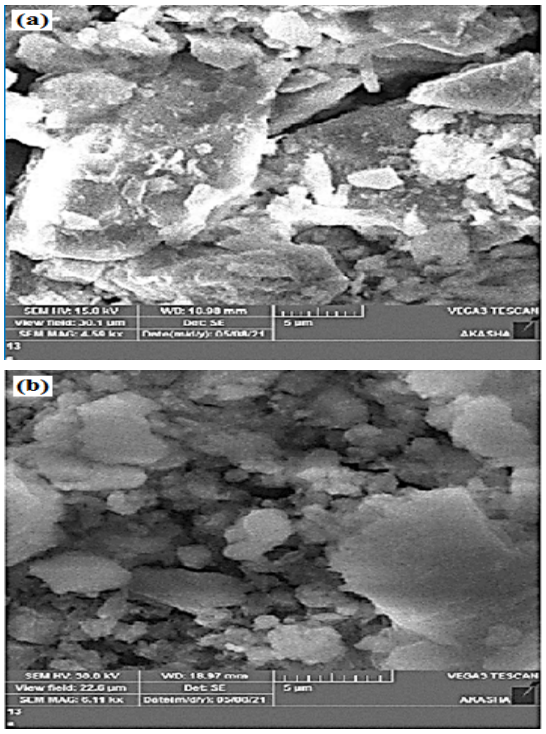

Fig. 2. SEM of (a) pure zeolite and (b) a catalyst composed of lithium and zeolite

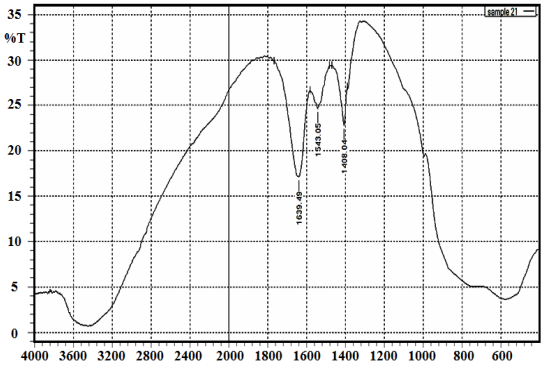

Fig. 3. Prepared Li-zeolite catalyst FT-IR spectra

Reaction conditions optimization

The impact of methanol concentration

The conversion of triglyceride molecules by transesterification into biodiesel is dependent on the methanol to oil ratio. The stoichiometric molar ratio, illustrated in Fig. 4, involves alcohol/ triglyceride 3:1, resulting in 3:1 alkyl esters and glycerol. Given the reversible nature of the transesterification reaction, an increased amount of alcohol is necessary to shift the equilibrium toward alkyl ester production, the primary component of biodiesel.

The study investigated ratios ranging from 3:1 to 12:1, using a 1% (by weight) concentration of a lithium-modified zeolite catalyst at a temperature of 60°C and a reaction duration of 60 minutes. Fig. 4 showed a positive relationship between the molar ratio of methanol to oil and the output of biodiesel this agreed with Verma & Sharma's study[26]. Achieving a methanol-oil molar ratio of 6:1 resulted in the highest biodiesel production, reaching a maximum of 90%. Nevertheless, a molar ratio of 3:1 resulted in a mere 18% FFA conversion and further increases in the molar ratio led to a reduction in biodiesel yield under consistent experimental conditions. Therefore, a molar ratio of 6:1 of methanol to oil was selected for making further modifications to other parameters in the practical activities.

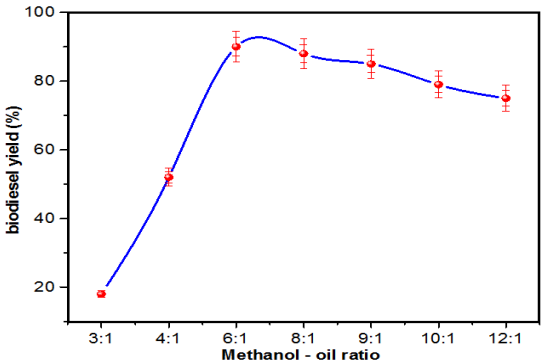

Fig. 4. Influence of methanol/oil molar ratio on the biodiesel yield

The impact of temperature

The study investigated the influence of temperature on Free Fatty Acid (FFA) conversion, as illustrated in Fig. 5. The temperature varied between 55 and 70°C while maintaining consistent conditions such as a molar ratio of 6:1 of methanol to oil, a catalyst loading of 1.5%, and a reaction time of 1 hour. According to the experimental findings, FFA conversions were found to be 90%, 92%, and 69% at 55°C, 60°C, and 70°C, respectively. The relationship demonstrated an increasing trend in biodiesel yield up to an optimum temperature of 60°C, achieving a 92% yield. Nevertheless, a noticeable decrease was found after this stage, dropping to 69% at a temperature of 70°C. Considering the endothermic characteristics of the transesterification reaction, the first heating process is of utmost importance. In systems with distinct components, such as the mixture of oil, methanol, and catalyst, thermal energy is crucial for overcoming barriers to diffusion across the various phases. Conversely, too high temperatures are undesirable. The quick

vaporization that occurs as the temperature near the boiling point of methanol causes a numerous bubble, which hinders the reaction that occurs at the interface between the two phases and reduces the amount of biodiesel that is produced.[27]

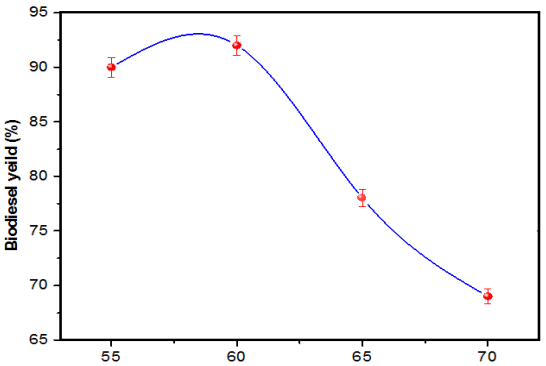

Fig. 5. Impact of temperature on the biodiesel yielding

The impact of catalyst concentration

The study explored the influence of catalyst concentration under optimum conditions a 6:1 molar ratio methanol/oil, 60 min reaction duration, and 60°C reaction temperature. A different catalyst concentrations of 1%, 1.5%, 2%, and 2.5% were examined. The findings revealed that a catalyst concentration of 1.5% yielded the maximum at 91%. Beyond 1.5% LiOH concentration as a catalyst, an increase resulted in more soap formation and a decrease in yield.[9]

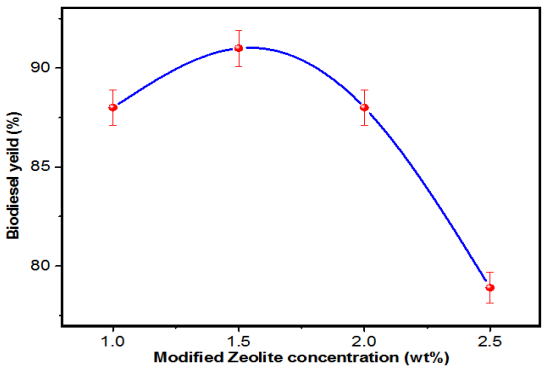

Fig. 6. Effect of modified Zeolite concentration on yielding of biodiesel

The function of the catalyst

The study examined the functionality of a catalyst consisting of lithium-modified Zeolite. Three consecutive cycles of applying this modified Zeolite catalyst resulted in a 12.8% drop in castor oil output. Following three instances of catalyst utilization, biodiesel production resulted in a yield of 79.2%. The catalyst particles, on the other hand, exhibited agglomeration following their recycling in the transesterification process, leading to a decrease in catalytic activity. Despite the decrease in trend %, treatment with catalyst regeneration can better tolerate a more extended period of activity. The same trend was described by Karabas.[28]

Properties of produced biodiesel

Table 4 demonstrates that the flash point is a highly important characteristic that determines the flammability of combustible substances, such as fuels, which can be ignited and explode. As a result, the flash point is necessary in order to properly handle, store, and secure flammable liquids and fuels, as well as to restrict the amount of unreacted alcohol that is present in the final fuel. The flash point of a flammable liquid is determined by its vapor pressure and is measured at an ambient temperature that may create a gas combination with air. In comparison to the criteria established by ASTM 6701, the density and viscosity of biodiesel that is created from castor oil as recorded in Table 4 compared to that recorded by Vashist and Ahmed[29], which findings corresponded to a density of 0.905 g/cm^3 compared to 0.81 in this study, also the kinematic viscosity of 12.5 cSt compare that were measured 1.24 cSt. The flash point was found to be 115°C compare to 62°C. These results are more closely to the diesel properties. The reduction in density and viscosity can be attributed to a decrease in the fraction of ricinoleic acid containing OH group, as compared to the prior research by Sanchez *et al.,*[30]. These outcomes were closer to the characteristics of diesel.

Table 4: Physical properties of produced biodiesel at 25°C

Physical properties	Yield
Viscosity	1.24 cSt
Density	0.81 g/cm^3
Flashpoint	62°C
Pour point	-30°C
Cloud point	0.5 °C
Water content	0.0197 wt%

It has been seen that using nano catalysts in the production of biodiesel has proven to be more efficient than bulk catalysis for several reasons. These include an enlarged surface area, improved thermal stability, and heightened catalytic activity. Erchamo *et al.,*[31]. Successfully enhanced biodiesel production from waste cooking oil by employing

egg-shell nanoparticles as an effective catalyst. Under favorable conditions, they achieved a yield of 94%. These conditions involved a molar ratio of 1:12 for oil to methanol, a catalyst loading of 2.5 wt.% at 60°C, and 120 minutes.[31]

CONCLUSION

This research involved the preparation of a catalyst by modifying Zeolite with lithium, aiming to facilitate the transesterification of castor oil with methanol. The resulting catalyst exhibited notable catalytic activity, attributed to its complex pore structure that enhanced the diffusion of small molecules like methanol. This structural complexity played a pivotal role in the successful synthesis of biodiesel. Furthermore, the catalyst demonstrated impressive reusability. Analysis through XRD patterns indicated that the synthesized catalyst possessed 11% crystallinity, which belongs to the monoclinic crystal form. SEM imaging revealed the impregnation of lithium ions in the zeolite structure. The castor oil composition showed a predominantly monounsaturated ricinoleic acid content of 71%, alongside other minor acids. To prevent saponification, early esterification was necessary, due to the elevated levels of Free Fatty Acid (FFA) in castor oil.

The heterogeneous transesterification of castor oil was investigated in this study, which focused on several aspects, including the molar ratio, reaction duration, temperature, catalyst concentration, and catalyst reusability. The study highlighted the significant effect that these components have on the process. The highest percentage of biodiesel production, reaching 92%, was achieved under the following ideal conditions: a methanol to oil ratio of 6:1, a reaction period of 60 min, a temperature of 60°C, and a catalyst concentration of 1.5%. These results indicate that the lithium-modified zeolite catalyst holds promise for efficient biodiesel synthesis. The research faces many challenges and limitations, such as scale-up challenges, studying the catalyst's stability, and characterizing it with advanced techniques such as TEM and NMR.

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Conflict of interest

The authors of this paper affirm that they are free from any ties or conflicts of interest that may have affected the results or interpretation of their work.

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