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Valorizing Seed Oils for Sustainable Biodiesel Production via Transesterification Process

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ABSTRACT

Biodiesel is a blend of mono-alkyl esters utilized as an alternative to traditional diesel fuel. It is produced by transesterifying vegetable oils or animal fats with light alcohol. This study valorizes two Euphorbiaceae plants, *Jatropha curcas* L. and *Ricinus communis* L., which can thrive in the most arid lands and withstand harsh weather conditions. Their mature seeds can produce a significant amount of vegetable oils through a simple heating process in methanol, resulting in biodiesel with a shallow sulfur content. Selecting these two plants as energy crops is justified by their readily available seeds containing non-edible high-energy-value oils with properties comparable to diesel. The encouraging findings show that the resulting biodiesels closely resemble petrodiesel in fuel characteristics, making them suitable substitutes for fossil diesel, meeting ASTM D6751 and EN 14214 standards.

KEYWORDS
Alternative fuels, euphorbiaceae, green revolution, renewable biofuels, sustainability, vegetable oil

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1. Introduction

The demand for biodiesel as a viable alternative to conventional fossil fuels and natural gas has increased drastically over the last few years, negatively affecting its price. Based on data from the International Energy Agency (IEA), the worldwide biofuel market has seen a 6% increase in 2022 compared to the previous year, resulting in a volume of 9,100 million liters per year (Razak *et al.*, 2024, Elboughdiri *et al.*, 2023). This growth is primarily attributed to the rising consumption of renewable diesel, which constitutes the highest proportion of this annual expansion (Razak *et al.*, 2024). As a result, the search for alternative renewable (organic materials) and sustainable (low-carbon) energy sources, including biofuels, has gained significant importance in most countries (Rosak-Szyrocka *et al.*, 2023, Anekwe *et al.*, 2023). Lignocellulosic plant biomass is a promising alternative to petroleum, as it is a biodegradable renewable energy source, and its production does not contribute to the increase in greenhouse gases. Lignocellulosic biomass refers to plant or plant-derived matter not used for human consumption or animal feed, mainly including agricultural residues, crops for energy production, and waste biomass (Nanda *et al.*, 2015).

Ricinus communis L. is a shrub native to Africa. The ideal growing parameters for castor beans include loamy to sandy loam soils alongside a temperature range of 20 to 30°C (Naik, 2018, Pina *et al.*, 2005). Nevertheless, this species has the potential to acclimatize, including tropical, subtropical, and semi-arid regions, demonstrating a remarkable capacity to withstand harsh environmental factors such as elevated temperatures and limited water resources. Castor oil contains a high percentage of ricinoleic acid, which imparts excellent lubricity and oxidative stability, making it ideal for biodiesel applications. Additionally, castor plants require minimal agricultural inputs, grow in marginal soils, and are not directly competing with food crops, addressing the food *vs.* fuel debate. At present, the oil derived from the seeds of this plant possesses a wide range of applications (Saadaoui *et al.*, 2017, Chakraborty and Chatterjee, 2020, Tenorio-Alfonso *et al.*, 2019, Neme *et al.*, 2022). These applications encompass various uses such as hydraulic oil, color driers, emulsifiers, varnishes, pharmaceuticals, organic soil amendments, biological pest control, and the manufacture of polymers and dyes (Glüge *et al.*, 2020, Thombare *et al.*, 2022). Additionally, it is worth noting that biodiesel production is another prominent utilization of this oil (Öner and Altun, 2009, Zhu *et al.*, 2023).

Jatropha curcas L., another oil-bearing plant species native to Central and South America, has been spread worldwide, especially in African and Asian countries (Taddese, 2014). *J. curcas* (Euphorbiaceae) can reach a height of up to 8 meters in specific regions and has a lifespan of over 50 years. The seeds of *J. curcas* are non-edible and contain 21% oil and 79% unsaturated fatty acids (UFAs). Its oil is particularly rich in UFAs, which enhance the cold flow properties of biodiesel, a critical attribute for operational efficiency in low-temperature conditions. Moreover, *J. curcas* thrives in drought-prone and nutrient-poor soils, requiring minimal water and fertilizer inputs, making it an economically and environmentally sustainable choice for biodiesel production.

The two cultivars selected for this study have the main agronomic advantage of being resistant to drought and semi-arid climates. They can also grow in relatively poor soils. Irrigation is not a problem for them as they are low-water-demanding species and require minimal fertilization and maintenance (Chakraborty and Chatterjee, 2020, Resul *et al.*, 2012, Mouahid *et al.*, 2017, Lateef and Ogunsuyi, 2021, Kibazohi and Sangwan, 2011).

Compared to first-generation biodiesel feedstocks, such as palm or soybean oils, which are associated with deforestation and high water usage, and second-generation sources, such as waste oils or animal fats, which face limitations in availability and oxidative stability, *J. curcas* and *R. communis* oils offer superior agronomic and chemical properties for sustainable biodiesel production.

Transesterified biodiesel made from *J. curcas* L. and *R. communis* L. oils could solve some problems with biofuels made from other

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sources, like first-generation oil, used cooking oils, and animal fat waste. These alternative sources have exhibited limitations regarding cold flow properties and oxidative stability, leading to storage complications (Keera *et al.*, 2018, Tapanes *et al.*, 2008). This research aims to formulate biodiesel from *J. curcas* and *R. communis* vegetable oils using the transesterification technique, transforming free fatty acids and triglycerides (TGs) into methyl esters (MEs) and glycerol. The resulting MEs were characterized and tested as biodiesel in diesel engines.

2. Experimental

The vegetable oils (from *J. curcas* L. and *R. communis* L.) were extracted, purified, and subsequently underwent various analyses to determine their physicochemical characteristics, including density, viscosity, water content, refractive index (RI), acid value (AV), saponification value (SV), and iodine value (IV). The analyses used the French Technical Standards (Memon *et al.*, 2024, Patil *et al.*, 2024).

2.1. Extraction and Purification of the Studied Oils:

The seeds were sorted, separated, and dried in a well-ventilated shaded area for 48 hours. They were then oven-dried at 80°C for 12 hours to remove residual moisture before grinding into a fine powder using a mortar and pestle. The powdered seeds (50 g) were placed in an extractor cartridge, and petroleum ether (250 mL, solvent-to-seed ratio 5:1 w/v) was used as the solvent. The extraction was performed using a Soxhlet apparatus at a constant temperature of approximately 60°C (near the boiling point of petroleum ether) for 6 hours. After extraction, the solvent-oil mixture was collected in a flask and evaporated using a rotary evaporator set to 50°C under reduced pressure. Based on seed weight, this process yielded approximately 36.5% and 40.2% oil for *J. curcas* L. and *R. communis* L...

2.2. Oil Extraction Performances:

Immediately after the extraction, both oils were centrifuged twice at 1300 rpm for 16 minutes each to remove debris.

2.3. Transesterification reaction:

Transesterification is the chemical conversion of ester molecules into different ester molecules by exchanging alkyl groups. This reaction is commonly used in biodiesel production, where TGs (esters) are converted into FA alkyl esters (biodiesel) and glycerol. Potassium hydroxide (KOH) is a catalyst commonly used in transesterification reactions. It helps to speed up the reaction and improve its efficiency. The optimal amount of KOH and methanol required for the transesterification reaction depends on various factors, including the type of feedstock, desired conversion rate, and reaction conditions. The catalyst concentration used was 1.5% (w/w) of the total oil weight. The molar ratio of methanol to oil used in the reaction was 6:1. The reaction temperature was set to 60°C, and the reaction time was 3 hours to ensure complete transesterification. The yield of the reaction was calculated using Equation (1):

$$R(\%) = \frac{m_b}{m_{oil}} \times 100 \quad (1)$$

where: m_b is the mass of biodiesel, and m_{oil} is the mass of oil.

2.4. Preparation of Date Palm Kernel Ash (DPKA):

Date palm kernels were soaked in water for 24 hours, thoroughly rinsed to remove impurities, and finally air-dried for a few hours (Dalila *et al.*, 2024, Badawi *et al.*, 2023, Manzoor *et al.*, 2023). The kernels were pulverized using a grinder to obtain a homogeneous mixture. Subsequently, the mixture was incinerated in an oxidizing atmosphere at 900°C using a muffle furnace of this type (Nabertherm B180, Germany) until complete combustion of the organic matter occurred. The diameter of the date palm kernel ash (DPKA) particles is approximately 100 µm. DPKA is chosen as a catalyst for its eco-friendly nature, low cost, and rich content of alkaline oxides like potassium oxide (K_2O), which are effective in catalyzing the transesterification reaction. DPKA offers several advantages over conventional catalysts, such as its sustainable production from agricultural waste and its ability to reduce the environmental impact of biodiesel production. Compared to chemical catalysts like KOH, DPKA has been shown to provide comparable biodiesel yields under milder conditions, making it a promising alternative for green biodiesel production. These ashes are used as natural catalysts in the transesterification reaction.

3. Results

3.1. Physicochemical properties of *Jatropha curcas* and *Ricinus communis* oils:

Vegetable oils, such as those of *J. curcas* and *R. communis*, exhibit diverse physicochemical properties essential for various applications. These properties encompass specific gravity (or density), kinematic viscosity (typically determined at 40°C), calorific value (CV), AV, SV, and RI. The specific gravity provides insights into the density of the oils, influencing their behavior in different environments, particularly in biodiesel production, where it affects mixing with alcohol and separation efficiency. Kinematic viscosity indicates the oil flow characteristics, which are crucial for their processing in engines or industrial machinery, influencing the efficiency of biodiesel production and other chemical applications. CV denotes the energy content of the oils, directly impacting their potential as biofuels and renewable energy sources. A higher CV indicates more excellent energy content, making the oils suitable for use in alternative fuels. The AV shows the oils' free fatty acid content, which affects their stability and suitability for consumption or industrial use. Oils with a low AV are more stable and less prone to oxidation, enhancing their shelf life and usability in food or cosmetics. SV measures the oils' average molecular weight, influencing their functionality in soap production, where a higher SV indicates better soap formation potential. RI provides information on the oils' optical properties, facilitating their identification and quality assessment in various applications, such as cosmetics and pharmaceuticals. Understanding these physicochemical properties is fundamental for optimizing the utilization of *J. curcas* and *R. communis* oils across diverse sectors, including energy, agriculture, and healthcare. The physicochemical properties of *J. curcas* and *R. communis* oils are listed in Table 1.

Table 1: Physicochemical features of *Jatropha curcas* and *Ricinus communis* oils.

Parameter	*J. curcas* oil	*R. communis* oil
Volumic mass (kg/m^3)	899.2	855
Carbon residue	0.64	-
Cetane index	51.0	53.0
Flash point (°C)	240	224
Distillation point (°C)	295	310
Sulphur (%)	0.13	-
CV (kJ/kg)	39926.38	40883.95
Pour point (°C)	8.0	2.6
Fusion point (°C)	-10	-12
Kinematic viscosity at 40°C(cSt)	50.73	226.2
Solidifying point (°C)	2.0	-10
SV (mg KOH/g)	192.64	174.6
IV (gI_2/100g)	97.65±1.10	87.03±3.50
Ester index	184.1±0.71	172.7±1.34
RI at 30°C	1.470	1.473
Impurities (%)	4.4331	1.0882
AV (mg KOH/g)	8.54±0.20	1.90±0.007
Palmitic acid (%)	4.2	1.8
Stearic acid (%)	6.9	0.78
Oleic acid (%)	43.1	4.2
Linoleic acid (%)	34.3	3.7
Ricinoleic acid	0.00	87.7
Other acids (%)	1.4	1.8

CV: calorific value, SV: saponification value, IV: iodine value, RI: refractive index, AV: acid value.

3.2. Biodiesel Synthesis (Transesterification Reaction):

3.2.1. Catalyst characterization

The contents of magnesium, phosphorus, sodium, potassium, and calcium were analyzed using X-ray fluorescence (XRF). The percentage of these elements in mg per 100 g sample was determined, as shown in Table 2.

Table 2: Mineral Composition of the Natural Catalyst (Date Palm Kernel Ash).

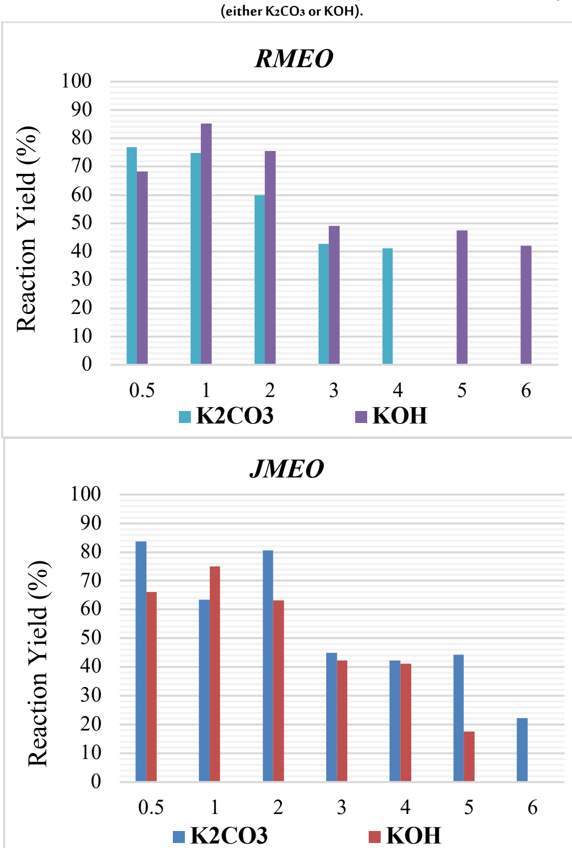
Mineral elements	Mg	P	K	Na	Ca
Content in (mg/100g of ash)	46.66	183.27	125.60	46.65	395.19

3.2.2. Reaction Yield

In transesterification reactions, the methanol quantity and the catalyst selection are critical parameters demanding precise control. These components play pivotal roles in the efficient conversion of TGs into biodiesel. Catalysts expedite the transesterification process and facilitate the formation of MEs. Various catalysts can be employed, such as KOH, potassium carbonate (K_2CO_3), and date stone ashes. The choice of catalyst is contingent upon several factors, including reaction conditions, availability, and cost.

The yield of the transesterification reaction varies depending on the plant species, the metabolite content within each species, and the solvent's nature and polarity used in extraction or fractionation processes (Figure 1).

Figure 1: Yields of (a) *Ricinus communis* methyl ester oil (RMEO), and (b) *Jatropha curcas* methyl ester oil (JMEO), obtained after transesterification in the presence of methanol and a catalyst (either K_2CO_3 or KOH).



The reactions were carried out using 100 g of oil with methanol in a 6:1 molar ratio of methanol to oil, and 2 wt% K_2CO_3 or 1 wt% KOH as catalysts.

3.3. Properties of the Biodiesel Obtained from *R. communis'* and *J. curcas'* oils:

Characterizations included density, viscosity, CV, and flow rate measurements. Table 3 summarizes the characteristics of the biodiesel obtained from the transesterification of *J. curcas' and R. communis'* oils, including test methods, results, and comparison with ASTM D6751 and EN 14214 standards (Sidohounde *et al.*, 2018, Ahmad *et al.*, 2014, Hamdy *et al.*, 2022).

Table 3: Physicochemical features of the biofuel.

Characteristics	Test Method	Result	ASTM D6751	EN 14214
ME (% wt)	EN 14103	98.8	-	≥ 96.5
Oxidation Stability at 110°C (h)	EN 141112	30	-	≥ 6
Density at 15°C (kg/m3)	ASTM D 1298	871	860-900	820-860
Density at 30°C (kg/m3)	ASTM D 1298	862	-	-
Flash Point (°C)	ASTM D 93	> 120	> 130	> 101
Water content (ppm)	EN 12937	279	-	≤ 0.05
AV (mg KOH/g)	ASTM D 664	0.29	< 0.5	< 0.50
IV (g I2/100g)	EN 141111	56	-	< 120
Linolenic Acid ME (% wt)	EN 14103	0.08	-	12
Methanol content (% wt)	EN 14110	0.01	-	≤ 0.20
Monoglyceride (% wt)	EN 14105	0.20	-	≤ 0.80
Diglyceride (% wt)	EN 14105	0.04	-	≤ 0.20
Triglyceride (% wt)	EN 14105	0.00	-	≤ 0.20
Free Glycerin (% wt)	EN 14105	0.02	-	≤ 0.02
Total Glycerin (% wt)	EN 14105	0.07	-	≤ 0.25
Cloud Point (°C)	ASTM D 2500	20	-3 to 12	101

AV: acid value, IV: iodine value, ME: methyl ester.
The results provided apply to biodiesel obtained from *Ricinus communis* methyl ester oil (RMEO) and *Jatropha curcas* methyl ester oil (JMEO).

3.4. Fourier transform infrared (FTIR) Analysis of *R. communis'* Methyl ester Oil (RMEO) and *J. curcas'* Methyl ester Oil (JMEO):

Figure 2 and Table 4 show the Fourier transform infrared (FTIR) analysis spectra of biodiesels (RMEO and JMEO).

The peaks at 2955 and 2855 cm^{-1} are assigned to the symmetric and asymmetric stretching of CH_2 bonds, respectively. The peak at 2925 cm^{-1} shows the symmetric stretching of the CH_3 group. The absorption peaks between 1300 and 1500 cm^{-1} represent the angular deformation of CH_2 and CH_3 groups. The peak at 720 cm^{-1} is attributed to the asymmetric planar deformation of the CH_2 group. Biodiesel is a mixture of MEs with both long and short chains. Thus, the primary signature distinguishing biodiesel production from diesel is its 1743 and 1169 cm^{-1} absorption, related to the stretching modes of the ester functional groups -C=O and -COC, respectively. Another distinctive feature of biodiesel production is related to saturated and unsaturated ME compounds, whereas diesel fuel consists primarily of saturated hydrocarbons. Consequently, biodiesel displays absorption at 3018 and 1652 cm^{-1}, reflecting the alkene functional groups =C—H and —C=C, respectively.

Figure 2: Fourier transform infrared (FTIR) analysis spectrum of *R. communis' methyl ester oil (RMEO) and J. curcas'* methyl ester oil (JMEO).

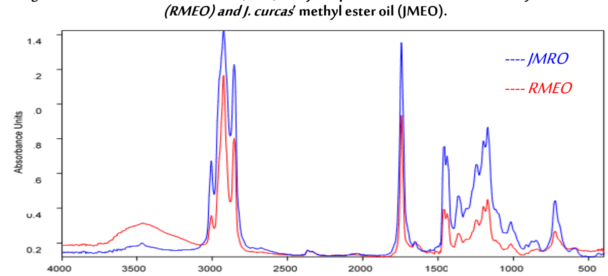


Table 4: Identified Chemical Bonds by Fourier transform infrared (FTIR).

Wavenumber (cm^{-1})	Chemical Bond
2955	Asymmetric stretching mode of CH_2
1500 and 1300	Angular deformation of CH_2 and CH_3
720	Asymmetric planar angular deformation of CH_2
3018	=C—H stretching mode in alkene
2925	Symmetric stretching of CH_3 in alkane
2855	Symmetric stretching of CH_2 in alkane
1743	Stretching of —C=O in ester
1652	Extended —C=C of alkene
1169	Stretching of —COC in ester

4. Discussion

Table 1 provides an overview of the composition of *J. curcas* crude oil, revealing that it contains 11.1% saturated fatty acids (SFAs) and 77.4%

UFAs. On the other hand, *R. Communis*'s oil seeds contain approximately 50 to 70% oil, specifically TGs, which consist of 2.3% SFAs and 87.7% UFAs (Fatma *et al.*, 2023). *R. communis*'s oil has a high viscosity, a moderately high density, and moderate saponifiability. It may be kept at 0°C and 10°C (refrigeration). According to the results presented in Table 2, the ashes of Deglet-Nour date palm kernels are rich in mineral matter. The predominant element is calcium, which is present as calcium oxide (CaO). Since CaO is essential, it reacts with methanol to make the methoxide anion (CH_3O^-). This anion acts as a nucleophile, speeding up the transesterification reaction (Kouzu and Hidaka, 2012, Avhad and Marchetti, 2015). The yield (conversion rate) of JMEO and RMEO obtained through transesterification was 85.33% and 74.9%, respectively, when KOH was used as the catalyst (1%) but was 76.89% and 83.63% when K_2CO_3 was used as the catalyst (0.5%) (Figure 1). These data mean that KOH is a better catalyst than K_2CO_3 for producing biodiesel from *J. curcas*'s oil, whereas K_2CO_3 is a better catalyst than KOH for producing biodiesel from *R. communis*'s oil. Density measurements reveal that both biodiesels exhibit lower densities than crude oil, indicating successful transesterification and a potential reduction in viscosity (Table 3). This reduction in density suggests improved flow characteristics, which is desirable for efficient fuel usage. The cetane index (CI) denotes the ignition quality of a fuel, typically increasing with the number of carbons and decreasing with the number of unsaturated carbon bonds (Ramalingam *et al.*, 2023, Achille *et al.*, 2023). Variations in the CI between JMEO and RMEO reflect differences in the number of carbons and unsaturated carbon bonds in each biodiesel. Other properties, such as oxidation stability, flash point, water content, AV, and glyceride content, fall within the acceptable range according to ASTM D6751 and EN 14214 standards. These results indicate the suitability of both biodiesels as alternative fuels and the potential of *J. curcas* and *R. communis* oils as renewable sources for biodiesel production, contributing to sustainable energy solutions.

The reaction mechanism of transesterification using KOH and K_2CO_3 as catalysts can be described in several steps:

- **Catalyst Activation:** The reaction begins with the activation of the catalyst in the reaction medium, typically methanol. For KOH, it dissolves in methanol to form CH_3O^-. In the case of K_2CO_3, it reacts with methanol to form CH_3O^- as well, but the reaction also produces carbonic acid (H_2CO_3), which decomposes into carbon dioxide (CO_2) and water. CH_3O^- is the crucial active species in the reaction that attacks the TG.
- **Acyl Exchange Reaction:** In this step, CH_3O^- acts as a nucleophile, attacking the glyceride molecule (TG). The nucleophilic attack occurs at the carbonyl carbon of the ester group in the TG, displacing the fatty acid chain (RCOOH). This leads to the formation of a ME (biodiesel) and the release of glycerol as a byproduct.
- **Reverse Reaction:** The glycerol produced in the previous step can sometimes react with another methanol molecule. This can lead to a TG molecule regeneration and water formation. However, this reverse reaction is generally negligible because glycerol is only sparingly soluble in methanol, and the equilibrium favors the formation of biodiesel and glycerol.
- **Approved Catalyst Range:** Catalyst concentrations within the optimal range promote efficient catalysis. For KOH, concentrations between 1% and 2% provide the best biodiesel yield. Similarly, K_2CO_3 concentrations between 0.5% and 2% are adeqaute. These concentrations ensure optimal catalyst activation, thus increasing the rate of the acyl exchange reaction. Furthermore, the available TGs are maximally utilized, yielding higher biodiesel.
- **Catalyst Excess:** Exceeding the recommended catalyst concentrations can produce undesirable consequences. An excess of KOH or K_2CO_3 can react with free fatty acids (FFAs) in the oil, forming soap through saponification. This is an unwanted side reaction, as the soap formed can create emulsions and hinder biodiesel separation. Moreover, excess catalyst can leave residues in the final product, necessitating additional purification steps to meet quality standards.

Using Deglet-Nour DPKA as a natural catalyst in the transesterification reaction resulted in a miscible (homogeneous) mixture, with the ash powder suspended in the reaction medium. This indicates that the ash was not dissolved in the medium. Dissolving the ash allows its active compounds to fulfill their role as catalysts and promote the transesterification reaction (Saetiao *et al.*, 2023, Chutia and Phukan, 2023, Tobío-Pérez *et al.*, 2021).

5. Conclusions

Vegetable oils are prominent renewable and sustainable energy sources, though their direct use in diesel engines necessitates specific modifications due to unique physicochemical properties deviating from standard parameters. In contrast, biodiesels derived from these oils closely emulate petrodiesel regarding physicochemical characteristics, offering advantages such as lower toxicity, biodegradability, high calorific values, and reduced greenhouse gas emissions.

The physicochemical properties of *J. curcas* and *R. communis* oils prove suitable for energy use, except for viscosity, which remains high. Nonetheless, this limitation can be mitigated through transesterification. Basic or acid transesterification is the most common process for converting vegetable oils into biodiesel due to its simplicity and cost-effectiveness. However, meticulous control of methanol quantity and catalyst type is pivotal for efficiently converting TGs into biodiesel.

In transesterification, methanol reacts with TGs in vegetable oils or animal fats, transforming them into methyl esters (biodiesel). The stoichiometric ratio of methanol to TGs, typically 3:1, ensures complete conversion and minimizes unreacted methanol in the final biodiesel product. While date seed ashes may exhibit catalytic activity during combustion, their efficiency as transesterification catalysts may be lower than that of conventional catalysts such as strong bases or enzymes.

The resulting biodiesels closely resemble petrodiesel in fuel characteristics, making them suitable substitutes for fossil diesel, meeting ASTM D6751 and EN 14214 standards. Limitations include the lower efficiency of date seed ash as a catalyst than conventional catalysts, the persistence of high viscosity in oils despite transesterification, and the high consumption of methanol, which can increase costs and environmental impact. Future research should focus on enhancing catalyst efficiency, optimizing reaction conditions, and exploring alternative feedstocks like waste oils. Investigating alternative alcohols for biodiesel production and conducting life cycle assessments would further improve sustainability and cost-effectiveness.

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