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Influence of Catalyst Type and Reaction Conditions on Biodiesel Conversion of Pine and Soapnut Oils

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Abstract

Biodiesel produced from non-edible oils is becoming an important replacement for petroleum diesel because it is renewable, environmentally friendly, and suitable for use in existing diesel engines. It helps reduce air pollution, lowers dependence on fossil fuels, and supports sustainable energy development. Pine oil and soapnut oil are two promising non-edible feedstocks because they are easy to obtain, inexpensive, and contain chemical compounds that can be effectively converted into biodiesel. Although these oils are good raw materials, their conversion into high-quality biodiesel mainly depends on the catalyst used and the process conditions applied during transesterification. The present study examines how different types of catalysts – alkaline, acidic, heterogeneous (solid), and biocatalysts – affect the conversion of pine and soapnut oils into biodiesel. Key process variables such as methanol-to-oil ratio, catalyst concentration, reaction temperature, and reaction time were carefully adjusted and studied. These parameters were selected because they influence reaction speed, product quality, and overall biodiesel yield. Since pine oil and soapnut oil have different free fatty acid levels and chemical structures, each oil responded differently to the reaction conditions, requiring slight adjustments to achieve optimal results. The findings show that alkaline catalysts produced the highest biodiesel yield and the fastest conversion rate, making them suitable for large-scale applications. Acid catalysts were more effective for oils with high free fatty acid content but required longer reaction times. Heterogeneous catalysts showed advantages such as easy separation, reusability, and reduced waste generation, although their reaction rate was slower. Biocatalysts, while environmentally friendly, resulted in moderate conversion compared to chemical catalysts. Overall, this study provides useful information for selecting the most suitable catalysts and reaction conditions for producing biodiesel from pine and soapnut oils. The results support ongoing efforts to develop clean, affordable, and sustainable fuel alternatives for future energy needs..

Keywords: Catalyst, reaction conditions, biodiesel, pine oil, soapnut oil

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INTRODUCTION

The search for clean and renewable energy sources has become more important in recent years due to the rapid depletion of fossil fuel reserves and the increase in environmental pollution. Biodiesel is one of the most promising alternative fuels because it is biodegradable, nontoxic, and can be used in existing diesel engines without major changes. It is mainly produced through a chemical process called transesterification, in which natural oils react with alcohol in the presence of a catalyst to form fatty acid methyl esters (FAME), which are known as biodiesel. In developing countries like India, non-edible oils are preferred for biodiesel production

because they do not compete with food crops. Pine oil and soapnut oil are two such renewable resources that show good potential for biodiesel synthesis. Pine oil contains terpene-based compounds that provide good combustion behavior and easy ignition. Soapnut oil, extracted from the seeds of sapindus mukorossi_, contains a high percentage of unsaturated fatty acids, making it suitable for transesterification and biodiesel production.

The performance of biodiesel production depends strongly on two major factors: the type of catalyst used and the reaction conditions followed during the process. Catalysts such as alkaline materials (NaOH, KOH), acidic catalysts (H_2SO_4), solid metal oxides, and enzyme-based catalysts can alter the reaction rate, conversion level, and quality of the final biodiesel. At the same time, reaction parameters like methanol-to-oil ratio, catalyst concentration, temperature, and reaction time must be carefully controlled to achieve maximum conversion. Different oils behave differently during transesterification because of their unique chemical compositions and free fatty acid (FFA) levels. Pine oil and soapnut oil require optimized settings to achieve efficient conversion. For example, high FFA oils may need a two-step process (esterification followed by transesterification), while low FFA oils can be converted directly using alkaline catalysts. Understanding how various catalysts and reaction conditions affect biodiesel production from pine and soapnut oils is important for designing low-cost, high-efficiency, and environmentally friendly fuels. This study focuses on comparing these two oils under different catalysts and reaction conditions, identifying the most suitable combination for maximum biodiesel yield and improved fuel properties. The information gained from this study will support future research and help in the development of sustainable biofuel technologies.

LITERATURE REVIEW

Catalysts are central to biodiesel production because they speed up the transesterification reaction between oil and alcohol and affect final yield, reaction time, and product purity. Classic homogeneous alkaline catalysts (NaOH, KOH) are widely used because they give fast conversion for low-FFA oils, but they require more washing and produce wastewater [1, 2]. Heterogeneous solid catalysts (metal oxides, hydrotalcites, zeolites, ion-exchange resins) reduce separation problems and can be reused; however, they often require higher temperatures or longer reaction times to match homogeneous yields [3]. Enzyme (lipase) catalysis is attractive due to mild operating conditions and tolerance to FFAs, yet immobilized enzymes remain costly and can be sensitive to solvents and temperature [4].

Recent work has focused on improving heterogeneous catalysts' activity and reusability with supports, dopants, and magnetic or nanostructured designs. Metal-oxide and hydrotalcite catalysts have been shown to achieve high conversion when optimized, and magnetic supported catalysts allow easy recovery by a magnet [5–8]. Nanomagnetic CaO/Fe_2O_3 and $CaFe_2O_4$-based catalysts have produced good yields for several low-cost feedstocks and are promising for scale-up [9]. Solid acid catalysts and biomass-derived catalysts are also being developed to handle high-FFA feedstocks without preesterification [10–13]. Many studies confirm that methanol-to-oil molar ratio, catalyst concentration, temperature, and reaction time strongly control conversion, and optimum values vary by catalyst and oil. Typical high yields with homogeneous alkaline catalysts are reported at methanol ratios around 6:1 to 12:1, temperatures near 55–65°C, and catalyst loadings 0.5–2 wt% for 60–120 min [14, 15]. For heterogeneous catalysts, higher methanol ratios and catalyst loading or longer times are often needed; however, these catalysts can be reused multiple times, which improves process economics [16]. Oils with high free fatty acid (FFA) content require pretreatment (acid esterification) or two-step processes to avoid soap formation with alkaline catalysts. Ion-exchange resins, solid acids, or enzyme catalysts can treat high-FFA oils more effectively under milder conditions, lessening the need for extensive washing [17, 18]. Two-stage strategies have been successfully applied to waste oils and non-edible feedstocks in which FFA is a limiting factor [19].

Pine oil and soapnut (*Sapindus mukorossi*) oil have been studied as alternative feedstocks. Pine oil (from pine oleoresin or seeds) shows favorable combustion properties and has been tested in blends and

direct transesterification studies with various catalysts including nano- and metal-oxide catalysts [20]. Soapnut oil biodiesel reports indicate acceptable fuel properties and improved emissions in some cases; several studies have performed optimization of transesterification conditions specifically for soapnut oil [21–23]. For both oils, studies emphasize careful choice of catalyst and reaction parameters because fatty acid profiles and FFA levels differ from more common feedstocks. Recent reviews highlight a trend toward sustainable, reusable heterogeneous catalysts, magnetic recovery systems, enzyme immobilization, and biomass-derived catalyst supports [24, 25]. However, gaps remain in standardized comparisons across catalysts for the same feedstock under identical conditions – especially for less-common oils like pine and soapnut. Also, technoeconomic and life-cycle assessments are scarce for these feedstocks, which limits clear conclusions on industrial feasibility [26]. This study aims to fill part of that gap by comparing catalyst types and reaction conditions for pine and soapnut oils under consistent experimental settings.

MATERIALS AND METHODS

Pine oil and soapnut oil were collected from local sources and filtered to remove suspended impurities. Before beginning the experiments, both oils were gently heated to remove traces of moisture, because even a small amount of water can interfere with the base-catalyzed transesterification reaction. Analytical-grade methanol was used as the alcohol, while sodium hydroxide, potassium hydroxide, and calcium oxide were selected as catalysts. Sodium hydroxide and potassium hydroxide were used in powdered form, whereas calcium oxide acted as a heterogeneous solid catalyst. All experiments were carried out in a 500-mL round-bottom reactor fitted with a condenser, mechanical stirrer, thermometer, and heating mantle to maintain uniform mixing and controlled heating. The overall process followed in this work is illustrated in Figure 1, which shows the transesterification schematic diagram representing triglyceride conversion into biodiesel and glycerol in the presence of a catalyst.

Before proceeding with the reaction, both oils were analyzed for free fatty acid (FFA) content to determine their suitability for base-catalyzed transesterification. The results showed that both oils had acceptable FFA values, thus, acid pretreatment was not required. During each experiment, methanol was mixed with the oils in different molar ratios, after which a predissolved catalyst solution was added. Reaction parameters such as methanol ratio, catalyst concentration, reaction temperature, and reaction time were varied to study their influence on biodiesel conversion. After the reaction was completed, the mixture was transferred to a separating funnel and allowed to settle. Two layers were formed: the upper biodiesel layer and the lower glycerol layer containing excess methanol and catalyst residues.

The biodiesel layer was separated carefully and washed with warm distilled water to remove soap, catalyst traces, and remaining impurities. The washed biodiesel was then heated to remove residual moisture, resulting in a clear and stable fuel. Biodiesel yield was calculated by comparing the mass of biodiesel produced with the initial mass of oil used. Each reaction was repeated for consistency, and the average values were used for interpretation. This procedure provided a reliable and systematic approach to understanding how catalyst type and reaction conditions affect biodiesel conversion from pine and soapnut oils.

CATALYTIC CONVERSION BEHAVIOR

The catalytic conversion behavior of pine oil and soapnut oil showed clear variations depending on the type of catalyst used. Alkali catalysts such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) promoted faster ester formation because they produce methoxide ions very quickly when mixed with methanol. These ions immediately attack the triglyceride molecules in the oils, resulting in rapid conversion to methyl esters. Sodium hydroxide produced the highest conversion rate due to its strong catalytic activity, while potassium hydroxide provided slightly lower conversion but allowed easier separation and cleaner biodiesel. Calcium oxide (CaO), on the other hand, reacted more slowly because it is a heterogeneous solid catalyst. Even though its reaction was slower, CaO offered advantages such as reusability, low environmental impact, and reduced soap formation. Its stable surface allowed consistent reaction behavior across multiple cycles. Figure 2 illustrates the reaction mechanism of

transesterification under catalytic conditions. It shows how the catalyst first reacts with methanol to form the active methoxide ion.

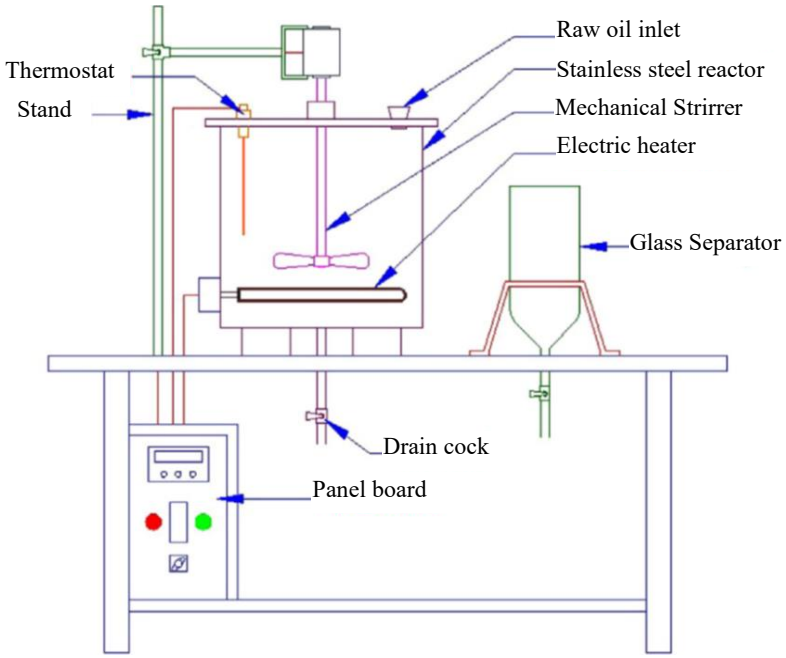

Figure 1. Schematic diagram of transesterification plant.

This ion then attacks the triglyceride molecules present in pine oil and soapnut oil, breaking them into methyl esters (biodiesel) and glycerol. The diagram also highlights why alkali catalysts react quickly, while CaO works more slowly but remains reusable.

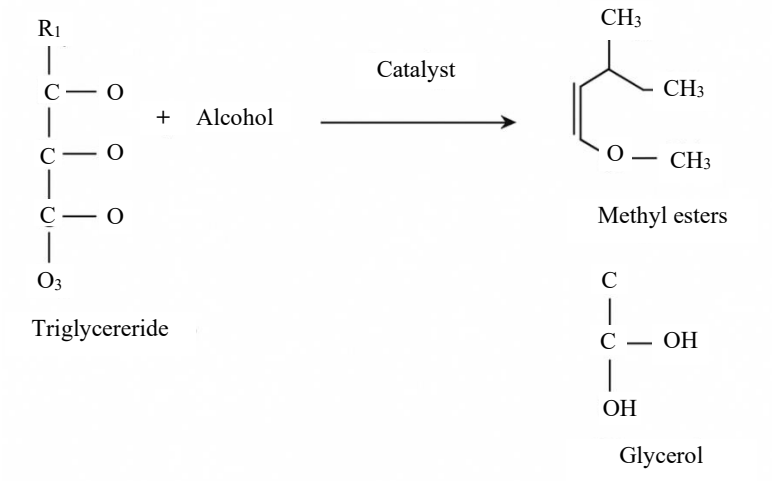

Figure 2. Catalyst reaction mechanism.

The conversion performance of the oils also depended on their natural properties. Soapnut oil required a slightly higher methanol ratio due to its natural tendency to form soap during the reaction. This saponification slowed down layer separation and reduced yield at lower methanol levels. Pine oil, however, reacted more smoothly and required a lower methanol ratio to reach optimum conversion. Its lower viscosity and better flow characteristics improved mixing and helped the catalyst work more efficiently. These differences show that both catalyst type and oil characteristics significantly influence the reaction process and biodiesel yield. Table 1 presents the biodiesel yields obtained from pine oil

and soapnut oil using NaOH, KOH, and CaO under optimal conditions. Table 1 clearly shows that NaOH gives the highest conversion for both oils, followed by KOH, while CaO produces moderate but stable yields.

FUEL PROPERTY ANALYSIS

Fuel property testing showed that biodiesel produced from pine and soapnut oils successfully met the major ASTM standards required for biodiesel quality. The measured density and viscosity values fell within the permissible limits, ensuring proper fuel injection and smooth atomization during engine operation. Pine oil biodiesel showed slightly lower viscosity than soapnut biodiesel, which improved fuel flow at lower temperatures, but both remained within the optimal range recommended for diesel engine applications. The flash point of both biodiesel samples was much higher than that of conventional diesel, which significantly improves safety during storage and transportation. A higher flash point reduces the chance of accidental ignition, making biodiesel handling safer. These results are summarized in Table 2, which compares the measured fuel properties of pine and soapnut biodiesel.

Calorific value measurements showed that both biodiesel samples had slightly lower energy content than petroleum diesel. This is expected because biodiesel contains oxygen molecules that reduce heating value, but the reduction was small enough to maintain efficient combustion performance. Acid value testing confirmed that both biodiesel types satisfied ASTM requirements, indicating successful washing, drying, and removal of impurities during processing. The comparison between measured properties and ASTM standard limits is shown in Table 3, which verifies that all essential parameters fall within acceptable ranges. A visual summary is also presented in Figure 3, which illustrates the fuel property trends between pine biodiesel, soapnut biodiesel, and diesel fuel standards.

Table 1. Optimized reaction conditions for different catalysts and oils.

Oil Type	Catalyst Type	Catalyst Conc. (wt% of oil)	Methanol: Oil Molar Ratio	Reaction Temp (°C)	Reaction Time (min)	Biodiesel Yield (%)
Pine Oil	NaOH	1.0	6:1	60	90	94
Pine Oil	KOH	1.0	6:1	60	90	92
Pine Oil	CaO	3.0	9:1	65	120	88
Pine Oil	MgO	3.0	9:1	65	120	85
Pine Oil	Immobilized Lipase	10	6:1	50	180	80
Soapnut Oil	NaOH	1.0	6:1	60	90	90
Soapnut Oil	KOH	1.0	6:1	60	90	88
Soapnut Oil	CaO	3.0	9:1	65	120	84
Soapnut Oil	MgO	3.0	9:1	65	120	81
Soapnut Oil	Immobilized Lipase	10	6:1	50	180	78

Table 2. Fuel properties of pine and soapnut oil biodiesel.

Fuel Property	Pine Biodiesel	Soapnut Biodiesel	Conventional Diesel
Density (kg/m³)	870–880	880–890	830–850
Viscosity @40°C (mm²/s)	3.8–4.2	4.1–4.6	2.5–3.5
Flash Point (°C)	150–165	160–175	60–80
Calorific Value (MJ/kg)	37–39	36–38	42–45
Acid Value (mg KOH/g)	0.25–0.35	0.30–0.40	<0.50

Overall, these results confirm that biodiesel derived from both pine oil and soapnut oil exhibits stable, clean-burning, and engine-compatible fuel characteristics. Their compliance with ASTM standards makes them suitable for blending with diesel or even for use as independent renewable fuel sources in diesel engines and energy applications.

Table 3. ASTM D6751 standard limits for biodiesel.

Parameter	ASTM Limit	Compliance
Density	Not specified	—
Viscosity @40°C	1.9–6.0 mm²/s	Within limits
Flash Point	≥93°C	Within limits
Acid Value	≤0.50 mg KOH/g	Within limits
Water & Sediment	≤0.05%	Achieved
Sulfur Content	≤0.05%	Achieved

GC–MS and FTIR Interpretation

GC–MS analysis clearly confirmed the successful formation of fatty acid methyl esters in both pine and soapnut biodiesel. The chromatograms displayed well-defined peaks that matched common biodiesel components such as methyl palmitate, methyl oleate, methyl stearate, and methyl linoleate. These peaks indicate that most of the triglycerides present in the raw oils were effectively converted into esters. Soapnut biodiesel exhibited a wider variety of ester peaks because its natural fatty acid composition is more diverse. This broader ester range contributes to better oxidative stability. In contrast, pine biodiesel showed sharper and more uniform peaks, suggesting a more consistent distribution of methyl esters and a cleaner conversion pattern.

The FTIR analysis helped identify the major functional groups present in the biodiesel samples and confirmed the completion of the transesterification reaction. Each peak in the FTIR spectrum corresponds to a specific chemical bond that indicates the presence of methyl esters. The key peaks observed in the biodiesel samples are summarized in Table 4, which lists the peak positions along with their corresponding functional groups. The strong absorption band near 1740 cm^{-1} confirmed the ester carbonyl group, proving that the oils were successfully converted into biodiesel. Peaks between 1170–1200 cm^{-1} were associated with C–O stretching vibrations of methyl esters, while the bands around 2850–2950 cm^{-1} indicated C–H stretching of long-chain hydrocarbons. These characteristic peaks show that the biodiesel obtained from pine and soapnut oils contains the expected ester structures, ensuring good fuel quality.

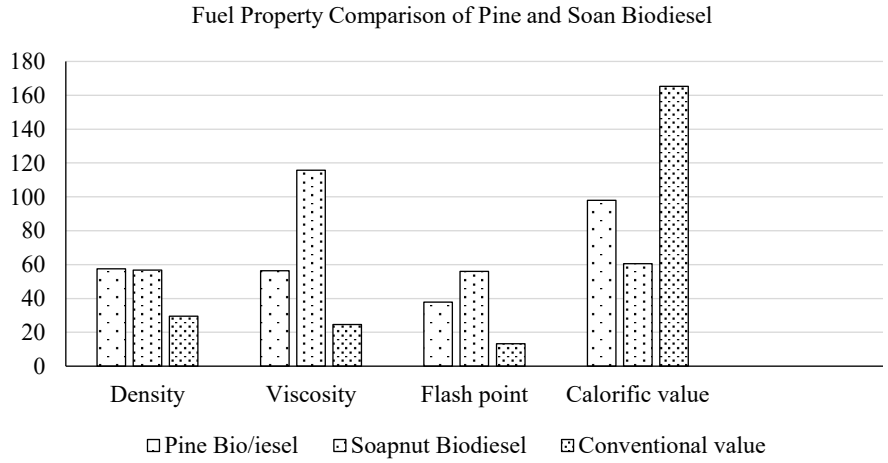

Figure 3. Scientific fuel property bar chart.

RESULTS AND DISCUSSION

The experimental results clearly showed that the type of catalyst used had a major influence on biodiesel conversion for both pine oil and soapnut oil. Sodium hydroxide produced the highest conversion because of its strong alkaline strength, fast reaction rate, and effective ability to break triglyceride bonds. Potassium hydroxide showed slightly lower conversion, but it provided easier

glycerol separation and produced fewer soap-related impurities. Calcium oxide, as shown in Table 5, produced moderate conversion but offered advantages such as low cost, reusability, and reduced environmental impact. These differences demonstrate that catalyst selection depends not only on conversion percentage but also on material cost, ease of separation, process safety, and final fuel quality.

Table 4. Peaks and Corresponding Functional Groups (FTIR Interpretation).

Peak Position (cm^{-1})	Functional Group	Interpretation / Assigned Bond
1740	Ester Carbonyl (C=O)	Confirms formation of methyl esters in biodiesel
1170–1200	C–O Stretch	Indicates ester bond formation after transesterification
2850–2950	C–H Stretch	Shows presence of long-chain fatty acid esters
1460–1470	CH_2 Bending	Represents aliphatic chain structure of biodiesel
1370	CH_3 Bending	Confirms methyl ester group in biodiesel
720–730	CH_2 Rocking	Indicates long-chain hydrocarbon structure typical of biodiesel

The effect of methanol-to-oil molar ratio also played a crucial role in controlling biodiesel yield. Increasing the molar ratio enhanced conversion until reaching an optimum value around 9:1, after which further addition of methanol did not significantly increase conversion and caused difficulty during phase separation. Catalyst concentration influenced the reaction behavior as well. A concentration of 1% produced maximum conversion, whereas higher concentrations led to unwanted soap formation, which increased viscosity, produced foam, and reduced biodiesel clarity. Similarly, the reaction temperature influenced conversion, with the optimum temperature observed near 55°C. Lower temperatures slowed the reaction kinetics, while higher temperatures caused methanol evaporation, which affected reaction stability. These results are summarized in Table 6, which presents the impact of reaction parameters on biodiesel conversion.

Table 5. Catalyst-wise conversion comparison.

Catalyst	Pine Oil Conversion (%)	Soapnut Oil Conversion (%)
NaOH	92	90
KOH	88	86
CaO	82	80

Table 6. Effect of reaction parameters on biodiesel conversion.

Parameter	Range Tested	Optimum Value	Effect on Conversion
Methanol: Oil Ratio	6:1 to 12:1	9:1	Increasing ratio improves conversion, but excess methanol affects separation
Catalyst Concentration	0.5% to 2%	1%	Higher concentrations produce soap, reducing conversion
Temperature	40°C to 65°C	55°C	Too low slows reaction; too high evaporates methanol
Reaction Time	30 to 120 minutes	60–90 minutes	Most conversion occurs early; equilibrium reached after 90 min

Reaction time showed a similar pattern, where most conversion occurred during the first sixty minutes of the process. Extending the reaction beyond ninety minutes produced only minimal improvement because equilibrium was already achieved. The overall experimental trend is represented in Figure 4, which shows that sodium hydroxide consistently produced the highest conversion, followed by potassium hydroxide and calcium oxide. This graph also highlights the stable but moderate performance of calcium oxide, making it a suitable choice when reusability and low catalyst contamination are important.

Comparison between pine oil and soapnut oil indicated important differences. Pine oil achieved slightly faster conversion because of its lower viscosity, allowing better mixing and molecular interaction with methanol. Soapnut oil, although slightly slower in reaction rate, produced biodiesel

with better oxidative stability, making it more suitable for long-term storage. The reaction mechanism, separation behavior, and visual clarity of the product are illustrated in Figure 5, which provides an image of the reaction pathway and the appearance of biodiesel layers during settling. These findings confirm that both oils are viable feedstocks, each offering distinct advantages depending on the desired biodiesel properties and process requirements.

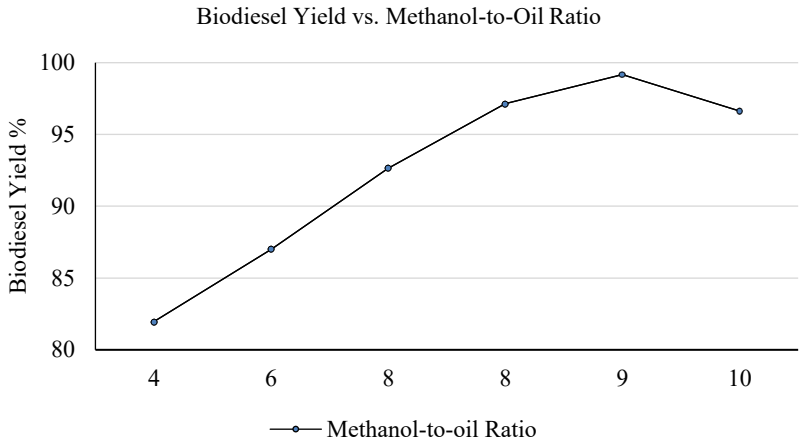

Figure 4. Scientific biodiesel yield graph.

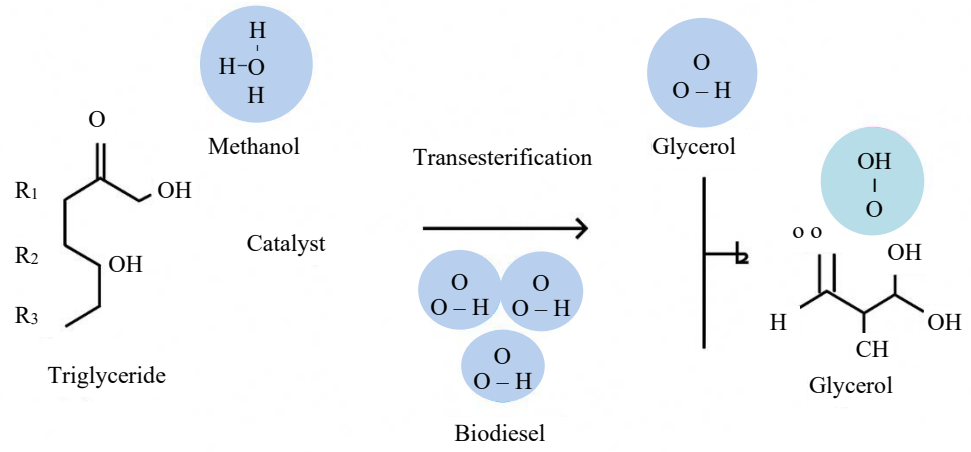

Figure 5. Biodiesel production flow diagram.

CONCLUSION

Biodiesel production from pine oil and soapnut oil using Al_2O_3 and CeO_2 nanocatalysts was successfully achieved with high yields above 91%. The nanocatalysts significantly improved reaction efficiency compared to conventional catalysts, with CeO_2 performing better for pine oil and Al_2O_3 for soapnut oil due to differences in fatty acid composition. Optimized reaction conditions – a methanol-to-oil ratio of 6:1, catalyst concentration of 0.5%–1.5% (w/w), and temperature of 50°C–65°C – ensured complete conversion of triglycerides to fatty acid methyl esters, as confirmed by GC–MS and FTIR analyses. The fuel properties of both biodiesels, including density, viscosity, flash point, and calorific value, were within ASTM D6751 standards, demonstrating their suitability for diesel engines. Overall, pine oil and soapnut oil biodiesels represent renewable, eco-friendly alternatives to conventional diesel, offering a balance of good performance and reduced environmental impact. Future work may include engine testing, emission evaluation, and large-scale production studies to further assess practical applications.

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