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Editorial

Optimizing biodiesel production from Madhuca Longifolia oil: Catalyst comparison and process parameters optimization

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Abstract: The current research delves into the transesterification of Madhuca Longifolia seed oil into biodiesel, employing both homogeneous catalysts (KOH and NaOH) and a heterogeneous catalyst derived from waste eggshell. For the homogeneous system, various process variables such as reaction temperature, catalyst quantity, and oil to molar ratio were meticulously optimized to assess their impact on biodiesel yield. The optimal conditions for the NaOH-catalyzed transesterification reaction were determined as follows: 0.4 gm NaOH, 1:6 oil to methanol molar ratio, 60°C reaction temperature, and a reaction time of 60 minutes. Results indicated that NaOH outperformed KOH, yielding a remarkable % conversion. In the case of the heterogeneous catalyst derived from waste eggshell, a batch reactor was employed. Here, 100 ml of Madhuca Longifolia oil was mixed with 26.67 gm of methanol, maintaining a methanol to Madhuca Longifolia oil molar ratio of 9:1. The eggshell-based catalyst was utilized at a proportion of 3 wt% relative to the oil weight, with a reaction temperature of 60°C and a reaction time of 2 hours. This process yielded a biodiesel with an 87% conversion. The produced biodiesel was evaluated in accordance with the ASTM D6751 standard and was found to meet the acceptable quality limits. In summary, this study underscores NaOH as a superior catalyst for generating biodiesel with desirable properties from non-edible Madhuca Longifolia seed oil. Additionally, waste eggshell emerges as a viable option for producing biodiesel as a heterogeneous catalyst.

1. Introduction

The impetus behind undertaking this study on biodiesel production in the contemporary context is multifaceted. Firstly, there exists a pressing imperative for biodiesel as an eco-friendly alternative to fossil fuels, crucial for combating climate change and curbing greenhouse gas emissions. The pronounced dependency of India on imported crude oil underscores the urgency of diversifying the energy portfolio

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and harnessing domestic resources such as vegetable oils, animal fats, and waste cooking oil for fuel production. Furthermore, biodiesel holds the potential to significantly bolster energy security and sustainability, concurrently addressing air quality concerns and traffic congestion within the transportation sector. Moreover, the Government of India has acknowledged the pivotal role of biodiesel and introduced policies like the National Policy on Biofuels and the National Biodiesel Mission [1]. Therefore, it is imperative to scrutinize biodiesel production within the framework of these policies to gauge their influence, regulatory requisites, and potential incentives for producers.

There exists a diverse range of both homogeneous and heterogeneous catalysts available for biodiesel production. The judicious selection of cost-effective catalysts, both homogeneous and heterogeneous, is pivotal in optimizing the economic viability of biodiesel production. The assessment of economically viable catalysts can lead to a reduction in overall production costs, enhance market competitiveness, and potentially pave the way for the advancement of catalytic systems, further augmenting the potential for large-scale biodiesel production in India.

In summation, this study endeavors to make a significant contribution to sustainable energy development by examining the production methodology of biodiesel through the utilization of low-cost catalysts. It also aims to ascertain the optimum process conditions for this production method, thereby laying a foundation for a more environmentally-friendly and economically sustainable energy future.

1.1. Selection of Feedstock and Catalyst

Biodiesel production is widely recognized for its sustainability, owing to the abundant supply of raw materials. Globally, there are an estimated 350 varieties of oilseed crops available for this purpose. The accessibility of these feedstocks is contingent on factors such as geographical location, weather conditions, land type, and agricultural practices specific to each country. Given that feedstocks constitute 75% of the overall manufacturing cost, the judicious selection of suitable ones is imperative to ensure the viability of biodiesel production [2]. Raw materials for biodiesel production can be categorized into first-generation (edible oils), second-generation (non-edible oils), third-generation biodiesel (microalgae), and fourth-generation (Cyanobacteria, modified fatty acids) based on the material utilized in the synthesis [3]. Researcher studied blending an aqueous ammonia emulsion and a zinc sulfide/copper nanocomposite with algae biodiesel-diesel blend, identifying optimal parameters for enhanced performance and reduced emissions. Results highlight efficient nutrient removal through algae cultivation and determine optimal levels of 10% biodiesel, 14% aqueous ammonia, and 95 ppm of ZnS/Cu for the process.

Our study focuses on utilizing *Madhuca longifolia* seeds for biodiesel production. This species is primarily found in arid regions of India, particularly in states like Madhya Pradesh, Maharashtra, and Gujarat [4, 5]. According to a report from the Advanced Biofuel Centre, India possessed a potential of approximately 600,000 tons per year in 2022 using Mahua seeds [6].

Madhuca longifolia offers several advantages as a feedstock:

- It is a non-edible source, reducing competition with food resources.
- It boasts a high oil content.
- The plant is abundantly available in India.
- Remarkably, a single tree has the capacity to yield 200–300 kilograms of seeds per year.
- The production cost is low, contributing to economic viability.
- Additionally, the residue from the biodiesel extraction process can be utilized as a protein source, minimizing waste.

Exploring low-cost homogeneous and heterogeneous catalysts is essential in order to reduce the overall cost of biodiesel production, address environmental concerns, and enhance the efficiency of the biodiesel production processGuidelines can be included for standard research article sections, such as this one.

1.2. Novelty and Objectives of the Current Study

Amidst the rich tapestry of research in biodiesel production, this study carves out a distinct niche by addressing the critical need for economically viable catalysts. By delving into the selection and optimization of low-cost catalysts, both homogeneous and heterogeneous, this research aims to bridge the gap between laboratory-scale innovations and industrial applicability. The novelty of this study lies in its holistic approach towards enhancing the economic feasibility and environmental sustainability of biodiesel production. Through a systematic investigation of catalyst performance under varying process conditions, this study seeks to unlock new avenues for large-scale biodiesel production in India and beyond.

In summary, this study represents a concerted effort to contribute towards sustainable energy development by elucidating the role of low-cost catalysts in biodiesel production. By contextualizing the research within the broader landscape of biodiesel synthesis methodologies and highlighting its novelty, this introduction sets the stage for a comprehensive exploration of catalyst-enabled biodiesel production processes.

2. Methods and Materials

The Madhuca longifolia seeds were harvested from the Naswadi taluka forest, situated in the Chhotaudepur district of Gujarat, India. Procured from a local vendor, the raw seeds were obtained at a cost of Rs 10/kg, which was below the government's prescribed minimum support price of Rs 29/kg. Oil extraction was carried out using an oil expeller, resulting in an extraction yield of 45%. The extracted oil was employed directly as a feedstock, without undergoing any additional purification. Analytical reagent-grade Methanol (99.9% purity, Merck), pellet form Potassium Hydroxide and Sodium Hydroxide, and Sulfuric acid (95–97% purity) were the chemicals used in the study.

In the initial step, the esterification reaction was conducted to reduce Free Fatty Acids (FFA) at 65°C and atmospheric pressure using sulfuric acid as a catalyst. The experimental setup included a 250 ml three-neck flat-bottom flask, a reflux condenser, a temperature controller, and a magnetic stirrer, as depicted in Figure 1. A preheated 100 ml of Madhuca Longifolia oil at 60°C was combined with a homogeneous mixture of 1 ml of sulfuric acid and 30 ml of methanol. A batch time of 60 minutes was selected for complete acid-catalyzed esterification reaction. The resulting mixture was then cooled to room temperature and transferred to a separating funnel for phase separation. Two distinct layers with different colors emerged. The upper layer, containing the esterified sample, was isolated, purified with water, and set aside for subsequent transesterification.

Moving on to the final step, transesterification was carried out employing KOH and NaOH as homogeneous catalysts, utilizing a similar experimental assembly. The 100 ml of esterified sample oil was preheated and introduced into the flask under vigorous stirring. Transesterification occurred at various temperatures (ranging from 50–70°C), with varying amounts of methanol (in molar ratios of 3:1 to 9:1, relative to oil), and catalyst (0.3–0.5 gm). After 60 minutes of reaction time, the resulting product was cooled and transferred to a separating funnel, yielding two distinct layers with different colors. The upper orangish layer contained the biodiesel (methyl ester), while the lower brownish layer contained glycerol. The upper layer was isolated, purified with warm water 2–3 times to remove any dissolved homogeneous catalyst, and then prepared for further analysis after heating.

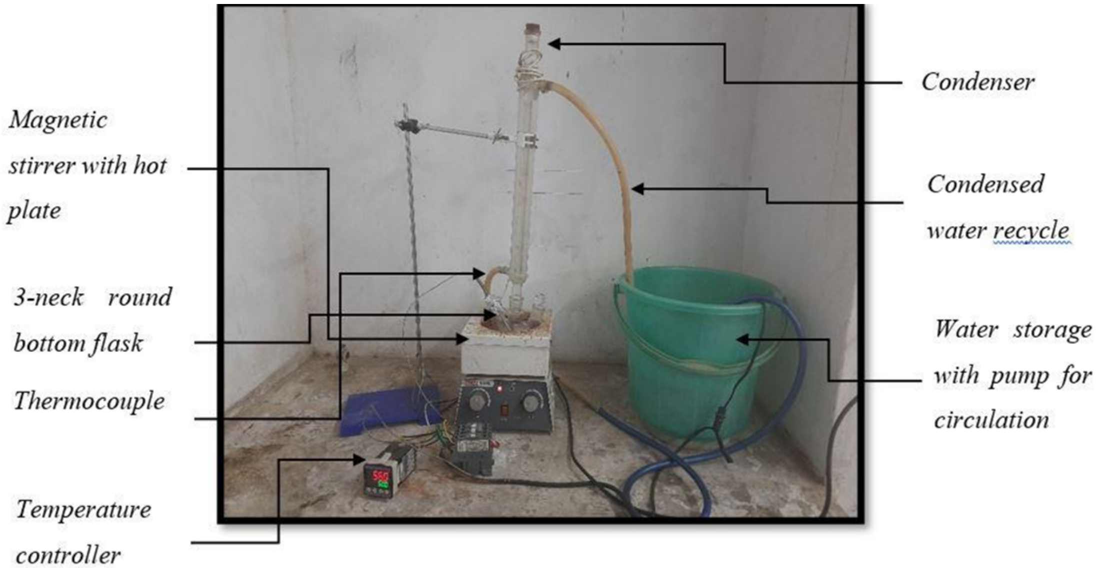

Figure 1. Biodiesel batch reactor.

3. Result & Discussion

3.1. Characterization of properties

This investigation focused on evaluating the notable physical and chemical properties of Madhuca longifolia oil, along with their respective methyl esters, following the ASTM D 6751 standard [7]. These properties encompass factors such as acid value, free fatty acids (FFA), viscosity, density, flash point, fire point, cloud point (CP), pour point (PP), saponification value, molecular weight, ester value, and more.

Table 1. Properties of Madhuca Longifolia (ML) Oil.

Properties of Madhuca Longifolia seed oil	Unit	Value
Kinematic viscosity at 40°C	Cst	40.70
Density	gm/ml	0.91
Acid value	mg of potassium hydroxide/gm of oil	7.51
Free Fatty Acid	%	3.75
Saponification value	mg of potassium hydroxide/gm of oil	422
Molecular weight	gm/mol	866.52

3.2. Catalyst selection

The transesterification process employing a homogeneous catalyst was conducted under the conditions outlined in Table 2. It was noted that, under similar process conditions, NaOH yielded higher results compared to KOH for the given oil sample. Additionally, NaOH is a more cost-effective option than KOH. Given these findings, further optimization studies were conducted with NaOH, focusing on the transesterification reaction parameters using a homogeneous catalyst.

Table 2. Comparison of two homogeneous catalysts.

Catalyst	Cost of Catalyst/ 0.5 kg (Rs)	Catalyst concentration	Methanol: Oil ratio	Reaction time (hr)	Temperature (°C)	Biodiesel Yield (%)	References
KOH	576	0.4 % (w/v)	06:01	1	60	86	Present work
NaOH	333	0.4 % (w/v)	06:01	1	60	96	Present work

3.3. Quality comparison of ML Biodiesel and ASTM standard

The transesterification procedure utilizing a homogeneous catalyst was executed under the conditions specified in Table 3. It was noted that, under these specific conditions, NaOH demonstrated a superior yield compared to KOH for the given oil sample. Additionally, it was observed that NaOH is more cost-effective than KOH. Consequently, the subsequent optimization study focused on NaOH, which delves into the optimization of transesterification reaction parameters employing a homogeneous catalyst.

Table 3. Comparison of ML biodiesel and ASTM standard.

Properties	**Unit**	**ML Biodiesel** *(Homogenous Catalyst-NaOH)*	**ASTM** *Standard (D6751)*
Density	(gm/ml)	0.86	0.860–0.900
Acid value	(mg of KOH/gm of oil)	0.47	$\leq$ 0.50
Kinematic viscosity	at 40°C (Cst)	5.87	1.9–6.0
Flash point	°C	154	130–170
Fire point	°C	180	140–215
Cloud point	°C	11	-3 to 12
Pour point	°C	-3	-15 to 10
Yield %		96	

3.4. Effect of catalyst concentration on ML Biodiesel yield

By maintaining a constant temperature of 60°C and a reaction time of 60 minutes, the impact of catalyst loading was investigated. Catalyst weight was varied within the range of 0.3 to 0.5 grams. The results indicated a notable increase in yield from 55% to 96% as the catalyst loading was adjusted from 0.3 to 0.4 grams. However, a subsequent decrease in yield from 96% to 74% was observed when the catalyst loading was further increased from 0.4 to 0.5 grams, as depicted in Figure 2.

This variation can be attributed to the fact that catalysts furnish active sites where the interaction between triglycerides and alcohol transpires. Augmenting the catalyst loading implies a greater abundance of active sites available for the reaction to occur. This heightened availability of active sites fosters better contact between the reactants, thereby expediting the transformation of triglycerides into biodiesel.

However, at higher catalyst loadings, there is an elevated likelihood of catalyst particles approaching each other or aggregating. This aggregation of catalysts can give rise to larger catalyst clusters or even distinct catalyst phases, resulting in an uneven dispersion within the reaction mixture. Consequently, this may lead to a reduction in overall biodiesel yield, potentially exacerbated by increased soap formation during the water washing step [8,9].

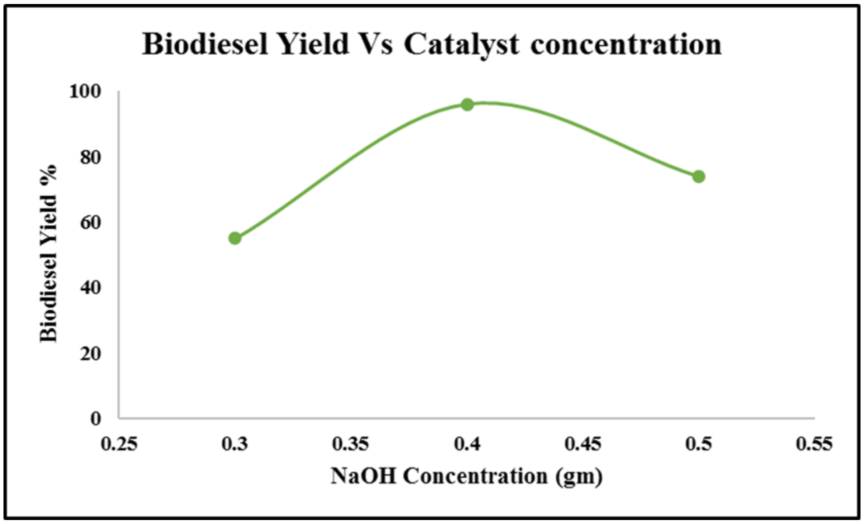

Figure 2. Effect catalyst concentration on biodiesel yield.

3.5. Effect of molar ratio on ML biodiesel yield

Following an elevation in temperature from 50°C to 60°C, there was a notable surge in biodiesel yield, escalating from 31% to 96%. Subsequently, a decline in yield was observed, plummeting from 96% to 50%, within the temperature range of 60°C to 70°C, as illustrated in Figure 3. The use of a 70°C reaction temperature surpasses the boiling point of methanol (64.7°C). Consequently, this results in the facile evaporation of methanol into the gaseous phase, thereby reducing the available methanol quantity for the reaction [10].

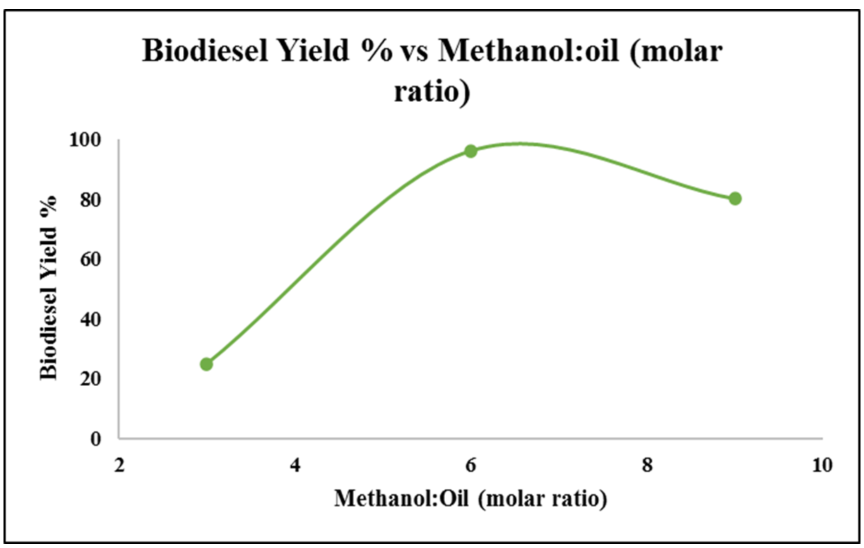

Figure 3. Effect of molar ratio on ML biodiesel yield.

3.6. Effect of reaction temperature on ML biodiesel yield

The biodiesel yield exhibited an ascent from 31% to 96% with a temperature rise from 50°C to 60°C. However, this yield subsequently declined from 96% to 50% within the temperature range of 60°C to 70°C, as depicted in Figure 4. The application of a 70°C reaction temperature surpasses the boiling point of methanol (64.7°C). Consequently, this leads to the rapid evaporation of methanol into the gaseous phase, resulting in a reduction of available methanol for the reaction [10].

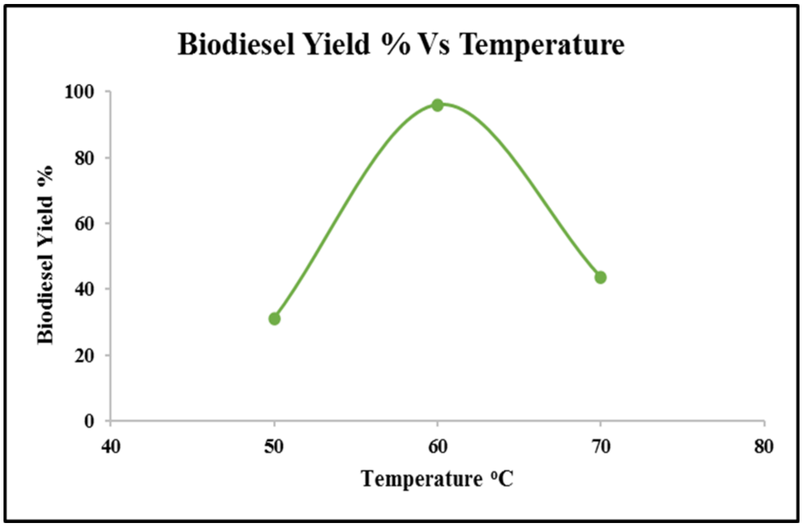

Figure 4. Effect of temperature on ML Biodiesel yield.

3.7. Effect of reaction time on ML biodiesel yield

When the reaction time was adjusted within the range of 40 to 70 minutes while keeping other parameters constant, a noteworthy increase in biodiesel yield was observed. Specifically, yields rose from 54% to 96% for reaction times of 40 minutes and 60 minutes, respectively, as depicted in Figure 5. However, with a further extension of the reaction time to 70 minutes, the yield decreased to 70%. This decline in yield can be attributed to the existence of an optimum time frame for biodiesel production to occur effectively. Prolonged reaction times may lead to the evaporation or degradation of biodiesel, and excessive time favors reversible reactions, ultimately reducing the biodiesel yield [11].

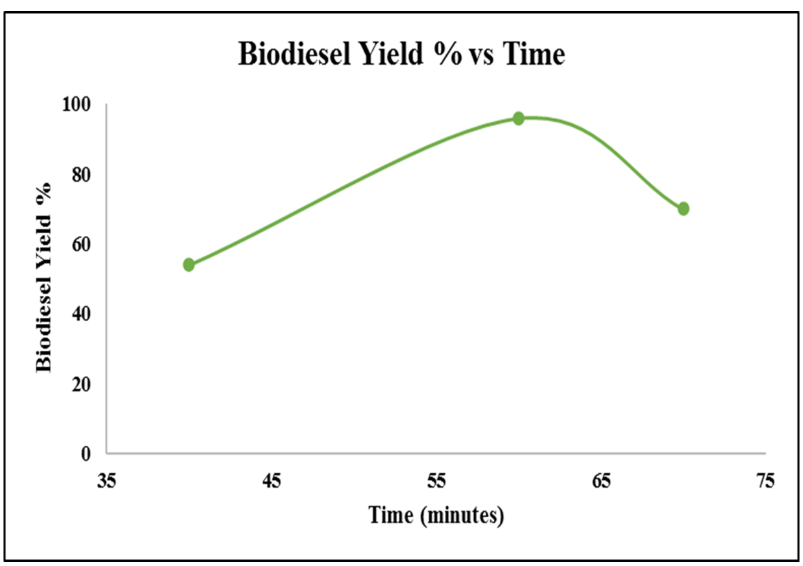

Figure 5. Effect of reaction temperature on ML biodiesel yield.

3.8. GC-FID

The identification of FAME was performed by using GC-FID and comparing authentic retention times. In performance of experiment 1.005 gm of sample was taken and heptane solvent (AR grade) was utilized. The oleic acid (18:1) peak was obtained at standard retention time of 27.899. Similarly Linoleic acid (18:2), Stearic acid (18:0), Palmitic acid (16:0), Lenonic acid (18:2), Ricinoleic acid (18:1).

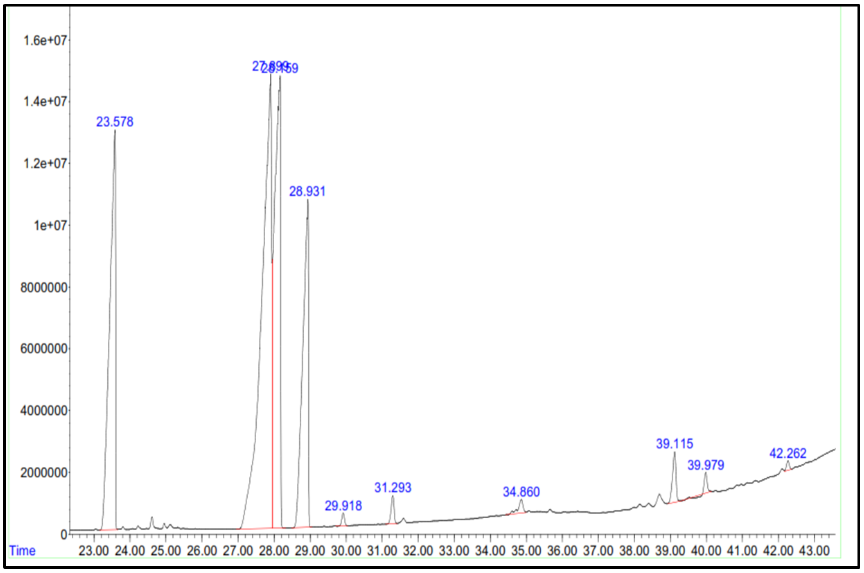

Figure 6. GC-FID chromatogram of the biodiesel produced from Mahua oil.

4. Technical, Economic & Environmental Benefits

Technical:

- Through meticulous experimentation, the study identified NaOH as a superior catalyst for biodiesel production from Madhuca Longifolia seeds, optimizing critical parameters such as reaction temperature and catalyst quantity to achieve a remarkable 96% yield.
- Utilizing advanced analytical techniques such as GC-FID, the quality of the produced biodiesel was rigorously assessed, ensuring compliance with ASTM standards and suitability for widespread use as a fuel.

Economic:

- The selection of NaOH as a catalyst offers a cost-effective solution for biodiesel production, presenting significant savings compared to alternative catalysts like KOH.
- By utilizing Madhuca Longifolia seeds as a low-cost feedstock, the study contributes to economic viability while minimizing waste through the utilization of by-products for additional purposes.

Environmental:

- Biodiesel production from non-edible sources like Madhuca Longifolia seeds reduces reliance on fossil fuels, mitigating greenhouse gas emissions and contributing to climate change mitigation efforts.
- The utilization of biodiesel in the transportation sector promotes air quality improvement by reducing emissions of harmful pollutants, fostering environmental sustainability and public health benefits.

5. Conclusions

This study embarked on an exploratory journey to transform Madhuca Longifolia seed oil into biodiesel through the process of transesterification. This involved the utilization of both homogeneous catalysts—specifically KOH and NaOH. Within the realm of homogeneous catalysis, the researcher meticulously fine-tuned critical variables such as reaction temperature, catalyst quantity, and the oil-to-molar ratio, aiming to understand their impact on biodiesel yield. The results unveiled NaOH as the superior performer over KOH, achieving an impressive 96% yield under identical reaction conditions. The optimal parameters for the transesterification process employing NaOH included 0.4 grams of the catalyst, a 1:6 ratio of oil to methanol molar proportions, a reaction temperature of 60°C, and a reaction duration of 60 minutes.

Biodiesel produced with a homogenous catalyst was characterized using GC-FID. The analysis confirmed that the biodiesel met the established norms. The heterogeneous catalyst was characterized through FTIR and TGA, revealing properties suitable for a biodiesel catalyst. The quality of the produced biodiesel was evaluated in accordance with the ASTM D6751 standard and found to meet the specified criteria.

In conclusion, this study highlights the effectiveness of NaOH as a catalyst in producing biodiesel with favorable attributes from non-edible Madhuca Longifolia seed oil.

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