Journal of Bioresources 10 (2): 30–35, July – December 2023

JOURNAL OF BIORESOURCES

ISSN: 2394-4315 (Print)
ISSN: 2582-2276 (Online)

journal webpage: https://jbr.rgu.ac.in

RESEARCH ARTICLE

Production and optimization of biodiesel produced from seeds of *Sesamum indicum* L.

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Article No.: KJBR07; Received: 15.04.2023; Revised: 05.05.2023; Accepted: 15.10.2023; Published: 31.12.2023
Doi: https://doi.org/10.5281/zenodo.10513653

Abstract

The emergence of alternative fuels is prompted by the environmental problems associated with the utilization of petroleum-based products and the geopolitical tactics used to manipulate crude oil. Fatty acid methyl ester is the primary component of biodiesel, a non-toxic and biodegradable alternative fuel for diesel engines. Making bio-diesel from vegetable oils is the most effective technique to use them as fuel in compression ignition engines. Animal fat and both edible and non-edible vegetable oils are used to create bio diesel, an alkyl ester of fatty acids. In the present study, biodiesel was produced from a raw Sesame oil extracted from the seeds of *Sesamum indicum* L. (Lamiaceae) using double stage transesterification process. During the process of experimentation, different parameters were measured such as molar ratio, reaction temperature, reaction time, and catalyst concentration to determine the best parameter for optimal bio-diesel yield. The maximum biodiesel yield of 95% was obtained at 1.5% catalyst concentration, 3:1 molar ratio, reaction temperature of 55 minutes. The different properties of bio-diesel like calorific value, density, dynamic viscosity, kinematic viscosity, cloud point, pour point, and oxidation stability were measured. All test fuels physio-chemical parameters were analyzed and compared with those of diesel Standards (IS: 15607) to decide the rationality of utilizing Biodiesel from Sesame oil as a fuel for compression ignition engines.

Keywords: *Sesamum indicum*; Biodiesel; Catalyst; Compression Ignition; Alkyl ester; Transesterification.

1. Introduction

India produces over 6,80,000 tons of Sesame oil annually, making it the second-largest producer in the world. However, substandard methods adopted for seed harvesting have significantly raised the oil production cost as well as the seed cost (Tabatabaei et al., 2019). Being tropical and subtropical drought tolerant crop, Sesame (*Sesamum indicum* L.) can be grown anywhere, including in deserts, low lying areas, and areas with low moisture content. Large-scale cultivation is thus feasible to enhance Sesamum oil extraction for commercial purposes. Once commercial oil production is feasible on large scale, up to 45% of seed yield could be converted to oil (Anilakumar et al., 2019). Due to the substantial sum of money that would otherwise be spent on importing petroleum, using the transesterification method, the Sesamum oil can be easily transformed into biodiesel, enabling India to achieve self-sufficiency in locomotive fuel sector. India might save millions of dollars with even a slight reduction in petroleum use. In the current context, Indian domestic energy requirement comes from a variety of sources, including nuclear energy (2.2%), traditional biomass (7.8%), and contemporary renewable energy sources (10.4%) while 79.5% energy requirement is derived from fossil fuel. Of them, the transportation sector requires about 50% of the total energy depending on the usage of fossil fuels. In the present scenario, reducing reliance on fossil fuels through the use of sustainable and renewable energy is necessary. Since the application of renewable energy in the field of transportation may reach up to 40% in 2030, the national objectives for Industrialized nations for 2018 include using bio-diesel as an alternative fuel (Tesfa et al., 2010; Verma et al., 2021).

Animal fats, discarded cooking oil, yellow grease, and different edible and non-edible vegetable oils are examples of renewable resources that can be utilized to manufacture the mono alkyl esters that are used to make biodiesel (Sivanathan and Thangavel, 2011). The oil in the lubricating system, the formation of sludge, insufficient spray atomization, poor combustion, and injector choking were some of the factors that Dr. Rudolf Diesel used to build the diesel engine in 1895 (Hossain et al., 2010). The physical and chemical properties of methyl ester in relation to its efficiency in compression ignition engines are evaluated as technical qualities of bio-diesel. Vegetable oils, which are quite viscous, are composed of triglycerides. Vegetable oil can be converted into molecules with lower molecular weights through transesterification or cracking (Shah et al., 2014; Datta et al., 2016). Sesame is preferable to utilize biodiesel since it is nontoxic, and has a high cetane number, lubricious and contains little Sulphur (Wakil et al., 2014). The amount of monounsaturated fatty acids in vegetable oil that can be used to make biodiesel is higher than that of polyunsaturated fatty acids (Ferdous et al., 2013). Therefore, selecting the appropriate raw material (feedstock) is essential to produce vegetable oil. Due to its high level of unsaturation, enhanced cold flow properties, and high level of oxidative stability brought on by antioxidants phytocompounds such as Sesamin, Sesamol, Sesamolin and vitamin E (tocopherol) present in Sesame seed oil is a good option (Pullen et al., 2014). Because to the inclusion of Sesamolin, Sesamin, and lignin's, the most important characteristic of SSO is its resistance to oxidation (Lee et al., 2008). Sesame biodiesel has a lower NOX emission rate than other biodiesels. Sesame seed oil have the environmental advantages and potential to ensure energy security which have made it more appealing for the manufacturing of biodiesel (Mujtaba et al., 2020).

The present investigation was intended to increase the essential aspects of the methyl ester transesterification of Sesame oil obtained from seeds of *Sesamum indicum* cultivars obtained from Bipuria region of Assam. While Sesame oil has not yet been extensively studied for the synthesis of biodiesel, it is important to optimize the critical quality components like molar ratio, reaction temperature, reaction duration, and catalyst concentration. Moreover, Sesame oil and its methyl ester's characteristics need to be compared with diesel standards.

Table 1. Comparison of the properties of Diesel (D100), Raw Sesame oil and Sesame methyl ester (B100)

Properties	Unit	Diesel (D100)	Raw Sesame oil	Sesame methyl ester (B100)
Calorific Value	kJ/kg	42900	-	31741.75
Dynamic Viscosity	MPa.S	1.352	-	5.252
Kinematic viscosity	mm^2/s	4.9	34.97	6.037
Cloud point	°C	-6	+9	+6
Pour point	°C	-16	+5	-3
Flash point	°C	42	230	112
Fire point	°C	68	275	158
Density	g/cm^3	0.823	-	0.870

2. Materials and methods

2.1. Collection of raw materials

Sesame seeds derived from *Sesamum indicum* L. (Lamiaceae) was collected from Bipuria region in Lakhimpur district of Assam. Sesame (*Sesamum indicum* L.) is a broadleaf summer crop which has characteristics aromatic smell, chime formed blossoms and inverse leaves. Sesame is an erect and annual herb that can attain at 4 - 7 feet high when grown under high moisture conditions.

2.2. Extraction of Sesame seeds

Sesame oil was extracted using a mechanical oil expeller (Figure 1). In this technique, the seeds were put in a hooper and crushed. The oil collected was filtered and oil cake was then collected from the expeller.

2.3. For the determination of %FFAs (Free fatty acids) using simple titration method

0.56 Grams of catalyst (KOH) was dissolved in 100 ml of distilled water. 50 ml of alcohol (methanol) was thoroughly mixed with 0.9 Grams of Sesame oil in a conical flask. The oil-to-alcohol mixture in the conical flask was then added three to four drops of phenolphthalein indicator. Titrate with 0.1N NaOH and shake the continuously until a light pink color appears which lasted for 25 seconds. For a better result, the experiment was carried out two to three times. Acid value and %FFA was determined by the following equation:

Acid value = $M \times N \times d \div \textit{Weight of oil sample}$

Where N is the normality of titration solution = 0.1 for NaOH

%FFA = $\textit{Acid value} \div 2 \times 100$

2.4. Determination of type of esterification

If the FFAs (Free fatty acids) value of sample oil is less than 4, then single stage is carried out. And if the FFA value is more than 4 than double stage (transesterification) is applied. As sesame oil has high FFA content therefore, it was carried out with two stage process.

2.4.1. Transesterification

Potassium-methoxide was produced by dissolving some KOH (about 1.5% w/v oil) and methanol. The oil was heated to 45 °C before being placed in a three-necked reactor and then mixture of potassium hydroxide and methanol was put into the reactor and kept at 60°C to 70°C for 30-55 minutes and then after some visible separation, it was transferred to separation funnel and kept for 3-6 hours for better separation. Glycerol was removed for significance as an industrial application.

2.5. Biodiesel preparation procedure

The following steps were followed during preparation of biodiesel from sesame oil seeds (Figure 1 and 2):

1. Measure the required amount of methanol or ethanol necessary for the transesterification reaction to prepare alcohol.
2. Prepare a solution of the chosen catalyst (sodium hydroxide or potassium hydroxide) by dissolving it in a small amount of alcohol; this catalyst solution is commonly known as the methoxide or ethoxide solution.
3. Add the catalyst solution to the alcohol and mix thoroughly, creating the methoxide or ethoxide solution.

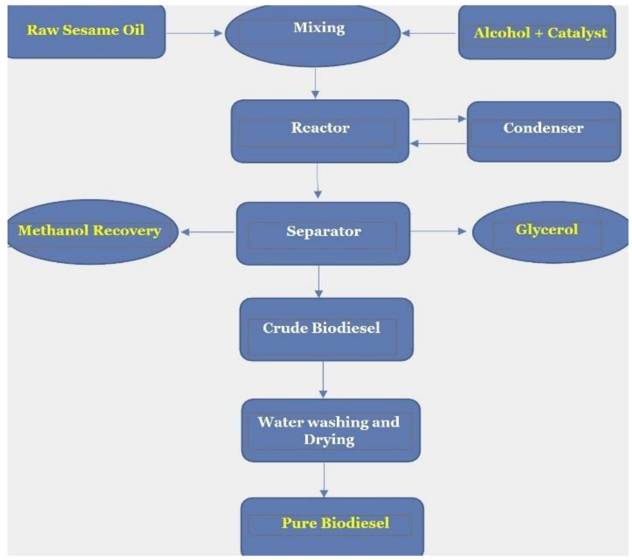

Figure 1. Flowchart showing preparation and production process of the biodiesel

Figure 2(A). Filtration of biodiesel; (B). B (100) Sesame methyl ester

4. Heat the vegetable oil or animal fat in the reaction vessel to a specific temperature, typically around 60-70°C.
5. Introduce the methoxide or ethoxide solution into the heated triglycerides, stirring the mixture vigorously to ensure comprehensive mixing.
6. Allow the transesterification reaction to progress for a variable period, usually taking 1-2 hours. During this time, the triglycerides react with the alcohol, resulting in the formation of biodiesel and glycerol.
7. After the reaction is complete, allow the mixture to settle. The denser glycerol settles at the bottom, while biodiesel floats on top. Enhance separation using a separation funnel or suitable equipment.
8. Wash the biodiesel to eliminate any remaining impurities, including catalyst residues and soap formed during the reaction. Rinse the biodiesel with water and allow it to separate.
9. Dry the washed biodiesel to remove any water content. Use drying agents or allow the biodiesel to sit and naturally evaporate water.
10. The resulting product is biodiesel, subject to further testing and refinement, if necessary, before its use as a fuel.

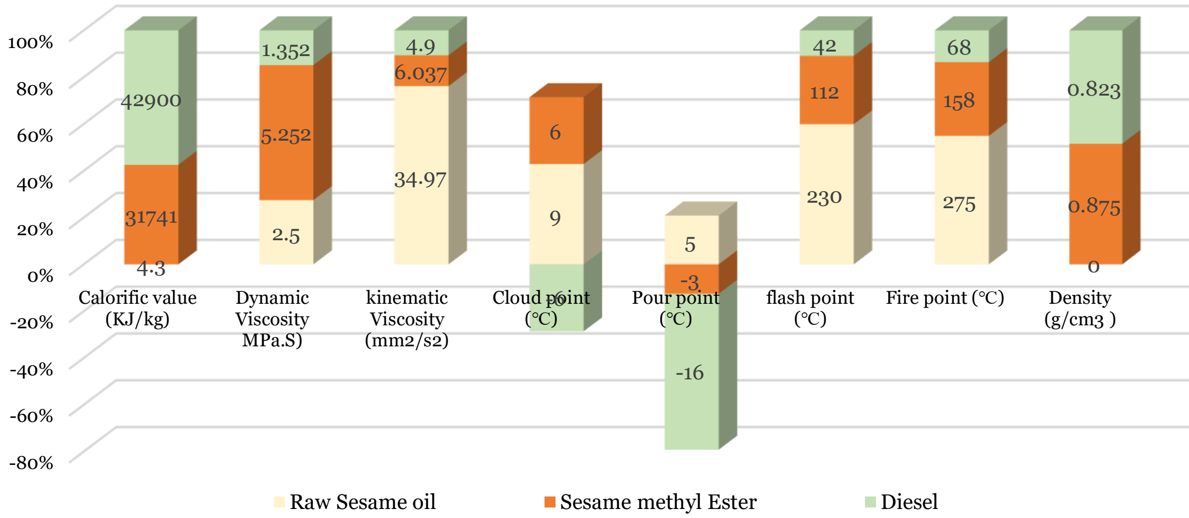

Figure 3. Comparison of properties

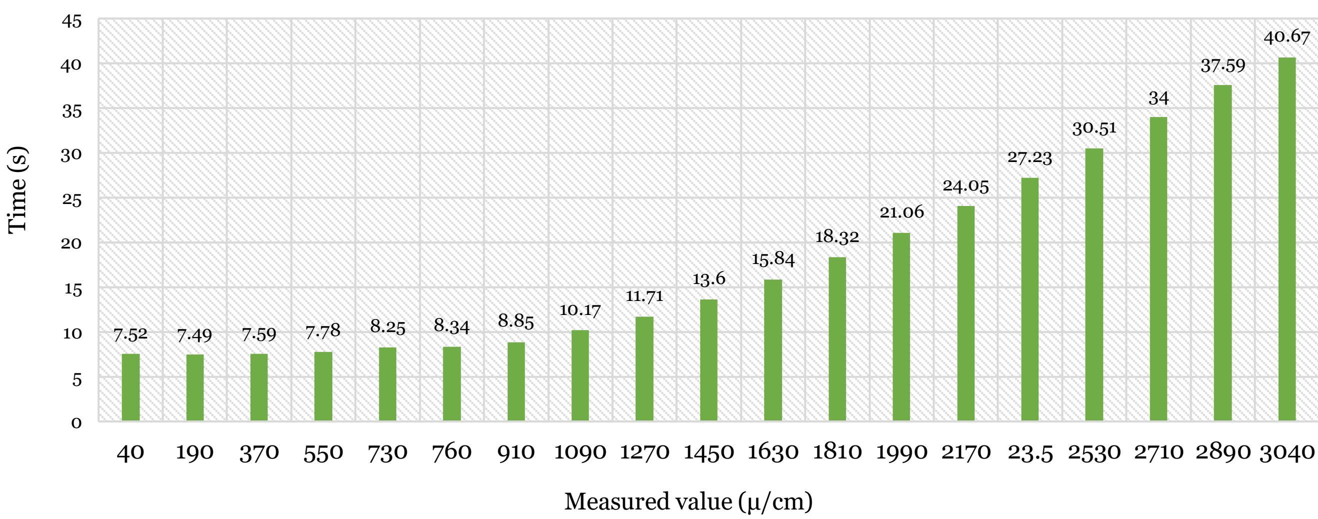

Figure 4. Oxidation Stability, Instrument : Rancimat 743

2.6. Heating and filtration

Sesame oil was heated to remove water content in it, and then it was filtered through filter paper to remove any suspended particles.

2.7. Transesterification

Transesterification is a chemical reaction characterized by the exchange of ester groups between an ester compound and an alcohol. This process involves the reaction of triglycerides, present in fats and oils, with an alcohol typically methanol or ethanol in the presence of a catalyst. The catalyst, potassium hydroxide, facilitates the production of biodiesel and glycerol. Through this transformation, triglycerides undergo conversion into fatty acid methyl or ethyl esters, commonly known as biodiesel, and glycerol emerges as a by-product.

The fundamental representation of the transesterification reaction is expressed as:
Triglyceride + Alcohol → Biodiesel + Glycerol.

2.8. Separation method

The separation technique capitalizes on the distinct density contrast between glycerol and biodiesel. Due to its higher density, glycerol spontaneously settles at the bottom of the flask. After a separation duration spanning 3-6 hours, the glycerol phase is meticulously extracted. This method provides a practical and uncomplicated approach to segregating glycerol from biodiesel, facilitating subsequent refinement and the utilization of the biodiesel product (Figure 1).

2.9. Water washing and drying

Water washing and drying play vital roles in the biodiesel production process. After the transesterification reaction converts triglycerides into biodiesel, the product often contains impurities like glycerol, soap, and excess methanol. Water washing was employed to eliminate the impurities by mixing water with biodiesel, extracting water-soluble impurities into the aqueous phase, and then separated the water. Subsequently, a drying process was carried out to eliminates any remaining water content to enhance biodiesel quality and to prevent issues such as increased oxidation and instability. These steps contribute to producing high-quality biodiesel, and a meticulous control is essential to meet required standards.

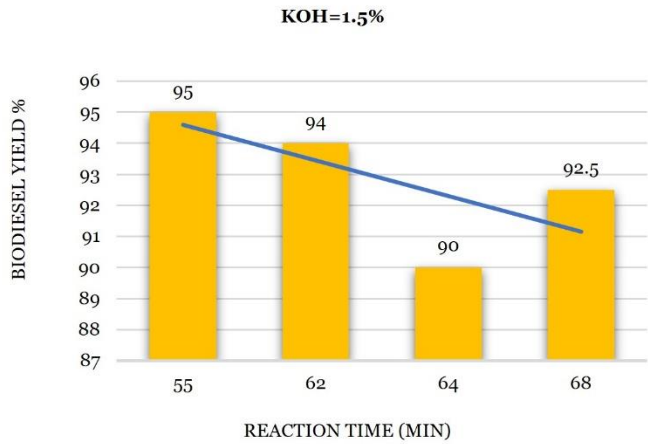

Figure 5. Biodiesel yield % for different reaction time at 1.5% KOH concentration, 3:1 oil to methanol ratio and varying temperature.

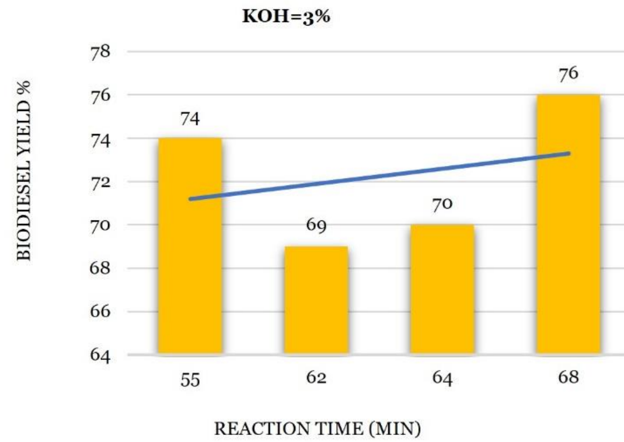

Figure 6. Biodiesel yield % for different reaction time at 3% KOH concentration, 4:1 oil to methanol ratio and varying temperature.

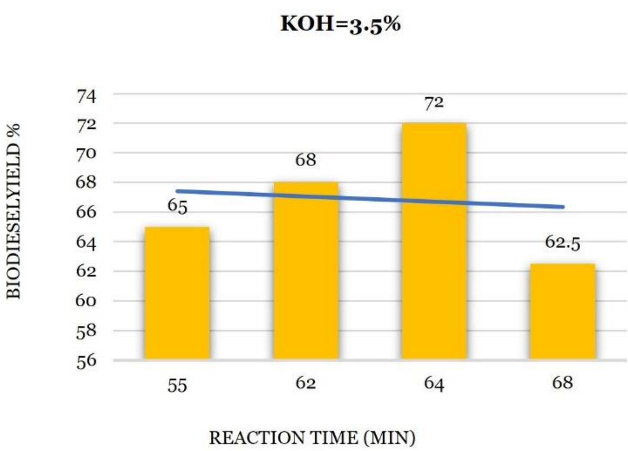

Figure 7. Biodiesel yield % for different reaction time at 3.5% KOH concentration, 4:1 oil to methanol ratio and varying temperature.

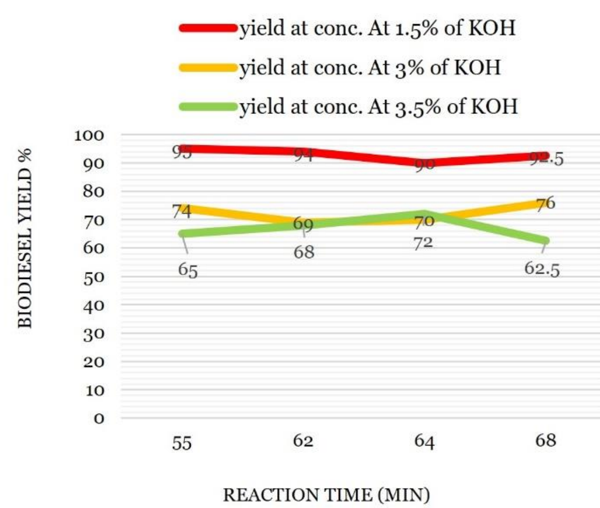

Figure 8. Summarized statistics of Figure 5, 6, and 7.

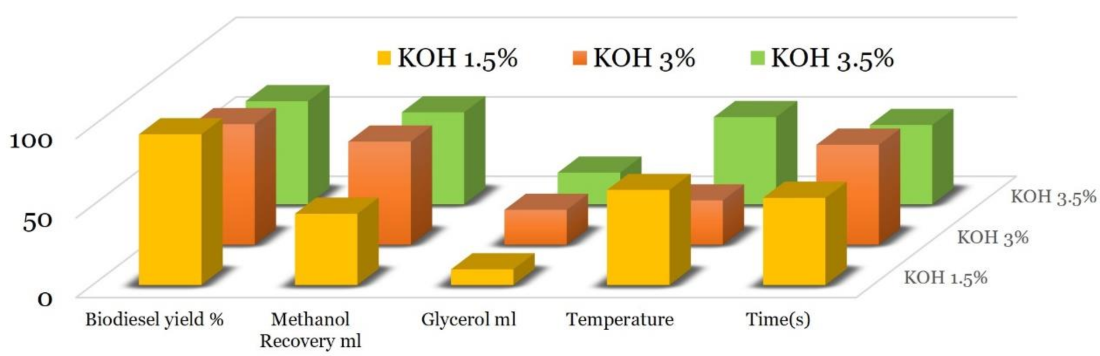

Figure 9. Comparison of quality data of optimization

2.10. Filtration of Biodiesel

To achieve a cleaner and more refined end product, the filtration process was carried out which requires passing biodiesel through a filter medium, effectively capturing and retaining impurities. This critical step is integral for meeting stringent quality standards, averting clogging in engine systems, and guaranteeing the optimal performance of biodiesel across diverse applications (Figure 2).

2.11. Oxidation stability test

Rancimat (743) instrument was used for study of oxidation stability (Figure 4). The tests involve frequently raising the temperature and adding oxygen or air to speed up the oxidation of the test fuel and hence the test's execution.

2.12 Optimization

A variety of variables, including the methanol to oil ratio, the concentration of KOH, the reaction time, and the reaction temperature, were considered in the optimization process for the transesterification reaction to produce biodiesel. Optimization was carried out by changing these parameters. The response was maintained in varying temperature and alcohols to oil ratios 4:1 and 3:1 was optimized, and the catalyst concentration was changed for each of the alcohol to oil ratios by 1.5%,3% and 3.5%. Various samples were collected, and the optimal parameter for the transesterification process was chosen as the combination of the various factors that produced the highest yield. In contrast, 3:1 alcohol to oil ratio, and 1.5% KOH concentration, and a 55-minute reaction time produced the best results.

3. Result and discussion

3.1. Comparison of properties

The experimental results indicate notable variations in the properties of diesel (D100), raw sesame oil, and sesame methyl ester (B100) which is presented in Table 1 and Figure 3. The calorific value of sesame methyl ester (B100) is observed to be 31741.75 kJ/kg, demonstrating its potential as a biodiesel fuel source. Additionally, the dynamic viscosity and kinematic viscosity of B100 are higher than those of diesel (D100), highlighting differences in fluid characteristics. The cloud point and pour point of B100 are also within acceptable ranges for biodiesel application, and its flash and fire points suggest improved safety compared to raw sesame oil. The density of B100 is slightly higher than diesel, indicating a denser fuel.

3.2. Oxidation stability

Despite possessing 85% of the fats are unsaturated, Sesame oil has demonstrated high oxidation stability (Figure 4). Sesame oil is more resistant to oxidative rancidity than other vegetable oils (Soybean, Sunflower, Coconut, and Olive) due to the presence of unique tocopherols and lignans, such as p-hydroxyphenyl-propane. The Rancimat instrument utilized in the study reveals the oxidation stability of sesame oil, attributed to the presence of unique tocopherols and lignans.

3.3. Optimization of biodiesel production and comparison of quality data of optimization

The biodiesel yield percentage is presented in Figure 5, 6, 7, 8 and the quality data biodiesel yield, methanol recovery and glycerol percentage are presented in Figure 9 for reaction times of 55, 62, 64 and 68 minutes for a 3:1 oil-to-methanol ratio at a varying temperature. The maximum yield obtained was 95% at 1.5% KOH concentration at 55 minutes of reaction time; and at 3% KOH concentration, the maximum yield obtained was 76% at 68 minutes of reaction time; and at 3.5% KOH concentration, the maximum yield obtained was 72% at 64 minutes of reaction time. Therefore, based on the above result, it can be concluded that at 1.5% KOH concentration and 55 minutes of reaction time, the highest biodiesel production (95%) was obtained for a 3:1 ratio of oil to methanol at 55-65 °C temperature. The byproduct of transesterification can be utilized for further experimentation of pure glycerin, methanol distillation, and it can be used as lubricants for vehicles. The optimization process for transesterification considered various parameters, and the resulting biodiesel yield was found to be optimal at a 3:1 oil-to-methanol ratio, 1.5% KOH concentration, and 55 minutes of reaction time. This aligns with the findings of recent studies (Alok et al., 2017; Jennifer et al., 2018) which emphasized the importance of careful parameter optimization for efficient biodiesel production. Additionally, the byproducts of transesterification, such as glycerol and methanol, can be repurposed for further applications, aligning with the sustainability aspect of biodiesel production (Ferdous et al., 2013; Wang et al., 2011).

4. Conclusion

Sesame oil was used as a feedstock for biodiesel production using two-stage transesterification processes. Optimization process was carried out by varying the process parameters such as the molar ratio, catalyst concentration, type of catalyst, steering speed, temperature, time period, etc. Oxidation stability result of Sesame biodiesel shows more resistant to oxidation compare to other biodiesel fuel due to presence of antioxidant. All the physio-chemical characteristics were tested as per ASTM Standard and observed that they were found to be closer to diesel fuel. Biodiesel from sesame oil could be used as a supplementary fuel for compression ignition engines. However, to achieve better optimization for maximum biodiesel yielding, further studies are needed to be done on the fatty acid composition, catalyst concentration, reaction time using of dual catalysts.

Acknowledgement
The authors are thankful to the Director, Northeastern Regional Institute of Science and Technology (NERIST), Nirjuli for lab and logistic support.

Authors' contributions
The second author (PL) designed the experiment, finalized the manuscript. First and third authors conducted the experiment and generated data, developed draft manuscript.

Conflict of interest
Authors have no conflict of interest

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