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Production of Sustainable Biodiesel from Sunflower Oil over Libyan Kaolin Clay Catalyst

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Biodiesel, Sunflower oil, Libyan Kaolin, Meta-kaolin, Heterogeneous catalysis, Transesterification.

ABSTRACT

This study investigates the conversion of sunflower oil to biodiesel as an eco-friendly alternative to fossil fuels using heterogeneous catalysts derived from Libyan Kaolin clays (Agar Ashatti region). The catalyst was thermally activated at 900 °C to produce meta-kaolin, enhancing its catalytic activity. Transesterification reactions were performed under operating conditions of a 20:1 methanol-to-oil ratio, 5 wt % catalyst loading, and a temperature of 70 °C for 4 hours. Results indicated that meta-kaolin achieved a high average conversion efficiency of 91.09%, outperforming raw kaolin at 87.83%. The produced biodiesel was characterized and compared with ASTM D6751 standards, demonstrating high quality with a kinematic viscosity of ~3.8 cSt and a heating value of 40.56 MJ/kg. The study concludes that Libyan Kaolin is a cost-effective and efficient catalyst for sustainable biodiesel production.

انتاج الديزل الحيوي المستدام من زيت دوار الشمس باستخدام محفيز من الكاولين الليبى

عبد الوهاب أبوعوينة[1]، فتحية عمرمبارك[2]، محمد العباسي[1]، عائشة العباسي[3]

تناول هذا البحث دراسة تحويل زيت عباد الشمس إلى ديزل حيوي (Biodiesel) كبديل صديق للبيئة للوقود الأحفوري، وذلك باستخدام محفزات غير متجانسة مشتقة من طينات الكاولين الليبية (منطقة أقار الشاطئ). تم تحضير المحفز وتنشيطه حرارياً عند 900 درجة مئوية للحصول على الميتا-كاولين (Meta-kaolin) لزيادة نشاطه الحفزي. أُجريت تجارب تفاعل الاستبدال الاستري (Transesterification) عند ظروف تشغيلية شملت نسبة ميثانول إلى زيت 20:1، ووزن محفز بنسبة 5%، عند درجة حرارة 70 درجة مئوية لمدة 4 ساعات. أظهرت النتائج أن الميتا-كاولين حقق كفاءة تحويل عالية بلغت في المتوسط 91.09%، متفوقاً على الكاولين الخام الذي سجل 87.83 .%كما تم توصيف الديزل الحيوي الناتج ومقارنته بالمعايير العالمية (ASTM D6751)، حيث أثبت جودة عالية من حيث اللزوجة الكينماتيكية (cSt 3.8) والقيمة الحرارية 40.56 . (MJ/kg) تخلص الدراسة إلى أن الكاولين الليبي يمثل محفزاً فعالاً ومنخفض التكلفة لإنتاج وقود حيوي مستدام.

INTRODUCTION

The global energy crisis and the rapid depletion of fossil fuel reserves have become major challenges to sustainable development (Sufe, 2026). In addition to the finite nature of petroleum resources, the extensive use of fossil fuels has resulted in serious environmental problems, including increased emissions of greenhouse gases such as carbon dioxide and sulfur oxides, which contribute significantly to global warming and climate change (Hannun & Razzaq, 2022; Mohamed, Betiha, & Negm, 2023; Salim, 2022). Consequently, the development of renewable and environmentally friendly energy resources has become an international priority (Lin, Cunshan, Vittayapadung, Xiangqian, & Mingdong, 2011).

Among the available renewable fuels, biodiesel has attracted considerable attention because it is biodegradable, non-toxic, and can be produced from renewable feedstocks such as vegeet al oils and animal fats (Lin et al., 2011). Compared with conventional diesel fuel, biodiesel can reduce the overall carbon footprint by as much as 75% owing to its participation in the natural carbon cycle. Sunflower oil is considered one of the most promising feedstocks because of its high oil content, widespread availability, and suitability for transesterification reactions (Ozili & Ozen, 2022; Patil & Waghmare, 2025). Biodiesel is commonly produced through the transesterification of triglycerides with short-chain alcohols, typically methanol, in the presence of a catalyst to produce fatty acid methyl esters (FAME) and glycerol as the main by-product(A. Al-Abbasi, 2025; Aisha Al-Abbasi et al., 2023; Al-abbasi A., Belkher N., Ahmida K., & M., 2023; Almahdi, 2024; Salim, 2022).

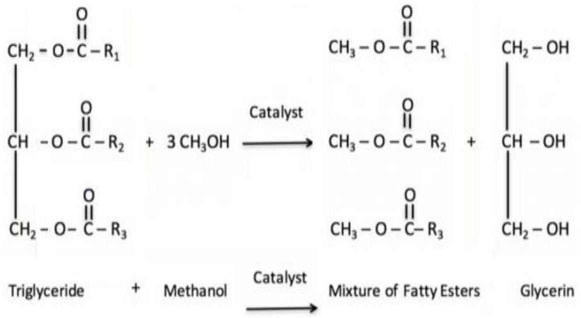

Fig. (1): Transesterification Reaction

Although homogeneous catalysts provide high biodiesel yields, their separation from the reaction mixture, catalyst recovery, and wastewater generation remain significant drawbacks. Consequently, increasing attention has been directed toward heterogeneous catalysts because of their simple separation, reusability, lower operating costs, and reduced environmental impact. Among these materials, kaolin has emerged as an inexpensive, abundant, and efficient catalyst support for biodiesel production. Various modification strategies, including alkali impregnation, thermal activation, hydrothermal treatment, plasma treatment, and nanostructuring, have successfully enhanced the catalytic performance of kaolin, achieving biodiesel yields generally exceeding 90% from sunflower oil, soybean oil, palm oil, waste cooking oil, and other vegeet al oils (Abukhadra, Mostafa, El-Sherbeeny, Ahmed Soliman, & Abd Elgawad, 2020; A. Al-abbasi, and Shana, I., 2021; Dang, Chen, & Lee, 2013; Ebrahimi, Haghighi, Ghasemi, & Bekhradinassab, 2024; Jalalmanesh, Kazemeini, Rahmani, & Zehtab Salmasi, 2021; Jalalmanesh S, 2020; Mohadesi, Aghel, Gouran, & Razmehgir, 2022; Purwanto, 2025; J. Ramirez-Ortiz, J. Medina-Valtierra, & M. M. Rosales, 2011; Widayat, Okvitarini, & Philia, 2020). These studies clearly demonstrate that catalyst composition and surface characteristics play a decisive role in improving biodiesel yield, catalyst stability, and reusability.

Southern Libya possesses abundant clay deposits, particularly in the Agar Ashatti region near Sebha. Previous investigations conducted by the Industrial Research Center (IRC) have demonstrated that these deposits represent an important mineral resource with significant industrial potential. X-ray fluorescence (XRF) analyses confirmed that the clay satisfies the ASTM C618-03 specifications, with a kaolin content exceeding 50% and suiet al silica and alumina contents for catalytic applications. Despite these favorable characteristics, the catalytic potential of Libyan kaolin for biodiesel production has received very limited attention.

Therefore, the present work investigates the feasibility of producing biodiesel from sunflower oil using catalysts derived from Libyan kaolin. Particular emphasis is placed on optimizing the transesterification conditions, including catalyst loading, reaction temperature, and reaction time. In addition, the physicochemical properties of the produced biodiesel are comprehensively characterized and compared with the ASTM D6751 specifications to evaluate its suitability as an alternative diesel fuel.

MATERIALS AND METHODS

Materials and Chemicals

Commercial sunflower oil was used as the primary feedstock for biodiesel production. The catalyst used was prepared from raw kaolin extracted from the “Agar Ashatti” region in Libya (Fathyah Omar Imbarik, 2024). The chemicals used in the activation and conversion processes included high-purity methanol, 2.0 N sulfonic acid, and ethanol used as a precipitating agent, in addition to distilled water for washing and purification (Hosseini, Niaei, & Salari, 2011; Potdar, Jun, Bae, Kim, & Lee, 2007).

Preparation and Synthesis of the Catalyst

The process of preparing and characterizing kaolin samples involved treating the raw material extracted from the “Agar Ashatti” area in southern Libya. The steps began with grinding and sieving the raw material to obtain a fine particle size of less than 0.5 mm, followed by a thermal activation (calcination) stage at a temperature of 900 °C for two hours in an electric furnace to convert the kaolin into the more active “meta-kaolin” phase (Fathyah Omar Imbarik, 2024). To extract the alumina, 20 g of metakaolin was mixed with 80 ml of 2.0 N sulfonic acid solution in a 250-ml beaker, and the mixture was stirred and continuously heated at 70 °C for 3 to 6 hours using a magnetic stirrer and a reflux condenser; After the reaction was complete and the solution was cooled and filtered, it was gradually added at a rate of 5 ml/min to 550 ml of ethanol, which acts as a precipitating agent to form aluminum sulfate from the ionic solution. The precipitate was then washed with distilled water and dried at 70 °C for one hour, followed by final calcination at 900 °C for two hours to ensure the stability of the desired catalytic structure (Fathyah Omar Imbarik, 2024).

Biodiesel Synthesis

Biodiesel production experiments were conducted using a three-neck flask, in which 0.936 g of kaolin catalyst (equivalent to 5% of the oil weight) was mixed with 172 ml of methanol and 218 ml of vegeet al oil to achieve a molar ratio of 20:1. The process began by mixing the kaolin with the methanol and treating them in an ultrasonic device to ensure homogeneity of the mixture and activate the catalytic surface before adding the vegeet al oil and initiating the reaction under heating and magnetic stirring while maintaining a constant temperature of 70 °C for four hours to complete the esterification reaction. Once the specified time had elapsed, the mixture was centrifuged at 2000 rpm for five minutes to separate the solid residue, followed by the use of a separating funnel to isolate the biodiesel phase from the glycerin and excess methanol. The ester layer was then purified by washing it with hot distilled water in three consecutive stages to remove all remaining impurities. The process concluded by drying the samples in an oven to remove moisture and weighing the final product to determine the yield and analyze its physical and chemical properties to assess its quality.

RESULTS AND DISCUSSION

The Libyan kaolin clay from the Agar Ashatti was previously investigated by means of chemical analysis and X-ray diffraction (XRD), and it was found that kaolinite is the major mineral phase. The kaolin contained about silica (SiO_2, 48.01%), alumina (Al_2O_3, 33.53%), loss on ignition (L.O.I., 12.91%), iron oxide (Fe_2O_3, 2.15%), titanium oxide (TiO_2, 1.38%), potassium oxide (K_2O, 0.98%), calcium oxide (CaO, 0.98%), and sodium oxide (Na_2O, 0.11 (Fathyah Omar Imbarik, 2024).

Biodiesel Characterization by FT-IR

Figure A compares the FTIR spectrum of crude sunflower oil and that of the obtained sunflower biodiesel after transesterification. This comparison is also the most significant spectroscopic evidence for the successful transformation of the triglycerides in the vegeet al oil to fatty acid methyl esters (FAMEs), which make up the largest part of biodiesel.

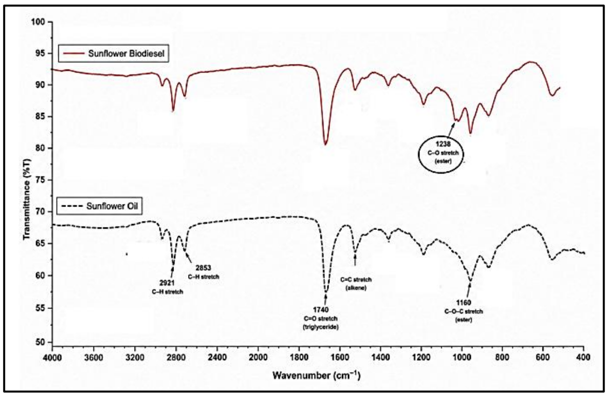

Fig. (2): FT-IR spectra of sunflower oil and its biofuel

FT-IR Analysis of Sunflower Oil

The sunflower oil (dashed black line) spectrum has several peaks that are typical of vegeet al oils. Intense peaks are observed at 2921 cm^{-1} and 2853 cm^{-1} from the asymmetric and symmetric stretching of C–H bonds in the methylene (-CH_2-) and methyl (-CH_3) groups along the long hydrocarbon chains of the fatty acids. At 1740 cm–1, a strong, distinctive and reliable peak can also be observed, ascribed to the vibration of the carbonyl (C=O) group in the triglyceride molecules. This peak can be utilized as a measure of the amount of vegeet al oil in its natural state. There is a signal too at c. 1650 cm^{-1} related to C=C bonds from unsaturated fatty acids, a natural constituent of sunflower oil. The peak at 1160 cm^{-1} is assigned to C-O-C vibrations of the ester group in the triglyceride.

FT-IR Analysis of Sunflower Biodiesel

After the transesterification reaction, the biodiesel spectrum (red curve) shows clear changes confirming that the desired chemical transformation has occurred. Although C–H peaks continue to appear in the 2950–2850 cm^{-1} region, indicating the persistence of long hydrocarbon chains, there are noticeable changes in the ester region. The carbonyl (C=O) peak remains present because the ester group does not disappear during the reaction, but the surrounding chemical environment changes as a result of the conversion of triglycerides to methyl esters. One of the most important pieces of evidence for the success of the conversion is the appearance and intensification of signals in the 1240–1170 cm^{-1} region, where a distinct peak appears at 1238 cm^{-1} attributed to the C–O vibrations of the resulting methyl esters. This change is considered one of the important spectral signatures confirming the formation of biodiesel.

Structural Modification After Transesterification

In fact, in comparing the two spectra, the peak around 1238 cm^{-1} appears clearly in the Sunflower Biodiesel spectrum, and it is missing or not clear in the spectrum of crude sunflower oil. This peak has been assigned to the stretching vibrations of the C–O bond associated with the fatty acid methyl ester (FAME) moiety, the chemical moiety produced directly from the transesterification reaction.

During the process of reaction, the fatty oil triglycerides are converted to methyl esters, and glycerol is one of the byproducts. This process changes the surrounding environment of the ester group and generates a new absorption peak or enhanced absorption peak at about 1238 cm^{-1}. Hence, the manifestation of this signal in the biodiesel spectrum and its attenuation or disappearance in the original oil spectrum is unequivocal evidence for esterification of the fatty acids.

Conversion Efficiency and Activation Effect

The reaction was conducted under constant operating conditions (temperature 70 °C, time 4 hours, catalyst concentration 5%) to ensure the accuracy of the comparison. The results in Et al 1 clearly demonstrate the superiority of metakaolin.

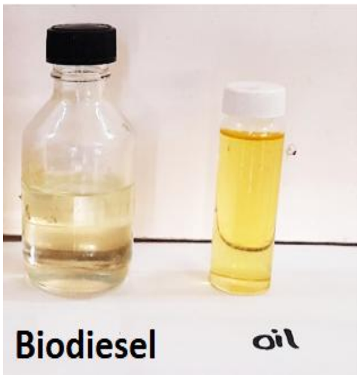

Fig. (3): Physical appearance of biodiesel

Scientific Analysis of Results: Meta-kaolin recorded an average conversion efficiency of 91.09%, while raw kaolin recorded 87.83%. This superiority is attributed to the fact that the thermal activation process creates structural disorder in meta-kaolin, leading to an increase in surface area and the generation of more efficient active acidic sites. In the esterification reaction, the solid catalyst activates methanol molecules to form methoxide ions that attack the triglyceride molecules in the oil. Due to the open and irregular structure of meta-kaolin, the reaction rate increases and the yield of fatty acid methyl esters (FAME) improves (J. Ramirez-Ortiz, J. Medina-Valtierra, & J. Rosales, 2011).

Et al (1): Catalyst Performance and Biodiesel Conversion Efficiency.

Run	Catalyst	Efficiency %	Mean
1	kaolin	88.09	87.83
2	kaolin	87.85	
3	kaolin	87.54	
4	meta-kaolin	93.75	91.09
5	meta-kaolin	92.3	
6	meta-kaolin	87.21	

Catalyst Stability and Reusability

Catalyst reusability is the cornerstone of economic viability in the catalytic industry. Scientific Analysis: The results in Et al 2 show a 4.11% decrease in the efficiency of meta-kaolin upon reuse, while raw kaolin maintained its stability and even recorded a slight increase (0.64%). The decrease in metakaolin's efficiency may be due to "pore clogging" caused by byproducts such as glycerin, or the loss of some active sites as a result of direct interaction with free fatty acids (de Oliveira et al., 2021; Ramamurthy, 2016). The stability of raw kaolin, on the other hand, reflects the rigidity of its crystalline structure, which was not affected by the first reaction cycle, thereby preserving the available reaction sites without any fundamental change.

Et al (2): Catalyst Performance after reusability

Run	Catalyst	Efficiency %
1	Reused kaolin	88.47
2	Reused meta-kaolin	86.98

Biofuel Quality Evaluation (Physicochemical Quality Evaluation)

Compliance of the physicochemical properties with international standards (ASTM D6751) is the determining factor in the fuel's acceptance for motor use (Et al 3).

Density: The density of the resulting biodiesel was 0.9183 g/cm³ at 15 °C for both the kaolin and metakaolin catalysts. Although this value is slightly higher than the standard range specified in ASTM D6751, which is between 0.875 and 0.900 g/cm³, it represents a significant physical change compared to the heavier crude sunflower oil. This slight increase in density can be scientifically explained by the 91.09% conversion efficiency obtained using the metakaolin **catalyst;** the presence of a small proportion of monoglycerides and diglycerides intermediate in the product typically contributes to a higher overall density than that of pure methyl esters. However, this was the result predicted by FT-IR spectroscopy, which confirmed the complete conversion of large triglyceride molecules into smaller fatty acid methyl ester (FAME) molecules.

Kinematic viscosity: The recorded values (3.8 cSt) were highly successful, falling squarely within the permissible range (1.9–6.0 cSt). The reduction in viscosity from the high value of crude oil to this value confirms the breakdown of large triglyceride molecules into lighter methyl esters, ensuring efficient injection and combustion within the engine.

Cold Flow Properties: The pour point was approximately -17 °C. This value is considered excellent compared to other types of biodiesel, indicating that the resulting fuel will not freeze or clog filters when used in cold regions, giving it a competitive advantage. Sulfur content: The result was nil, making it a clean fuel that does not produce toxic sulfur oxides, which is a key objective of green energy.

Et al (3): Biodiesel Analysis Report vs. ASTM Standards.

Test	Kaolin	Meta-Kaolin	ASTM Standard
Density @ 15 °C, g/cm³	0.9183	0.9183	0.875–0.900
Kinematic Viscosity @ 40 °C (cSt)	3.8092	3.8167	1.9 – 6.0
Kinematic Viscosity @ 100 °C (cSt)	8.646	8.615	No direct ASTM limit
Total Sulphur % wt.	Nil	Nil	≤0.05%
Pour Point °C	-17	-17	-3 to -15
Cloud Point °C	+2.2	+2.2	-3 to +12

Calculation of Calorific Value

This value represents the energy stored in the fuel's chemical bonds that is released during combustion. The study relied on the Dulong equation to estimate the calorific value, a quick and approximate method used to calculate the heat of combustion based on the elemental composition of the material.

$$\text{Calorific Value} = CV = \left(\frac{O}{8} - H\right) 1.442 + C \times 0.3383$$

This equation is primarily based on the mass fractions of the three main elements in biodiesel: Carbon (C), Hydrogen (H), and Oxygen (O).

To arrive at accurate figures, the research relied on the known composition of sunflower oil, which is rich in unsaturated fatty acids, particularly linoleic acid (omega-6), which constitutes 55–70% of the oil, followed by oleic acid at 20–35% [47, 48]. When this oil is converted into biodiesel (methyl esters), the approximate elemental composition of the product is as follows:

- Carbon: ranges from 76% to 78%.
- Hydrogen: ranges from 11% to 12%.
- Oxygen: ranges between 10% and 12%.

Based on the average of these ratios (carbon 77%, hydrogen 11.5%, oxygen 11.5%), the study concluded that the calculated calorific value was approximately 40.56 MJ/kg.

$$CV \approx 40.56\ (MJ/Kg)$$

This value falls squarely within the typical global range for vegeet al oil-derived biodiesel, which usually ranges between 37 and 42 MJ/kg. This result confirms that biodiesel produced from sunflower oil has a high energy content, making it an effective and powerful alternative to conventional petroleum diesel (which typically ranges between 42–45 MJ/kg), as it provides the energy needed to power engines efficiently (Adhikesavan, Ganesh, & Charles Augustin, 2022).

.CONCLUSION

This study concludes that the production of biodiesel from sunflower oil using heterogeneous Libyan clay catalysts represents a promising and sustainable approach to reducing dependence on fossil fuels and supporting global trends toward clean energy. Practical experiments have demonstrated the high efficiency of these catalysts extracted from the Agar Ashatti region, particularly when using the metakaolin phase, which achieved an average conversion rate of 91.09%, outperforming raw kaolin, which recorded a rate of 87.83%, under optimal operating conditions, including a temperature of 70 °C and a reaction time of 4 hours. This success is attributed to the structural properties and active surface area of the thermally activated catalyst, which facilitated the esterification process and the production of biofuel with superior technical specifications.
Furthermore, the results of physical and chemical characterization confirmed that the produced biodiesel complies with international standards (ASTM D6751), as it exhibited optimal kinematic viscosity and flow quality, in addition to a high calorific value of approximately 40.56 MJ/kg, making it a competitive fuel in terms of energy content and completely environmentally friendly due to its sulfur-free nature. Furthermore, the study demonstrated the economic and practical feasibility of using Libyan kaolin as a low-cost, structurally set al catalyst, with the potential to be reused for several reaction cycles without a significant decrease in its catalytic activity, thereby providing a solid scientific foundation for the development of industrial production processes that rely on local natural resources to support the circular economy and sustainable development.

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