

Synthesis, Characterization, and Production of Biodiesel from Tamarind Seed Oil

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Abstract

Because of the increasing number of power plants, automobiles, and factories, the increase in these automobiles and power plants produces more emissions of CO, HC, and NO_x. The world is searching for an alternative fuel that will not harm the environment and would be less costly. Biodiesel is one of the leading solutions to the global energy crisis, nonetheless, production from food-grade oils is not economically feasible due to high prices. Almost 80-85% of biodiesel is feedstocks. Presently, the use of non-edible waste oils is unable to support large-scale biodiesel production. Therefore, the search for other non-edible oil-bearing feedstocks should be continued. The present investigation attempts to use non-edible tamarind seed oil as an inexpensive, sustainable, and potent feedstock for biodiesel synthesis. Tamarind seed oil was converted to biodiesel by acid (HCl), base (KOH), and enzyme-catalysed transesterification. Tamarind seed biodiesel was found to have a low iodine value (27-33) and a high cetane number (66-68). The catalyst was characterised through acid, base, and enzyme catalysis, and the biodiesel was analysed using GC-MS and FTIR. The highest biodiesel yield of $96.57 \pm 0.81\%$ was achieved in 3.35 ± 0.15 h under optimal conditions of a 9:1 molar ratio of methanol to oil (MRMO) and a 15 wt% catalyst concentration at a temperature of 65°C. The reusability of the catalyst was successfully examined up to the third cycle, with $82.76 \pm 0.80\%$ biodiesel. The feasibility of the resultant methyl esters was verified through chromatography-mass spectrometry (GC-MS) analyses. The developed catalyst can be considered an active catalyst, benefiting from its cost-effectiveness and environmentally friendly nature.

Keywords: Tamarind, Transesterification, Biodiesel, Catalysis, Pour Point.

Introduction

Global warming, the insufficiency of fossil fuels, the ever-increasing population, the increasing costs of petroleum fuels, and climate change are the major issues driving the researcher's interest in developing alternative, sustainable, and renewable biofuels. (Jeswani, Chilvers, and Azapagic 2020)(Othman, Helwani, and Idris 2018). *Tamarindus indica* L., commonly known as the Tamarind tree, a member of the Leguminosae family, is one of the fruit species mainly grown in African countries. *Tamarindus indica* (TS) is a

renewable energy source in Nigeria, India, Malaysia, and Africa. The tree has a long life, and a massive area is reached under favourable conditions. The average tree is about 40 feet tall, and a single tree can provide about 10–90 kg of seeds. Tamarin seeds are not used and are considered waste; using them as a pyrolysis feedstock seems to be a promising opportunity to reduce waste and make a profit.

The thermochemical and biochemical processes are two major conversion technologies used to convert biomass into a distinct form of energy. The thermochemical process alters biomass into various forms of energy within a few hours or seconds, depending on the process, while the biochemical process takes days or months. Also, the thermochemical process technique is employed to transform plastic waste into renewable fuel, which microbial activities cannot break down. (Mateos 2025). Thermochemical techniques such as combustion, Hydrothermal Liquefaction, gasification, and pyrolysis are gaining considerable attention worldwide because they are more speedy than biological methods such as fermentation and anaerobic digestion. Among all the existing Thermochemical processes, pyrolysis has demonstrated greater responsiveness in the last few years due to its inherent advantage over other methods. Pyrolysis is the only thermochemical method that can produce energy in solid, liquid, and gaseous form. It can convert solid biomass into renewable fuel within seconds or minutes (depending on the pyrolysis type). Pyrolytic oil is a liquid product obtained from pyrolysis at a moderate temperature (400–700 °C). It has a black or brown colour with an irritating smell and is an option for fossil fuels and many chemicals. The pyrolytic oil has higher oxygenated compounds (35–60 wt.%) in various forms. (M et al. 2024). Before proceeding to the pyrolysis experiment, the investigation of kinetic analysis, such as frequency factor and reaction order, becomes crucial. Outstanding knowledge of kinetics parameters helps design and develop new pyrolysis, enhances various process constraints, and provides essential information about mathematical modelling simplification. TGA is the most efficient tool for estimating the kinetic analysis of biomass. A detailed kinetics study of biomass enlightened understanding of pyrolysis reaction mechanisms. For better data on pyrolysis reaction processes and process parameters to optimise, the kinetic parameters generated from pyrolysis are anticipated to provide precise data. (Rasaq et al. 2024).

These studies have shown a linear relationship between the two, with a change in product cost and biodiesel oil cost. Economically viable and cost-effective biodiesel can be produced from less expensive biodiesel oil feedstocks. However, the non-edible oils currently utilized are insufficient to meet the oil requirements for large-scale biodiesel production necessary to replace petrol fuels. Presently, only limited quantities of biodiesel are generated from non-edible sources, which are predominantly blended with petroleum diesel in low proportions ($\leq 20\%$). (Mushtaq et al. 2021)(Janković et al. 2024) In addition, to evaluate the multifaceted reaction of materials, model-free methods are used, which are given small or negligible errors. These methods can only validate an independent kinetic parameter for a specific conversion range (0.1–0.8).

Recently, GC/MS was recognised as the best tool among several other techniques for detecting pyrolytic products with better accuracy by equating GC–MS chromatographs in manifold operating situations. The main products include phenols, acids, amine hydrocarbons, and oxygenated chemicals. Also, the maximum n-hexadecanoic acid selectivity is 29.90 % at 400 °C (Baloyi and Patel 2024). Furthermore, Aromatic Compounds and petrol show distinct aromatic C=C stretching vibrations; tamarind oil may have fewer aromatic compounds, depending on its processing and composition. Understanding these spectral differences is crucial for evaluating the feasibility of tamarind-derived oil as an alternative fuel. The oxygenated compounds in tamarind oil could influence combustion properties, emissions, and energy content. Further studies, including performance testing and refining processes, are necessary to optimize tamarind-derived oil for use in engines designed for petroleum-based fuels.

The current study employs an innovative approach that involves the fractionation of tamarind seed oil (TSO) and subsequent transesterification to produce biodiesel. *Tamarindus indica* L. (tamarind), a member of the Fabaceae (Leguminosae) family, is primarily cultivated in regions of Asia and Africa. The seeds constitute approximately 40 wt.% of the fruit, representing an underutilised by-product. The composition of tamarind seeds includes 12–20% fiber, 50–55% carbohydrates, 20% tannins, and 4.5–16.2% oil. The presence of coloring compounds, such as tannins and other toxic substances, renders the seeds unsuitable for human consumption. Previous research has examined the emission characteristics of biodiesel derived from tamarind seed oil and its blends. Consequently, this non-edible seed oil serves as a viable alternative feedstock for biodiesel production. In this study, TSO was separated into distinct fractions using high vacuum fractional distillation, which, after subsequent transesterification, produced biodiesel with improved low-temperature performance compared to biodiesel from unfractionated TSO.

Materials and Methods

Sample collection and preparations

The *tamarind indica* seeds were purchased from the traditional market in Mubi-North, Adamawa State. The assembled seeds were distributed in distilled water, exposed to the sun for 24 to 48 hours, and then dried in a hot air oven for 1 hour at 105°C to eliminate homogeneous physically bound moisture. To achieve the suitable particle size (1 mm), the sun-dried feedstock was crushed and stored in air-tight plastic bags to prevent moisture. Tamarind seed oil was subjected to acid, base, and enzyme-catalyzed transesterifications of biodiesel. Base-catalyzed transesterification was carried out using pellets of potassium hydroxide (KOH) at concentrations of 0.2%, 0.4%, 0.6%, 0.8%, and 1% (w/w oil). The diverse concentrations of catalysts were mixed with tamarind seed oil (5 g) and methanol (2 g). The combination of oil, catalyst, and methanol was mixed on a magnetic stirrer at 60°C for 90 minutes. In acid-catalyzed transesterification, hydrochloric acid (HCl) at 20%, 40%, 60%,

80%, and 100% (w/w oil) was mixed with oil, dissolved in methanol and stirred on a magnetic stirrer for 4 hours at 60 °C. The molar ratio of oil to methanol was 6:1. In case of enzyme-catalyzed reactions, lipase immobilised on calcium alginate beads at concentration levels of 1%, 2%, 3%, 4%, and 5% (w/w oil), was mixed with methanol and oil, under constant refluxing at 40 °C for 24-hrs. The molar ratio of methanol to oil was 5:1. After the reaction completion, the mixture was placed overnight for the separation process of diesel from glycerol. Warm distilled water was used to remove excess methanol, unreacted catalyst, and soap. The washed product was purified to remove moisture. The final good quality biodiesel was subjected to chemical analysis. Fuel composition was tested by GC-MS analysis (Cozendey et al. 2025)(Alemu, Nallamotheu, and Lakew 2025)[3]. The density (g/ml) was determined by measuring the mass of 1 ml of each sample (Santhosh et al. 2025)(Koshani et al. 2025). Also, the reaction mixture was transferred to a separation funnel and left to rest for 24 hours, then the formation of two phases was observed: the upper one, containing biodiesel, and the lower one, containing glycerol, soaps and excess base and alcohol. The lower phase was collected in a 100 mL beaker and taken to the waste disposal site. 25 mL of 10% hydrochloric acid aqueous solution was added to the funnel containing the remaining phase, leaving it to rest for 15 minutes. The aqueous phase was removed, and the washing process was repeated with another 25 mL of 10% hydrochloric acid. Then, washing was done twice with 20 mL of saturated NaCl solution and once with distilled water. The absence of a catalyst was confirmed by measuring the pH of the last washing water, which must be neutral. To dry the biodiesel, the solution obtained was transferred to an Erlenmeyer flask and anhydrous Na_2SO_4 (desiccating agent) was added and waited 15 minutes. Afterwards, the mixture was filtered and the fraction obtained (biodiesel) was transferred to a beaker to measure its volume. The biodiesel obtained had a clean appearance and no turbidity.

Pry-FTIR analysis

The tests were conducted in an SS-304 chamber, a Brill cell part of a Pyroprobe 5150 series pyrolyzed (CDS Analytical, USA). It contains two optical windows of anti-reflection zinc selenide locked in place by brass end-caps within a cylindrical chamber. During the study, inert argon gas continually flowed through the chamber at 7 L h⁻¹. The chamber also included inlet and outflow vents. This flow rate was refined through many tests to prevent condensation inside the cell and maintain the vapours long enough for the FTIR spectrometer to scan them. The pyrolysates were prevented from condensing within the Brill cell by retaining the interface temperature at 200 °C. A Cary 660 FTIR spectrometer sample container was used for the whole setup. The Brill cell was positioned to continually study the pyrolysates so that the optical slits were in the FTIR beam path. The aim of this study is, sample 8 ± 0.5 mg. The studies were implemented at 500 °C with a fixed probe heating rate of 20,000 °C s⁻¹ for the 60 s pyrolysis period. A linear, sensitive MCT detector was the FTIR detector. The pyrolysates were examined at a 2 cm⁻¹ resolution range of

4000–600 cm^{-1} wave number. At 3 scans per second, the total scan time was kept at 40 scans per second.

Results

Preparation of Biodiesel

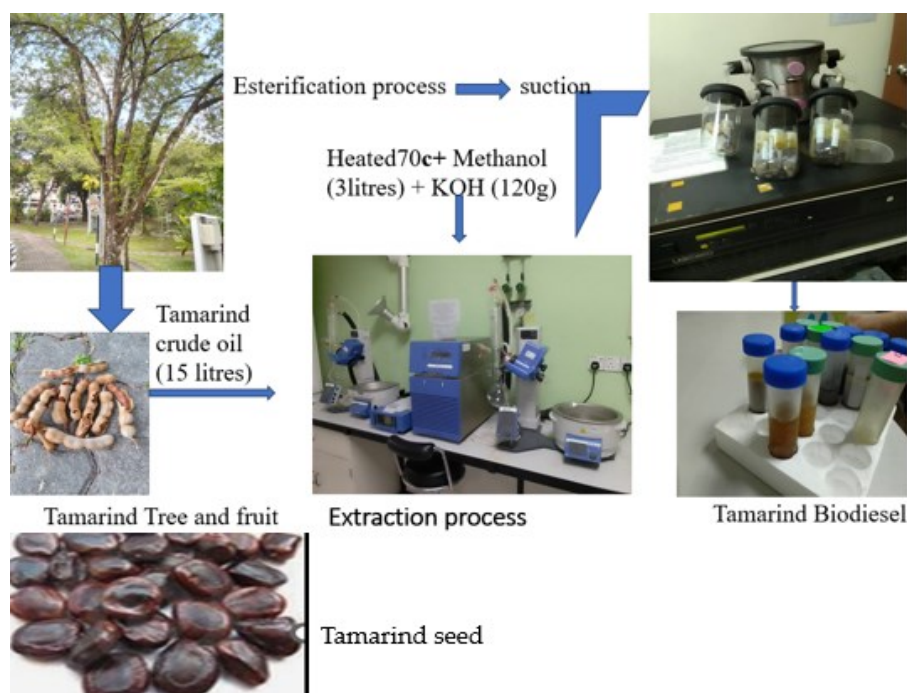


Fig 1. Preparation and production of Biodiesel from Tamarind seed.

Pyr-FTIR analysis

The FTIR spectrum of petrol typically shows: -C-H stretching vibrations, Absorption bands 2950 cm^{-1} , 2930 cm^{-1} , and 2850 cm^{-1} , corresponding to different types of C-H bonds in alkanes. Aromatic C=C stretching vibration bands near 1600 cm^{-1} and 1500 cm^{-1} indicating the presence of aromatic compounds. C-H bending near vibrations: - Absorption around 1465 cm^{-1} and 1375 cm^{-1} , associating with bending modes in $-\text{CH}_2-$ and $-\text{CH}_3-$ groups. These features reflect the complex mixture of hydrocarbons, including alkanes, cycloalkanes and aromatics, that constitutes petrol. When comparing the FTIR spectra of tamarind-derived oil and petroleum-based petrol. Hydroxyl groups and carbonyl groups -OH and C=O groups in tamarind oil suggest a higher content of oxygenated compounds, which are less prevalent in conventional petrol. Both hydrocarbon spectra exhibit C-H stretching and bending vibrations, indicating the presence of hydrocarbons. However, the specific patterns and intensities may differ due to variations in hydrocarbon chain lengths and structures. Aromatic compounds, petrol, show distinct aromatic C=C stretching vibrations, while tamarind oil compounds are based on their processing and composition.

Aromatic Compounds, Petrol shows distinct aromatic C=C stretching vibrations, while tamarind oil may have fewer aromatic compounds, depending on its processing and composition. Understanding these spectral differences is crucial for evaluating the feasibility of tamarind-derived oil as an alternative fuel. The oxygenated compounds in tamarind oil could influence combustion properties, emissions, and energy content. Further studies, including performance testing and refining processes, are necessary to optimize tamarind-derived oil for use in engines designed for petroleum-based fuels.

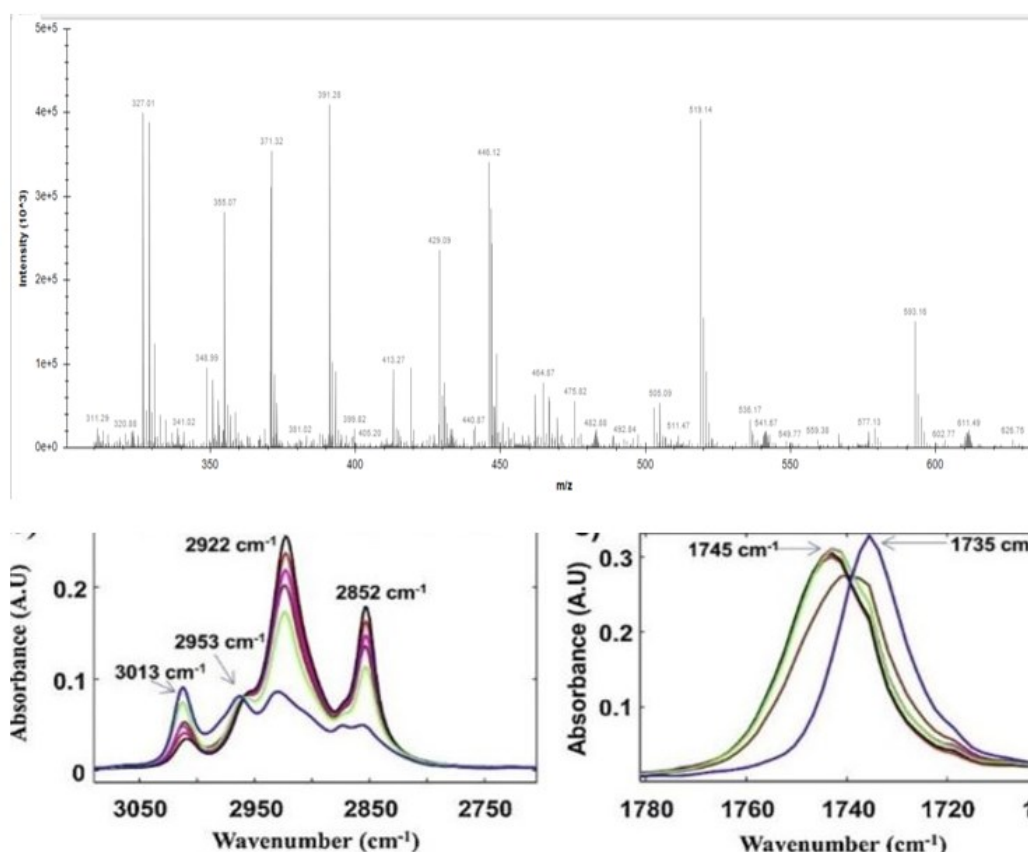


Fig 2. Chromatographic/GC-MS profile of a ternary sample of biodiesel (B519.14) and diesel. The biodiesel obtained had a clean appearance and no turbidity.

Results and Discussion

In the case of acid-catalyzed reactions, five concentrations of HCl were used to optimise the yield of biodiesels. Biodiesel yield increased with increasing catalyst concentration. A yield of 97% was obtained using HCl 40% (w/w). Biodiesel yield was decreased by a further increase in catalyst concentration, because of the decomposition of fatty acids at higher acid concentrations. Minimum yield (89%) was observed at HCl 100% (w/w). The same trend was recorded earlier during the making of biodiesel from rubber seed oil [5]. In the case of base-catalyzed biodiesel production, the maximum yield (86%) was achieved at KOH 0.6% (w/w). Biodiesel yield was reduced at higher concentrations due to gel through

soap formation. Minimum yield (77%) was obtained at KOH 1% (w/w). A similar trend was observed in another study, throughout the production of biodiesel from waste soybean oil [6]. Five concentrations of immobilised lipase were used to synthesise tamarind seed biodiesel. The optimum biodiesel yield (89%) remained on the available active site at lipase 3%. GC-MS analysis was performed to quantify the esters present in tamarind seed biodiesel. Palmitic acid, stearic acid, and linoleic acid, fatty acids in tamarind seed biodiesel. Caprylic acid, myristic acid, capric acid, behenic acid, gondoic acid, tricyclic acid, and arachidic acid were present in trace amounts,

Biodiesel is composed of a diverse array of fatty acid methyl esters, with the specific proportions of these esters determined by the feedstock applied in its production. Consequently, the physical and chemical properties of biodiesel are inherently linked to its fatty acid methyl esters composition ((Inam, Khan, and Nadeem 2019)). Various feedstocks can be employed in the synthesis of biodiesel, including animal fats, vegetable oils, municipal and industrial wastes, as well as insect-derived materials.(Mushtaq et al. 2021). Each of these feedstocks exhibits distinct fatty acid variations in the chemical composition and physical characteristics of the resultant biodiesel. These differences are crucial, as they can significantly influence both engine performance and the overall fuel properties of biodiesel.(Kalsoom et al. 2017).

Conclusions

The fuel properties of *Tamarindus indica* L (Tamarind) seed oil biodiesel were equivalent to and, in some cases, superior to conventional biofuels. The presence of low-molecular-weight fatty acids in Tamarind seed oil makes it a suitable feedstock for biodiesel production. Additionally, tamarind seed biodiesel has been found to have higher cetane numbers compared to biodiesel feedstocks. The impact of different catalysts on biodiesel yield is illustrated in Figure 1. Base-catalyzed transesterification offers several advantages, including lower catalyst costs compared to acid- and enzyme-based alternatives, greater market availability, milder reaction conditions, and faster reaction rates when compared to enzyme- and acid-catalyzed processes. Gas chromatography-mass spectrometry (GC-MS) analysis of tamarind seed oil (TSO) revealed significant fatty acid compositions, primarily linoleic acid (31.8%) and oleic acid (17.6%) (Figure 2). These findings suggest that hydrochloric acid (HCl) yielded the highest biodiesel production from TSO and its fractions. The reduced cloud and pour points of tamarind seed biodiesel make it a viable substitute for conventional fuels, especially in colder regions.

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Conflicts of Interest: The authors declare no conflict of interest.

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