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# "Sustainable Biodiesel Generation from Althaea officinalis: Non-Edible Oil as a Green Energy Resource"

**Short Running Title: - Biodiesel production from Althaea officinalis seeds oil**

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**KEYWORDS**

Althaea officinalis, non-edible seeds oil and biodiesel.

**ABSTRACT:**

**Introduction**: This research examined the production of biodiesel from Althaea officinalis seeds oil (AOSO), investigating its potential as a renewable and sustainable energy source.

**Objectives:** This research emphasized on the benefits of employing non-edible oils and alleviating food security issues as well as utilizing land that may otherwise remain unproductive.

**Methodology:** This study included the extraction of oil from Althaea officinalis seeds by using soxhlet apparatus and the subsequent base catalyzed transesterification process to generate biodiesel. The produced biodiesel (AOBD) was analyzed for its fuel characteristics, including density, viscosity, flash point etc.

**Results:** The oil content for the Althaea officinalis seeds was found to be 25.84 %. The FFAs value for the AOSO was reduced to 0.5% after treatment. The calorific value and biodiesel yield for the AOBD were found to be 36.27 mJ/Kg and 94.56% respectively.

**Conclusion:** The results demonstrated that AOSO is a feasible feedstock for biodiesel production, yielding biodiesel with acceptable fuel characteristics that satisfy industrial requirements. In summary, this study advocates for the potential of AOSO as a sustainable source of biodiesel, presenting a promising path for increasing the diversity and sustainability of renewable energy options.

## 1. Introduction

The growing worries regarding the exhaustion of fossil fuels and the environmental risks associated with their ongoing utilization have led to the investigation of alternative and renewable energy options. Biodiesel, a renewable energy alternative, is generated from a variety of oils, including those derived from plants, waste cooking oil, animal fats, and algae through the transesterification process.[1] Commonly referred to as Eco-diesel, it is acknowledged for its lower environmental footprint when compared to conventional fossil fuels.[2] Choosing a suitable feedstock for biodiesel necessitates an assessment of several essential factors, including local source availability, economic viability, accessibility, and technical feasibility.[3] In the pursuit of sustainable biodiesel production, non-edible seed oils are particularly attractive because they do not compete with food resources and can be cultivated on waste lands. One particularly promising feedstock for biodiesel is the seed oil extracted from Althaea officinalis.

Althaea officinalis, often called the marshmallow plant, belongs to the Malvaceae family. Various parts of the plant, including seeds, leaves, roots, and flowers, are primarily utilized in Unani Medicine. It is recognized for multiple pharmacological benefits such as diuretic,

lithotriptic, deobstruent and anti-inflammatory properties, which make it effective for treating kidney stones, jaundice, menstrual disorders, hepatitis, headaches, and more.[4] The graphical abstract for the given research work is shown in the figure 1. To the best of our knowledge, using AOSO signifies a novel approach to biodiesel production. Biodiesel can be produced through various methods; among them, transesterification is the most commonly employed.[5,6] The transesterification process typically involves the reaction of oils with methanol in the presence of a catalyst, resulting in biodiesel and glycerol as by-products.[7] Previous studies demonstrated that alkali-catalyzed transesterification occurs at a much quicker rate compared to acid-catalyzed processes.[8] The result of this process is the formation of FAMEs derived from seed oils, with glycerin as a secondary product. FAMEs, commonly referred to as biodiesel, exhibit qualities similar to petroleum-based diesel fuels.[9]

## 2. Objectives

This study aims to investigated the production of biodiesel from novel source AOSO, evaluating its potential as a sustainable energy source by employing base-catalyzed transesterification. NaOH serves as the base catalyst in this process. By examining the physicochemical characteristics of AOSO along with the fuel properties of the biodiesel, this research aspires to provide valuable insights into the broader utilization of AOSO in renewable energy production, presenting a promising pathway towards the advancement of greener fuels in the near future.

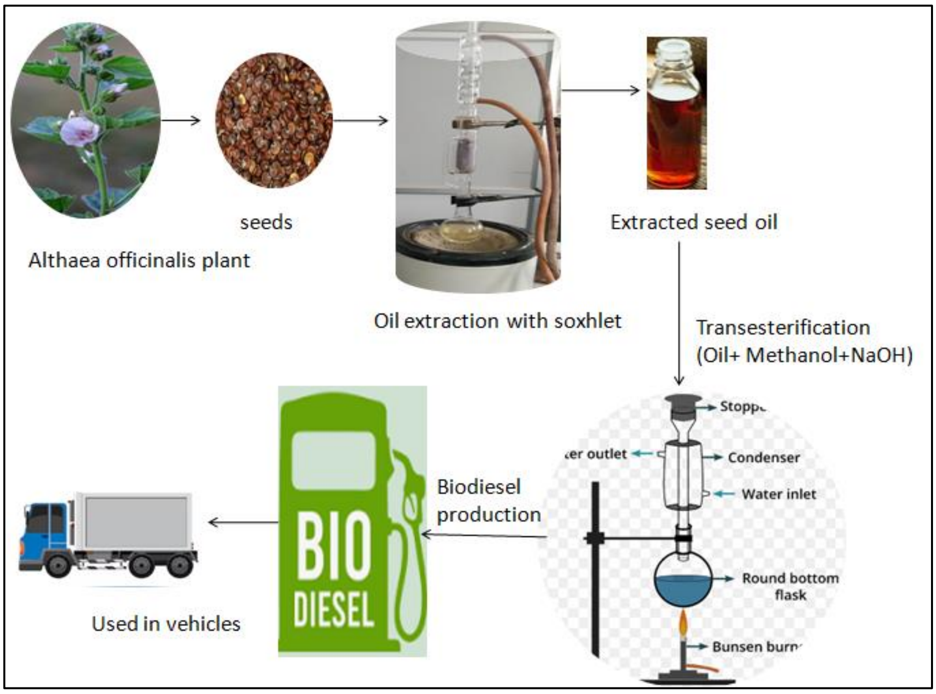

**Figure 1: Graphical abstract of biodiesel production from AOSO**

## 3. Methods:

The approach taken to study the production of biodiesel from AOSO includes several essential stages, namely feedstock preparation, biodiesel synthesis, and fuel properties assessment and comparison with the fuel standards.

### Feedstock Preparation:

**Collection and Preparation**: Fresh Althaea officinalis seeds were obtained from different locations in Rajasthan. The seeds were cleaned, dried, and grinded into a fine powder to enhance oil extraction.

**Oil Extraction**: Oil was extracted from the powdered seeds utilizing a n-hexane solvent through soxhlet extraction method. The extracted oil was filtered to eliminate impurities and particulates.

**Biodiesel Production:**

**Transesterification Process:** Biodiesel was synthesized through the base catalyzed transesterification process, in which AOSO is reacted with methanol in the presence of a sodium hydroxide used as base catalyst. This reaction occurred under regulated temperature and pressure conditions.

**Separation and Purification:** Following the reaction, biodiesel and glycerol were separated using centrifugation. The biodiesel was further purified through washing with water to eliminate any residual catalysts and impurities.

**Physicochemical properties Analysis**- AOSO was analyzed for the various parameters like SV, IV, Moisture content, Refractive index etc.

**Characterization of AOSO and AOBD:**

**Fourier transforms Infrared spectroscopy (FT-IR) analysis -**The analysis of functional groups in AOSO was conducted using a FT-IR instrument from Thermo Fischer Scientific.

**Gas chromatogram Mass spectroscopy (GC-MS) analysis-** The fatty acid composition in AOBD was determined by utilizing a GC-MS spectrophotometer from Thermo Scientific TSQ 8000.

**Fuel Properties**: The AOBD was analyzed for fuel properties, such as density, viscosity, flash point, CN, and AV etc. These properties were compared to those of conventional biodiesel such as ASTM-D6751 and EN-14214 standards to evaluate its suitability as a fuel.[10,11]

## 4. Result and Discussion

### Physicochemical properties of AOSO

The physicochemical properties of AOSO were evaluated using standard methods established by the AOCS.[12] Following purification, the AOSO displayed a light brown color and remained in a liquid state at ambient temperature. The properties of AOSO are summarized in table -I. The oil yield for AOSO was assessed at 25.84%, which is higher to the oil yields of Hibiscus sabdariffa (15.34%) and Hibiscus altissima (16.01%).[13] The moisture content of AOSO was measured at 1.7%, which exceeds the reported value of 1.2% for C. inophyllum seeds.[14] Reports indicated that extracting seeds with lower moisture content generally leads to higher oil yields. This aligns with the findings of Orhevba BA. et al. regarding Azadirachta seeds.[15] Elevated moisture levels initiate oxidation, which can diminish the oil's shelf life. The density of AOSO at 25°C was measured at 887 $Kg/m^3$, indicating that the oil is less dense than water due to the lack of heavy constituents. The physical characteristics of AOSO are akin to those of other biodiesels, and its environmentally friendly nature has been validated. The IV of the AOSO was found to be 127 g $I_2$/100 g oil determined by the Wij's method. The IV of the oil can be used to estimate its oxidation or drying potential, which correlates with the quantity of unsaturated fatty acids present in the oil. The oxidative stability of fats or vegetable oils can be assessed through their IV. The maximum permitted level according to EN 14214 (120 g $I_2$/100 g oil) the IV of AOSO slightly exceeds the standard value. The elevated iodine content may be attributed to the high levels of unsaturated fatty acids. With an IV above 100, AOSO could be classified as a drying oil.[16] SV indicates the average molecular weight of triglycerides. When lipids interact with an alkali, they undergo saponification, resulting in the formation of glycerol and fatty acid salts.[17,18] With an SV of 205 mg KOH/g, AOSO is comparable to Citrullus lanatus (205.4 mg KOH/g) and slightly higher than Jatropha seed oil (193.55 mg KOH/g) as reported by Akbar E. et al.[19, 20] Oils with a high SV typically contain triglycerides that are desirable for producing liquid soaps and shampoos. The elevated SV of the oil suggests a diverse fatty acid composition, which can complicate the separation of products and may account for the low yield in biodiesel production. In the primarily investigation, the extracted AOSO showed a much higher FFAs content of 1.5%. To address this, the AOSO underwent acid-catalyzed esterification using methanol treated with sulfuric acid. This process significantly reduced the FFAs content to 0.5%, making the oil suitable for subsequent base-catalyzed transesterification. During esterification, the sulfuric acid acted as a catalyst, converting the FFAs into esters through their reaction with acidified methanol.[21]

**Table -I: Physicochemical properties of AOSO**

| **Parameters** | **AOSO** |
|---|---|
| Oil (%) | 25.84 |
| Moisture content (%) | 1.7 |
| Saponification value (mgKOH/g oil) | 205 |
| Iodine value ($gI_2$/ 100g oil) | 127 |
| Refractive index | 1.476 |
| Relative density($Kg/m^3$) | 887 |
| FFAs (%) | 0.5 |
| Unsaponifiable matter (%) | 2.1 |

**FT-IR analysis of AOSO:**

The FT-IR spectrum for AOSO is presented in figure 2. The interpretation of the FT-IR spectrum for AOSO is as follows:

- 3357 $cm^{-1}$ - indicated $sp^2$ C-H stretching vibrations
- 2890 $cm^{-1}$ - corresponded to $sp^3$ C-H stretching vibrations
- 1780 $cm^{-1}$ – indicated the presence of the Ester functional group
- 1400 $cm^{-1}$: represented asymmetric stretching vibrations of $CH_3$
- 1070 $cm^{-1}$: signified stretching of $O-CH_3$
- 1780 $cm^{-1}$, 1400 $cm^{-1}$, 725 $cm^{-1}$; related to C=O stretching, asymmetric stretches of $-CH_3$ and $CH_2$ bending.
- 675-1000 $cm^{-1}$; indicated =C-H stretching
- 2800-3000 $cm^{-1}$; denoted C-H stretching bond

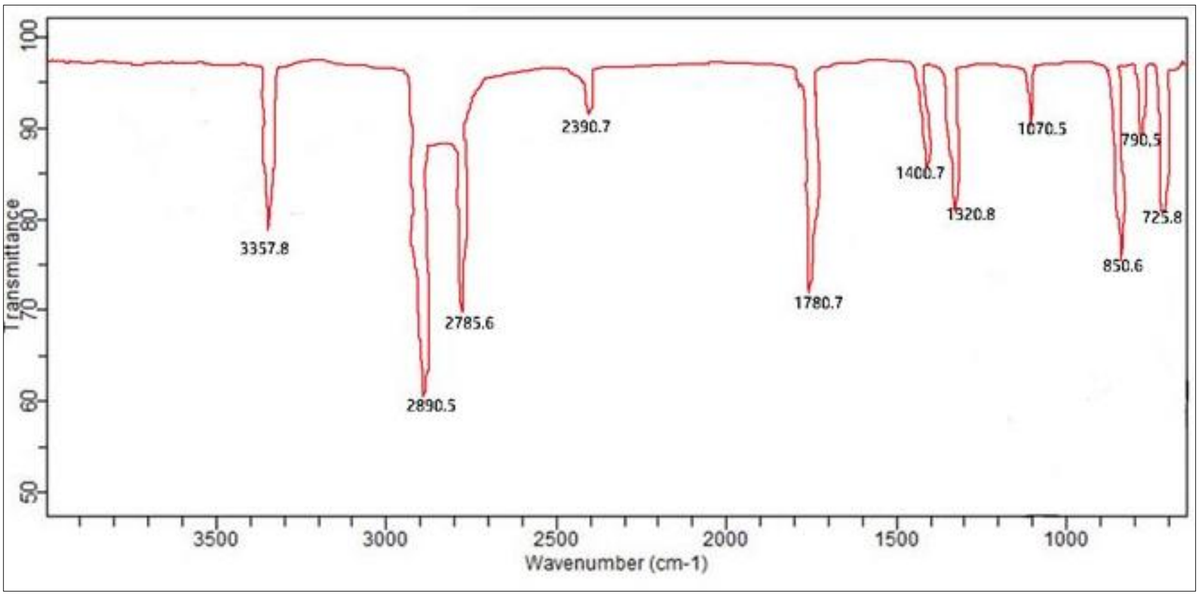

**Figure 2: FT-IR analysis of AOSO**

**GC-MS analysis of AOBD:**

The AOBD underwent a GC-MS assessment that determined its FAMEs composition. The components of FAMEs were identified by analyzing the gas chromatogram (GC) peaks and referencing a mass spectrometry (MS) database. Figure 3 illustrated the GC-MS spectral analysis of AOBD. Table- II and figure 4 displays the percentage of various fatty acids found in the AOBD feedstocks used for biodiesel production. These fatty acids included palmitic (9.5%), stearic (5.6%), oleic (32.5%), linoleic (40.2%), linolenic (6.5%), arachidic (3.1%), and others (2.4%). Examination of the AOBD in table II indicated that oleic acid and linoleic acid were the predominant fatty acids. Linoleic acid (40%) and oleic acid (32.5%) emerged as the most abundant fatty acids in AOBD. The reported oleic acid percentage for AOBD is notably similar to that of biodiesel derived from Mahua (36.4%) as cited by Gopinath A. et al. and from crude Calophyllum inophyllum oil (38.25%) according to Aparamarta HW. et al.[22, 23]

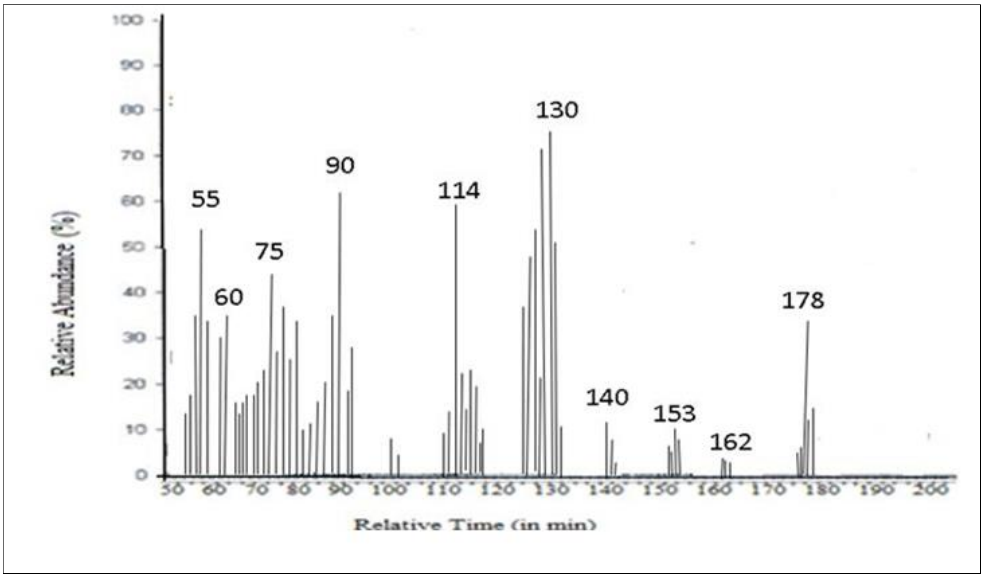

**Figure 3: GC-MS spectrum of AOBD**

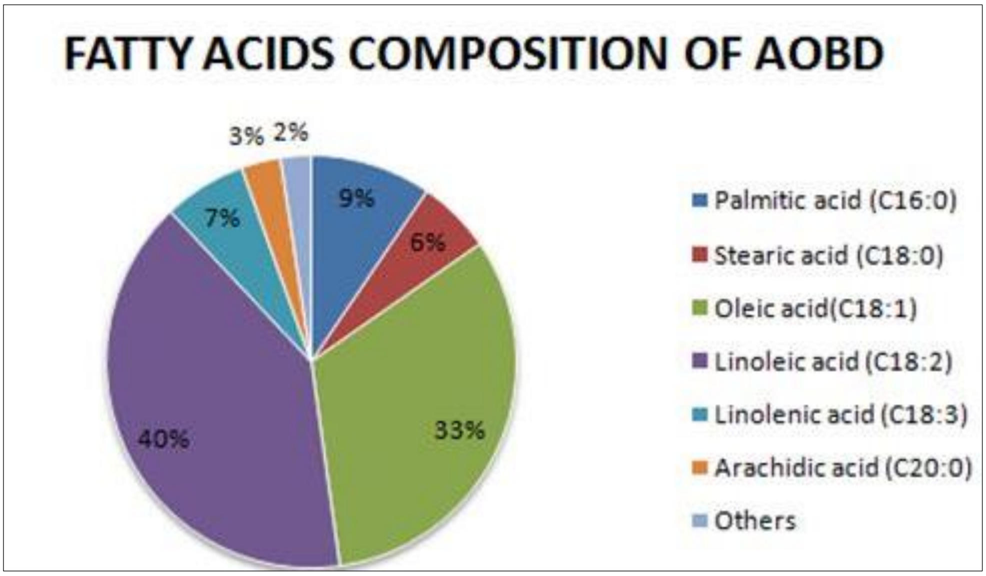

**Figure 4: Fatty acids composition of AOBD**

**Table II: Fatty acids composition of AOBD (uncorrected weight percent) by GC-MS.**

| **Fatty acids (%)** | **AOBD** |
|---|---|
| Palmitic acid ($C_{16:0}$) | 9.5 |
| Stearic acid ($C_{18:0}$) | 5.8 |
| Oleic acid($C_{18:1}$) | 32.5 |
| Linoleic acid ($C_{18:2}$) | 40.2 |
| Linolenic acid ($C_{18:3}$) | 6.5 |
| Arachidic acid ($C_{20:0}$) | 3.1 |
| Others | 2.4 |

### Fuel characteristics of AOBD

The fuel characteristics of AOBD are displayed in Table III. In comparison to Citrus sinensis (0.01 wt.%) and Thespesia populnea oil (0.015 wt.%), AOBD has a lower ash concentration of 0.007 wt.%.[24,25] The low ash content indicated that the oil contains minimal trace metals, which could lead to oxidation processes that result in rancidity, increased acidity, and other storage defects. The high ash content is detrimental as it hampers bio-oil production while raising the concentrations of biochar and gas-phase compounds.[26] According to the EN 14214:2003 standard, the maximum allowable carbon residue value is 0.3%. A high carbon residue value suggests the presence of significant amounts of FFAs, glycerides, polyunsaturated fatty acid methyl esters, and synthetic polymers. Therefore, exceeding this limit could result in severe engine fouling. The carbon residue value of AOBD (0.25%) is higher than that of Jatropha (0.2 wt. %), Pongamia (0.05 wt. %), and Moringa (0.05 wt. %) oils.[27-29] The ash content for AOBD was reported to be 0.028 (wt. %). The pour point represents the lowest temperature, expressed in multiples of 3°C, at which oil is observed to flow when cooled and tested under specific conditions.[30] AOBD exhibited a pour point of -4°C, which is lower than that of Cassia auriculata (6.7°C) and Mahua (13°C).[31,32] This value is close to the -3°C noted for Citrullus lanatus.[33] This indicated that AOBD is suitable as a raw material for producing lubricating oils in cold environments. The cloud point refers to the temperature where a cloud or haze first appears during controlled cooling. AOBD has a cloud point of -4.5°C, which is lower than the 10–11°C range observed for Jatropha oil.[34] Due to its high level of unsaturated fatty acids, AOBD is comparable to other non-edible seed oils for biodiesel production in colder climates. AOBD has a flash point of 155°C, with results for linseed oil biodiesel falling between the ranges reported by Sahin S. et al. (148°C) and Ullah F. et al. (177°C). [35,36] The recorded fire point for AOBD is 158°C. In this study, AOBD's PV measured 4.46 mEq/kg of oil. The PV is utilized for assessing oil quality and stability by determining the level of rancidity caused by storage, heating, or exposure to air. Oils are considered rancid when their PV is between 20 and 40 mEq/kg.[37] Oils with high PV values are indicative of lower quality. An increase in PV correlates with rising viscosity, cetane number, and corrosiveness of seed oil and biodiesel.[38,39] Thus, using biodiesel with a higher PV may cause corrosion in the fuel system and engine. Since AOBD’s PV is significantly below the acceptable range for biodiesel, it can be stored for extended periods without risk of deterioration. The AV for the AOBD was found to be 1.8 mg KOH/g oil. The KOH content in edible oils should not exceed 10 mg/g; however, chemical re-esterification or physical refining would be necessary to reduce the acidity of the oil.[40] This acidity is sufficiently low and complies with the standards for oils intended for industrial use. Elevated acid levels can lead to corrosion and blockages, causing challenges in engine performance. The acid concentration indicated here is acceptable and could be utilized in engines without leading to corrosion damage.[41] The CN observed for the AOBD was 48, which is slightly higher

the biodiesel standard limits (above 47). The CV determined for the AOBD was 36.27 mJ/kg, with a corresponding biodiesel yield of 94.56%. These values are comparatively similar to those reported for biodiesel derived from Argemone mexicana seed oil, which exhibited a higher CV of 38.7mJ/kg and biodiesel yield of 95.7%.[42] Similar results for the biodiesel yield (94.6%) was reported for the Ricinus communis by Jamil M.A.[43] The CV represents the amount of heat energy released during the combustion of a fuel and is a critical parameter in evaluating fuel performance. A higher CV is generally desirable, as it indicates greater energy release, thereby enhancing engine efficiency.[44] In the present study, the CV of AOBD falls within the acceptable range defined by ASTM standards, although it is slightly lower than that of conventional petro-diesel. This observation aligns with the general trend observed in biofuels, which typically exhibit lower CV due to their elevated oxygen content. Nonetheless, the CV of AOBD supports its viability as a supplementary or alternative fuel to petroleum diesel.

**Table III: Fuel properties of AOBD compared with ASTM-D6751 and EN 14214 biodiesel standards.**

| **Fuel properties** | **AOBD** | **ASTM D6751[10]** | **EN 14214[11]** |
|---|---|---|---|
| Cloud point ($^0$C) | -4.5 | −3 to −12 | ------ |
| Pour point ($^0$C) | -4 | −15 to −16 | ----- |
| Density (Kg/m$^3$) | 870 | 880 | 860–900 |
| Flash point ($^0$C) | 155$^0$C | 130$^0$C | Min. 101 |
| Fire point ($^0$C) | 158$^0$C | ----- | ----- |
| Kinematic Viscosity (mm$^2$/s) | 3.8 | 1.9–6.0 | ------- |
| Viscosity (mm$^2$/s) | 3.7 | ------ | 3.5-5.0 |
| Specific gravity (g/cm$^3$) | 0.756 | ------ | ------ |
| Ash content (wt %) | 0.028 | 0.05max | Max. 0.02 |
| Carbon residue (wt %) | 0.25 | 0.05 max | |
| Acid value (mgKOH/g) | 0.5 | 0.50 max | Max. 0.50 |
| Peroxide value (mEq/kg) | 4.46 | ------ | ----- |
| Cetane no. | 48 | 47 min | Min. 51.0 |
| Calorific value (mJ/Kg) | 36.27 | ----- | Min 35.0 |
| Biodiesel yield (%) | 94.56 | ----- | ------ |

## Conclusion:-

The present study investigates the potential of AOSO, a non-edible oilseed, as a viable feedstock for biodiesel production. AOSO reported to have satisfactory oil content (25.84%). The effective oil extraction and transesterification of AOSO to exceptionally high biodiesel yield of 94.56% highlighted its appropriateness for biodiesel production, presenting a promising answer to the challenges linked to using non-edible oils. Utilizing the non-edible seeds oil AOSO

aids in alleviating food security issues related to edible oil feed-stocks and effectively uses land that may otherwise remain unproductive. It offers a sustainable and cost-effective alternative that can expand the range of biodiesel sources while contributing to cleaner energy initiatives. Future research could investigate the scalability of this method, its long-term effectiveness in diverse applications, and the possibilities for incorporating Althaea officinalis cultivation into broader agricultural systems.

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**LIST OF ABBREVIATIONS:**

| |
|---|
| AV-ACID VALUE |
| AOBD- ALTHAEA OFFICINALIS BIODIESEL |
| AOCS- AMERICAN OIL CHEMIST'S SOCIETY |
| AOSO- ALTHAEA OFFICINALIS SEED OIL |
| ASTM- AMERICAN SOCIETY FOR TESTING MATERIAL |
| CV- CALORIFIC VALUE |
| CN- CETANE NUMBER |
| EN- EUROPEAN NORMS |
| FAMEs - FATTY ACIDS METHYL ESTERS |
| FFAs- FREE FATTY ACIDS |
| FT- IR - FOURIER TRANSFORMS INFRARED SPECTROSCOPY |
| GC- MS- GAS CHROMATOGRAPHY MASS SPECTROMETRY |
| IV- IODINE VALUE |
| PV- PEROXIDE VALUE |
| SV- SAPONIFICATION VALUE |

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