Journal of Mines, Metals and Fuels, *73(11): 3641-3650; 2025. DOI: 10.18311/jmmf/2025/49456*

Print ISSN : 0022-2755

Journal of Mines, Metals and Fuels

Contents available at: *www.informaticsjournals.co.in/index.php/jmmf*

Synthesis of Biodiesel from Extracted *Senna tora* Seeds Oil and Optimization Study

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Abstract

This study investigates the extraction of Senna tora seed oil using soxhlet and batch extraction methods to evaluate oil recovery efficiency and optimize process parameters. The soxhlet extraction method was employed to determine the maximum possible oil recovery, while the batch extraction method was assessed for its commercial viability. Various process parameters, including solvent selection, particle size, extraction temperature, and agitation speed, were analysed to enhance oil recovery in batch extraction. Among the tested solvents, petroleum ether was identified as the most suitable, yielding an optimal oil recovery of 66.21% at a particle size of 1.125 mm and an extraction temperature of 50° C. The extracted oil, rich in essential fatty acids, exhibits potential for both nutritional and industrial applications. Given the high oleic acid content, S. tora seed oil could be valuable in the oil industry. Furthermore, biodiesel production was optimized via transesterification using methanol as the solvent and NaOH as the catalyst under the conditions of 1-hour reaction time, a 1:6 molar ratio, 1.2 wt% catalyst concentration, and a reaction temperature of 65° C. This process yielded 87.6 mL of biodiesel and 28.4 mL of glycerol, achieving a conversion efficiency of 87.6%. The potential of S. tora seed oil, as a sustainable feedstock for biodiesel production and industrial applications, contributing to its economic viability and utilization in renewable energy sectors.

Major Findings: *The use of S. tora oil, a non-edible and locally available feedstock, contributes to sustainability by minimizing food resource competition and reducing the carbon footprint of diesel fuel.*

Keywords: *Biodiesel, Cassia Tora, Particle Size, Recovery, Yield, Soxhlet Extractor, Transesterification, Viscosity*

1.0 Introduction

The production of biofuels has gained significant attention due to increasing concerns about the availability of petroleum-based fuels and their impact on rising atmospheric CO_2 levels[1-7]. The growing use of fossil fuel-derived diesel exacerbates global warming through CO_2 emissions and contributes to air pollution[8]. Biodiesel has emerged as a promising alternative fuel due to its biodegradability, renewability, non-toxicity, lower gaseous emissions, and higher cetane number compared to petroleum diesel. Additionally, it is more cost-effective than conventional fuels[9].

Biodiesel addresses the rising global energy demand while enhancing energy security, which is currently reliant on depleting petroleum-based resources[10]. The utilization of biodiesel in diesel engines significantly reduces particulate matter, CO_2 emissions, smoke, and noise due to its higher oxygen content compared to petroleum diesel. Furthermore, since these fuels are derived from agricultural sources that undergo photosynthetic carbon fixation, their combustion does not contribute to net atmospheric CO_2 levels[11].

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Petroleum fuels such as gasoline, diesel, and jet fuel are composed of complex hydrocarbon mixtures. According to Runge, Camelina and Jatropha are two promising plant-based biofuels with global availability. These flowering plants offer advantages over traditional seed-based fuels like soybean as they can be cultivated in arid regions without encroaching on agricultural land. Despite their potential, Peter Taglia of Clean Wisconsin raises concerns about the large-scale adoption of Jatropha and similar feedstocks. He cautions that if these crops replace food production or natural ecosystems, they may create new environmental challenges. While Camelina and Jatropha offer unique advantages, their full potential remains untapped within the biofuel sector.

Hayatuddeen *et al*. reported that biodiesel, derived from various triglyceride sources, can effectively replace petro-diesel[12]. Their study focused on *Cassia tora* seed oil, which was esterified to reduce its Free Fatty Acid (FFA) content. A base-catalyzed transesterification process using sodium hydroxide pellets and methanol successfully converted the oil into biodiesel, with an initial FFA level of approximately 6.45%. To improve biodiesel yield, a two-step acid pre-treatment was performed to further reduce FFA content.

Ramakrishnan *et al*. examined the oil content of mature *C. tora* seeds to assess their nutritional and antioxidant properties[13]. The seed oil (41.8%) contained significant levels of unsaturated fatty acids, including oleic acid (13.31±1.72%), linoleic acid (11.25±1.13%), palmitic acid (12.78±1.21%), and free fatty acids (22.15±1.71%). The oil was found to be rich in amino acids (21.09±1.69%), making it valuable for food formulations aimed at treating oxidative and cardiovascular diseases.

Mankilik *et al.* investigated the physicochemical and fatty acid composition of *C. tora* seed oil[14]. Using soxhlet extraction with petroleum ether as the solvent, they obtained an oil yield of 17.76%, composed primarily of oleic acid (43.21%), stearic acid (19.37%), palmitic acid (15.83%), and other compounds (21.59%).

Yunfeng *et al*. analyzed the volatile components of *C. tora* seed oil using gas chromatography and mass spectrometry after supercritical CO_2 extraction[15]. The primary constituents included (Z,Z)-9,12-octadecadienoic acid (26.74%), oleic acid (24.15%), n-hexadecanoic acid (13.99%), chrysophanol (7.26%), (E)-9-octadecenoic acid (4.52%), and octadecanoic acid (4.44%).

Morakinyo *et al*. characterized biodiesel-diesel blends based on *C. tora* Seed (CTS) oil[16]. Their study revealed that blends containing 5%, 12.5%, and 20% CTS oil met the required physicochemical properties for combustion engines.

Yashvir *et al*. emphasized the necessity of exploring environmentally friendly fuels due to the rising pollution levels caused by fossil fuel usage[17]. They employed Response Surface Methodology (RSM) to optimize key variables influencing engine performance and emissions when using *C. tora* biodiesel blends in direct-injection diesel engines.

Yen *et al*. evaluated the antioxidant capacity of two *Cassia* species, *C. tora* L. and *Cassia occidentalis* L. Methanolic extracts from these species exhibited superior antioxidant activity and higher extract yields compared to other organic solvents[18].

Wong *et al*. identified novel naphthopyrone glycosides, including cassiaside and rubrofusarin-6-3-gentiobioside, from *C. tora* seeds[19].

Choi *et al.*[20] demonstrated the antimutagenic activity of a methanol extract from Cassia tora seeds against aflatoxin B1 (AFB1) using the Salmonella typhimurium assay.

Despite significant progress, several research gaps remain. There is a need for standardized methodologies in oil extraction and biofuel production to enhance comparability and reproducibility. Currently, a variety of techniques are used for *Senna tora* seed oil extraction and biofuel synthesis, requiring further research to determine the most efficient and sustainable approaches. Additionally, studies should evaluate the environmental impact of different processing methods and chemical solvents used in biofuel production.

Developing environmentally friendly biofuel production techniques, such as bio-based chemical applications and non-toxic solvent use, is essential. Further research is also needed to optimize catalysts and chemical additives in *S. tora* biodiesel production. While catalysts enhance production efficiency, their economic and environmental impacts must be carefully assessed.

Finally, the optimization of advanced processing techniques, including hydrodeoxygenation and transesterification, requires further exploration. Each method impacts biofuel quality, efficiency, and environmental footprint differently. Though *S. tora* oil contains significant fatty acids, essential for biofuels,

its viability as a large-scale biofuel source remains underexplored. Challenges related to oil extraction, fuel processing, and economic feasibility must be addressed to determine its full potential.

Notably, no prior research has investigated *S. tora* seed oil extraction using hexane as a solvent or biodiesel production via transesterification. This study explores these aspects by analysing oil recovery at varying raw material sizes and temperatures, providing valuable insights into optimizing *S. tora* as a biofuel source.

2.0 Materials and Methods

In this study, *S. tora* oil was used as the substrate, extracted from the *S. tora* plant sourced from Uttar Pradesh, India, for biodiesel production.

2.1 Equipment

The equipment used in this study included a syringe, burette, hand gloves, pH meter, weighing balance, hot plate, 250 mL bottom flask, weighing pan, specific gravity bottle, industrial sieve (70 µm pore size), beaker, soxhlet extractor, filter paper, retort stand, dropper, thermometer, and oven.

2.2 Chemicals or Reagents

For completeness, the extraction time was 6 hours, the solvent used was hexane, and the extraction temperature was maintained at approximately 60° C throughout the soxhlet process.

Hexane, methanol, distilled water, pellets of sodium hydroxide, and phenolphthalein.

2.3 Extraction of Oil from *S. tora* Oil

In this study, 70 g of crushed *S. tora* seeds were used, and the thimble was filled with the crushed seeds. Prior to filling, a layer of cotton was placed inside the thimble to provide support and function as a strainer. A total of 250 mL of hexane was added to a round-bottom flask, which was then placed on a heating mantle set to 600° C. The Soxhlet apparatus was assembled on the round-bottom flask, and a condenser was attached, as illustrated in Figure's 1 and 2. A continuous water supply was provided to the condenser to facilitate the removal of latent heat of condensation.

A thermometer was inserted into one of the necks of the round-bottom flask to monitor the temperature. Heating was initiated until the hexane solvent began to boil, which was observed at approximately 60° C. This temperature was maintained throughout the extraction process. The experiment was conducted for 6 hours,



Figure 1. Experimental setup for oil extraction in soxhlet extractor.



(a) **(b)**

Figure 2. **(a)** Single stage separation of mixture. **(b)** Separated liquids.

during which four complete extraction cycles were completed.

Following the extraction, the solution was subjected to a single-stage separation process. After separation, the solvent was recovered as the raffinate phase, while the extracted oil was obtained as the extract phase, yielding approximately 11.16 g of oil.

2.4 Determining the Properties of *S. tora* Oil

The properties of *S. tora* oil were analyzed and are presented in Table 1 and Table 2.

2.5 Biodiesel Optimization using Trans-esterification reaction

Transesterification Process and the Role of Methanol

Transesterification, also known as alcoholysis, is a chemical process in which alcohol reacts with inedible oil to produce biodiesel. Methanol and ethanol are the most commonly used alcohols in this process due to their availability and cost-effectiveness. This reaction facilitates the conversion of triglycerides into esters, significantly

Table 1. Fatty Acids Composition of Senna Tora Seed Oil

Fatty acids	Value (%)
Oleic	43.21
Palmitic	15.83
Stearic	19.37
Linoleic	15.38
Behenic	6.21

Table 2. Comparison of Physical Properties of Senna Tora Seed Oil

Property	Values	Ideal Values[17]
Specific gravity	0.87	0.88
Refractive Index at 25°C	1.46	1.43
pH at 24.1oC	4.3	4.41
Color	Golden Brown	Golden Brown

reducing the viscosity of inedible oils, making them suitable for biodiesel production.

Transesterification can be categorized into two types: Catalytic and non-catalytic transesterification. Catalytic transesterification, though widely used, presents certain challenges. The process is labour-intensive and requires extensive separation of saponified impurities, unreacted alcohol, oil, and catalyst from the final biodiesel product. In contrast, the supercritical transesterification process eliminates the need for a catalyst, preventing soap formation and simplifying the purification process. However, the high temperature and pressure requirements for supercritical alcohol transesterification significantly increase equipment costs, making the process economically demanding.

Methanol, an organic compound derived from vegetable sources, plays a crucial role both as a fuel and as a chemical reagent. It is a relatively inexpensive alcohol, appearing as a clear, colourless liquid with a mild alcoholic Odor. Methanol exhibits high solubility in a wide range of organic solvents, including alcohols, ketones, esters, and ethers. It is widely preferred in transesterification reactions over higher alcohols such as ethanol, butanol, and propanol, as these alternatives tend to reduce the efficiency and cost-effectiveness of the reaction. Additionally, in supercritical transesterification, methanol is favoured due to its lower dielectric constant in the supercritical state, enhancing the solubility of vegetable oils and improving overall reaction efficiency.

2.6 Procedure

To produce biodiesel, 100 mL of *S. tora* oil, 23 mL of methanol, and 1.02 g of NaOH were accurately measured. The measured chemicals and catalyst were thoroughly mixed in a conical flask before being transferred to a three-neck round-bottom flask. A thermometer was inserted into one neck of the flask, while the second neck was sealed with a stopper. A condenser was attached to the third neck, and a continuous flow of cold water was maintained for efficient condensation.

The reaction mixture was heated using a heating mantle, maintaining a temperature of 60° C for 1 hour. After completion of the reaction, the solution was allowed to cool and then removed from the round-bottom flask. The cooled mixture was transferred to a separating funnel and left undisturbed for 6 to 8 hours to facilitate phase separation. The biodiesel formed the top layer, while glycerol settled at the bottom.

To remove residual catalyst (NaOH) and methanol, the biodiesel layer was washed with water and allowed to stand for another 6 to 8 hours, enabling further separation. The purified biodiesel remained in the top layer, while the bottom layer contained residual byproducts. The final biodiesel was then analyzed for its physicochemical properties and compared with standard biodiesel specifications to assess its quality and suitability as a fuel.

2.7 Washing of Biodiesel

In this study, biodiesel was synthesized using *S. tora* oil through a transesterification process. A precise quantity of 100 mL of *S. tora* oil, 23 mL of methanol, and 1.02 g of NaOH were measured and thoroughly mixed in a conical flask. The mixture was then transferred to a three-neck round-bottom flask. A thermometer was inserted into one of the necks for temperature monitoring, while another was sealed with a stopper. A condenser was attached to the remaining neck, with a continuous flow of cold water maintained to facilitate condensation.

The reaction was carried out by heating the mixture using a heating mantle at a constant temperature of 60° C for one hour. Upon completion, the reaction mixture was allowed to cool before being removed from the round-bottom flask. The cooled solution was then transferred to a separating funnel and left undisturbed for 6 to 8 hours to enable phase separation. The upper layer, containing biodiesel, was carefully separated, while the lower layer, consisting of glycerol, was discarded.

To eliminate residual NaOH and methanol, the biodiesel was subjected to a washing process using water, followed by an additional settling period of 6 to 8 hours for effective separation of impurities. The purified biodiesel was then collected, and its physicochemical properties were analysed and compared against standard biodiesel specifications to evaluate its fuel quality and suitability.

2.8 Determining the Properties of Biodiesel

The properties of *S. tora* oil were analyzed and are presented in Table 4.

3.0 Result and Discussion

The extraction of oil from Senna Tora seeds using the Soxhlet extraction method yielded 159.55 g of oil from



(a) **(b)** **(c)**

Figure 3. **(a)** Experimental set up. **(b)** Single stage separation of mixture. **(c)** Separated liquids.



(a) **(b)**

Figure 4. **(a)** Water wash experimental setup or single stage separation. **(b)** Separated biodiesel.

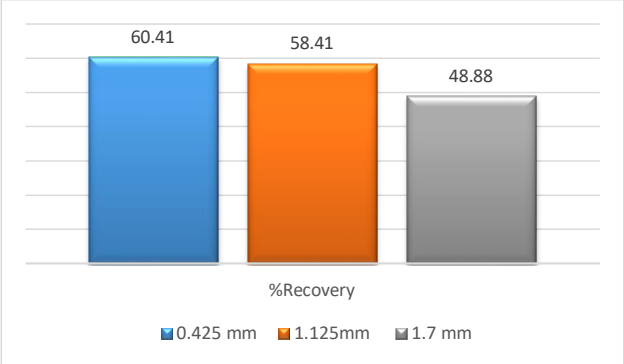


Figure 5. Graphical representation of % recovery of oil at different particle sizes.

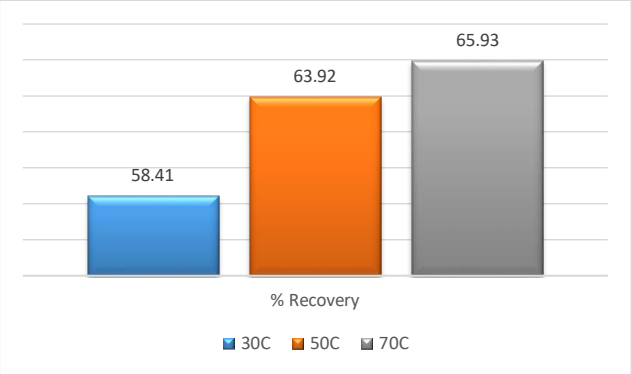


Figure 6. Graphical representation of % recovery of oil at different temperature.

257 g of seeds, resulting in an extraction efficiency of 62.08%. Additionally, the percentage recovery of oil was evaluated under varying parameters, including different particle sizes and extraction temperatures, to optimize the extraction process.

The diagram illustrates that as particle size increases, the percentage of oil recovery decreases due to the reduction in surface area available for extraction. The data indicate that particles with a size of 0.425 mm achieved a recovery rate of 60.41%, while particles of 1.125 mm and 1.7 mm exhibited recovery rates of 58.41% and 48.88%, respectively, as shown in Figure 5.

The diagram illustrates that as temperature increases; the percentage of oil recovery also increases. This is because higher temperatures accelerate reaction rates and enhance mass transfer efficiency, leading to faster conversion of reactants, improved yield, and better separation efficiency. The data indicate that at 30° C, the recovery was 58.41%, which increased to 63.92% at 50° C and further to 65.93% at 70° C, as shown in Figure 6.

The diagram illustrates that as temperature increases;

6. Specific gravity

Weight of empty sample -9.65g
Weight of botte with oil – 14.06 g
Weight of bottle with water – 14.72g

$$\text{Specific Gravity of Oil} = \frac{\text{Wt of bottle and oil sample} - \text{Wt of empty bottle}}{\text{Wt of bottle with water} - \text{Wt of empty bottle}}$$

$$\text{Specific Gravity of Oil} = \frac{14.06 - 9.65}{14.72 - 9.65}$$

= 0.869

The diagram indicates that as temperature increases, biodiesel production also increases. Higher temperatures accelerate reaction rates and enhance mass transfer efficiency, leading to faster reactant conversion, improved yield, and better separation efficiency, ultimately enhancing overall process productivity. The data show that at 60° C, 82.5 mL of biodiesel was produced, which

Table 3. Temperature Difference Variation

Temp	Reaction Time	Catalyst	Molar ratio (alcohol : oil)	Amount of oil and alcohol	Amount of biodiesel produced ml	Amount of Glycerol produced ml	% Yield
60° C	1 hr	1.2% of oil (1.02gm)	1:6	Oil=100 alcohol=23	82.5 ml	33.2 ml	82.5 %
65° C	1 hr	1.2% of oil (1.02gm)	1:6	Oil=100 alcohol=23	87.6 ml	28.4 ml	87.6 %
70° C	1 hr	1.2% of oil (1.02gm)	1:6	Oil=100 alcohol=23	93 ml	29.4	93 %

Table 4. Comparison of physical properties of *S. Tora* biodiesel oil

Properties	ASTM standards[15]	*S. tora* Biodiesel Oil
Density	0.75-0.84	0.846
Kinematic Viscosity (mm^2/s)	1.9 - 6.0	4.2
Dynamic viscosity kgm-1s-1	4.3	$6.49*10^{-3}$
Acid value (MgKoH/g)	0.1	0.16

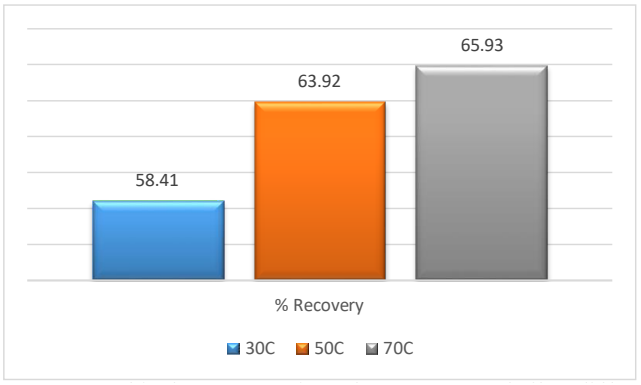


Figure 7. Graphical representation of biodiesel produced at different temperature.

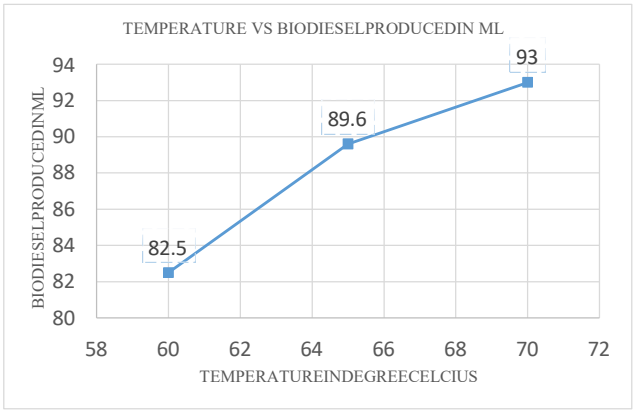


Figure 6. Graphical representation of % recovery of oil at different temperature.

increased to 89.6 mL at 65° C and further to 93 mL at 70° C, as illustrated in Figure 7.

This study confirms the sustainability benefits of *S. tora* oil biodiesel production, particularly In reducing net CO_2 emissions and offering a renewable, eco-friendly alternative to fossil fuels. In the diagram, the effect of temperature variation on biodiesel synthesis through the

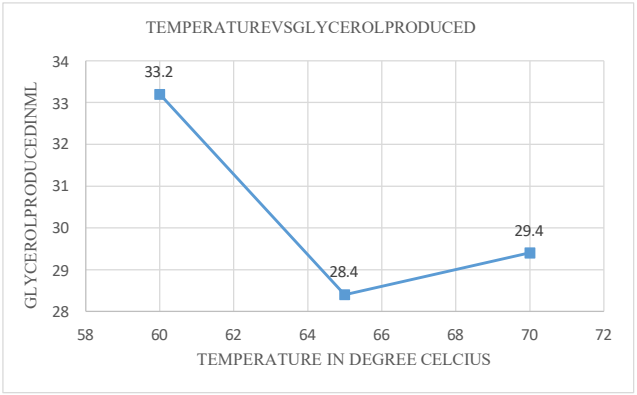


Figure 8. Graphical representation of glycerol produced at different temperature

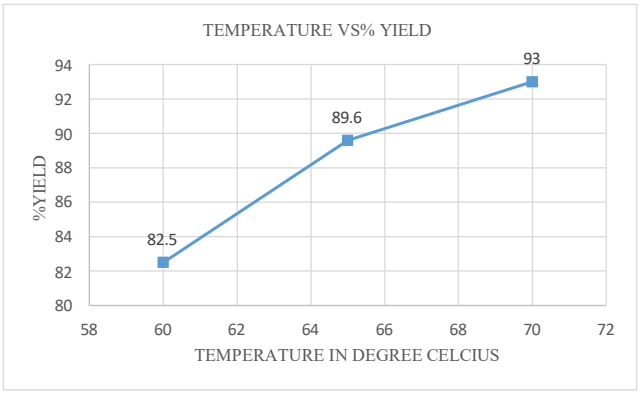


Figure 9 Graphical representation of % yield at different temperature.

transesterification process is analyzed. The observations indicate that at 60° C, the biodiesel yield is 82.5 mL, increasing to 87.6 mL at 65° C and reaching 93 mL at 70° C, as illustrated in Figure 3 and presented in Table 8.

4.0 Conclusion

This study demonstrates that oil recovery from *S. tora* seeds increases as particle size decreases. The maximum oil recovery of 66.21% was achieved under optimized extraction conditions, including a particle size of 1.125 mm, a temperature of 50° C, and an agitation speed of 550-600 rpm. The extracted *S. tora* oil exhibits potential for industrial applications.

Furthermore, the results indicate that increasing the reaction temperature enhances biodiesel production while reducing glycerol formation. Higher temperatures accelerate the transesterification reaction, improving biodiesel yield, increasing reaction rates, and minimizing

the time required for maximum conversion. Additionally, given its medicinal properties, *S. tora* oil holds promise for applications beyond biofuel production, potentially contributing to its economic value.

5.0 Future Work

Future studies should focus on scaling up biodiesel production and exploring its practical application in motorcycles and as a renewable substitute for petroleum diesel. Additionally, refining the experimental setup to address identified limitations could further improve efficiency and yield.

6.0 Author Contributions

Dr. Mahesh A. Bote: Conceptualization, methodology, Data analysis, writing – original draft.

S. A. Chougule: Experimentation, validation, resources, writing – review & editing.

S. G. Mane: Supervision, project administration, critical revision.

7.0 Acknowledgment

The authors express their gratitude to the Department of Mechanical Engineering, Sanjay Bhokare Group of Institutes, Miraj, for providing laboratory facilities and support during the course of this research. The authors also thank Fabtech College, Sangola, for their cooperation and technical assistance.

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