



**International Journal of Biology, Pharmacy
and Allied Sciences (IJBPAS)**

'A Bridge Between Laboratory and Reader'

www.ijbpas.com

**A NOVEL RESEARCH ON COMPARISON OF FUEL PROPERTIES OF
CASSIA ANGUSTIFOLIA -BASED BIODIESEL SYNTHESIZED WITH
AND WITHOUT EGGSHELLS NANOCATALYST**

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Received 10th Dec. 2024; Revised 25th Dec. 2024; Accepted 28th Jan. 2025; Available online 15th March 2025

<https://doi.org/10.31032/IJBPAS/2025/14.3.1034>

ABSTRACT

Biodiesel represents a renewable and sustainable substitute for fossil fuels, providing both environmental and economic advantages. This research examined the production and fuel characteristics of biodiesel sourced from *Cassia angustifolia* seed oil (CASO), comparing two production techniques: one that employed an eggshells nanocatalyst (ESN) and another that did not use a catalyst. To the best of our knowledge, for the first time, CASO was analyzed as an alternative feedstock for biodiesel production by utilizing ESN. Eggshells, which are a plentiful waste product, were transformed into CaO nanocatalyst through calcination in muffle furnace, offering an eco-friendly and economically viable catalyst. CASO oil contained obtained by soxhlet apparatus is 25.08 %. *Cassia angustifolia* biodiesel (CABD)_c yields were markedly increased in the process utilizing the ESN due to enhanced reaction efficiency. The fuel characteristics, including density, viscosity, cetane number, flash point, pour point, and calorific value, were assessed in accordance with ASTM D6751 and EN 14214 standards.

The outcomes revealed that (CABD)_c produced with the ESN displayed superior fuel attributes, such as decreased viscosity (3.0mm²/s) and acid value (0.30 mgKOH/g) as well as elevated calorific value (45.895

mJ/Kg) and biodiesel (85.90%), in comparison to the CABD. The recorded flash point and fire point for (CABD)_C were higher 135°C and 140°C respectively. This comparative analysis highlighted the potential of using ESN in (CABD)_C synthesis, fostering waste utilization and sustainable energy alternatives.

Keywords: Biodiesel, *Cassia angustifolia*, Eggshell nanocatalyst and Transesterification

INTRODUCTION:

The increasing exhaustion of fossil fuel reserves, coupled with the environmental issues they cause such as greenhouse gas emissions and air quality deterioration has heightened global attention towards renewable energy options. The primary environmental challenge facing the world today is global climate change, which is exacerbated by the rising levels of CO₂ emissions linked to increased economic growth and energy consumption [1]. Advancements in renewable technology could improve the efficiency of current fossil fuel usage, thereby decreasing energy consumption in manufacturing processes [2]. Innovations in renewable energy technologies have the potential to deliver clean energy solutions to the market and positively influence energy portfolios [3, 4]. This trend emphasizes the critical need for sustainable and renewable energy solutions, such as biodiesel. The benefits of biodiesel as an energy source include its renewability, non-toxic nature, ease of mobility, and its environmentally friendly characteristics [5]. Biodiesel, derived from fats and oils, boasts

lower hydrocarbon content, decreased CO emissions, reduced smoke and particulate matter, as well as providing better engine lubrication and a higher cetane number in comparison to petro-diesel [6, 7]. The use of edible oils for biodiesel production has sparked concerns regarding food security and competition for agricultural land. To mitigate this issue, in the present research work non-edible seeds oil from *Cassia angustifolia* (commonly known as Senna or Cassia) was used for the biodiesel production. This plant, part of the Fabaceae family, flourishes in arid and semi-arid zones, including Rajasthan, and is recognized for its medicinal benefits such as antimicrobial, antifungal, antiviral, antiparasitic, insecticidal, and antioxidant properties. Furthermore, this plant exhibited anti-diabetic, anti-cancer, hepatoprotective, and hypolipidemic effects [8]. Growing *Cassia angustifolia* in Rajasthan provides a dual advantage: it utilizes marginal lands that aren't suitable for food crops and offers a sustainable income source for local communities. The seeds of *Cassia angustifolia*, found an oil content of 25.08%,

serve as a promising feedstock for biodiesel production. Notably, the non-edible nature of this oil ensured there is no competition with food crops. Here the seeds oil obtained from *Cassia angustifolia* seeds oil is represented as CASO. Biodiesel produced from CASO with catalyst represented as (CABD)_C and without catalyst as CABD both were subjected to comparative analysis. CABD and (CABD)_C produced from CASO were also assessed against ASTM-D6751 and EN-14214 [9, 10]. An innovative method for producing biodiesels from CASO has incorporated the use of eggshells nanocatalyst (ESN). Eggshells, primarily made of CaCO₃, are an abundant, low-cost waste resource that can be converted into highly effective nanocatalyst for transesterification processes. The most explored area concerning the potential application of eggshell waste is in catalysis. When properly processed, it can facilitate the conversion of waste oils into biodiesel [11]. The morphological and physicochemical characteristics of nanoscale catalysts provide considerable benefits, such as a large surface area, favorable area-volume ratio, increased reactivity, photocatalytic properties, and reduced cytotoxicity, all of which enhance catalytic performance during transesterification reactions [12]. Employing ESN not only boosts the efficiency of

biodiesel production but also aligns with the concepts of waste valorization and a circular economy.

This research evaluated the viability of generating (CABD)_C by utilizing ESN. The study concentrated on:

1. Extracting and analyzing CASO.
2. Synthesizing and analyzing ESN.
3. Assessing the fuel characteristics of the biodiesel produced from CASO, and comparing these with the ASTM-D6751 and EN14214 specifications.

By merging sustainable raw materials with innovative catalytic methods, this research aimed to enhance biodiesel production techniques, foster environmental sustainability, and aided in rural development in less affluent areas like Rajasthan.

MATERIALS AND METHODS

The approach to optimizing biodiesel production from non-edible CASO in Rajasthan included the following main steps:

1. Seed Collection and Preparation

- Seed Collection: *Cassia angustifolia* seeds were collected from various arid and semi-arid locations in Rajasthan.
- Seed Drying: The seeds harvested were thoroughly cleaned and dried in a shaded area to minimize moisture content, ensuring effective oil extraction.

• Grinding: The dehydrated seeds were crushed or grounded into a fine powder to enhance the surface area for improved oil extraction efficiency.

2. Oil Extraction

• Solvent Extraction Method: CASO was extracted using a solvent extraction technique, utilizing n-hexane, in a Soxhlet apparatus, which maximizes the oil yield from the seeds.

• Oil Purification: The extracted CASO was purified to remove impurities, including any remaining solvents and particulate matter, rendering it suitable for biodiesel production.

3. Analysis of Physicochemical Properties of CASO

The physicochemical properties of CASO, including saponification value, iodine value etc. were assessed in accordance with the standard protocols set by the American Oil Chemists' Society (AOCS) [13].

4. Biodiesel Production (Transesterification)

• Preparation of ESN

Eggshells were collected from local poultry shops, thoroughly rinsed with water to eliminate contaminants, and subsequently dried in an oven to remove moisture. After drying, the eggshells were ground into a fine powder using a mortar and pestle. This powdered form was undergone calcination at

900°C in a muffle furnace for 4 hours to synthesize the ESN.

• Transesterification Process: The transesterification reaction was conducted with and without the ESN, where in one assembly the CASO was combined with methanol simultaneously in another assembly mixing CASO with methanol, and the ESN and both the assemblies were refluxed for 4hrs under controlled temperature and agitated to get CABD and (CABD)_C respectively.

• Purification: Both the resulting biodiesels were purified through repeated washings with distilled water, followed by drying to remove any leftover catalyst, methanol, or by-products.

5. SEM Analysis: The structure of the ESN was evaluated through SEM with a Zeiss SEM-EVO18

6. TGA-DTA: The thermal stability and decomposition characteristics of the ESN were investigated using TGA-DTA with a Perkin Elmer Diamond TG/DTA.

5. Characterization of CABD and (CABD)_C

• **FT-IR Analysis:** FT-IR spectroscopy was employed by using Thermo Fischer Scientific FT-IR to identify functional groups in the biodiesels and to confirm the transformation of CASO into CABD and (CABD)_C

• **GC-MS:** The fatty acid composition in CABD and (CABD)_C were determined using

the Thermo Scientific TSQ 8000 GC-MS spectrophotometer to verify percentage composition of fatty acids.

• **Fuel Property Analysis:** The critical fuel characteristics of the biodiesels produced, such as specific density, viscosity, flash point, AV, PV, CN and CV etc., were evaluated using standard testing protocols and compared with ASTM-D6751 and EN14214 standards to determine its viability as a diesel alternative.

RESULTS AND DISCUSSION:

Physicochemical Characteristics of CASO

The physicochemical characteristics of CASO are presented in **Table 1**. The oil yield of CASO obtained is 25.08%. These results align closely with those reported by John Abraham AP. *et al.* for Cassia fistula (27.03%) [14]. The iodine value (IV) measures the level of unsaturation in oil and serves as an important indicator for assessing the number of double bonds in the oil, reflecting its susceptibility to oxidation [15]. The IV determined for CASO was 94 gI₂/100 g oil, which is significantly higher than the 26.48 g I₂/100 g reported by Dangarembizi R for Cassia abbreviate [16]. The IV is comparable to the values of 99 gI₂/100 g and 96 gI₂/100 g reported by Mathur A. *et al.* for Calotropis gigantea and *Jatropha curcas*, respectively [17]. Lower iodine content indicates fewer unsaturated bonds and a reduced tendency of the oil to undergo

oxidative rancidity. Non-drying oils typically have iodine values below 100 g I₂/100 g of oil, while semi-drying oils have iodine values between 100 and 130 g I₂/100 g, and drying oils have values above 130 g I₂/100 g. Because of their non-drying nature, non-drying oils are not suitable for making ink and paint, yet they can be useful in soap manufacturing and are classified as liquid oils [18]. Industry standards for inputs like biodiesel often include the IV as it reflects total unsaturation. However, the relationship between physical and chemical properties and fatty acid composition cannot be established solely through the IV index due to its overly broad scope [19]. Accordingly, oils extracted from CASO are categorized as non-drying. The moisture content and FFA levels in CASO were recorded at 0.72% and 1.2%, respectively. The SV of CASO was found to be 192 mg KOH/g, which is significantly higher than the SV reported by Azeez AM. *et al.* (SV = 159.9 mg KOH/g) and nearly identical to the values seen in the studies by Akbar E. *et al.* (SV = 193.5 mg KOH/g) and Aransiola EF. *et al.* in 2012 (SV = 193 mg KOH/g) for *Jatropha curcas* seed oil [20-22]. The SV of an oil measures its oxidation potential during storage and indicates its degradation level. A higher SV suggests the presence of fatty acids with a greater number

of carbon atoms. It reflects the average molecular weight and, thus, the chain length of a lipid [23]. The elevated SV of CASO

suggests that it is suitable for soap production. The refractive index of CASO is measured at 1.467.

Table 1: Physicochemical properties of CASO.

Parameters	CASO
Oil (%)	25.08
Moisture content (%)	0.72
Saponification value (mgKOH/g oil)	192
Iodine value (gI ₂ / 100g oil)	94
Refractive index	1.467
Relative density (Kg/m ³)	832.67
FFAs (%)	1.2
Unsaponifiable matter (%)	1.12

SEM Analysis:

The SEM image of the ESN is displayed in the **Figure 1**. ESN has porous and irregular surface structure with nanoparticles evenly distributed across a size range of 100 nm. This rough, interconnected porous architecture offers a substantial surface area that boosts reactant adsorption and enhanced catalytic performance. It indicated a honey comb like porous surface as observed by Hu *et al.* [24].

The process of calcination significantly altered the structure of the eggshell, breaking down organic materials and creating a stable inorganic matrix primarily made up of CaO. After undergoing thermal treatment, the ESN maintained its structural integrity, ensuring its durability and reusability. These characteristics, along with the plentiful active sites, rendered the ESN highly effective and suitable for sustainable catalytic applications.

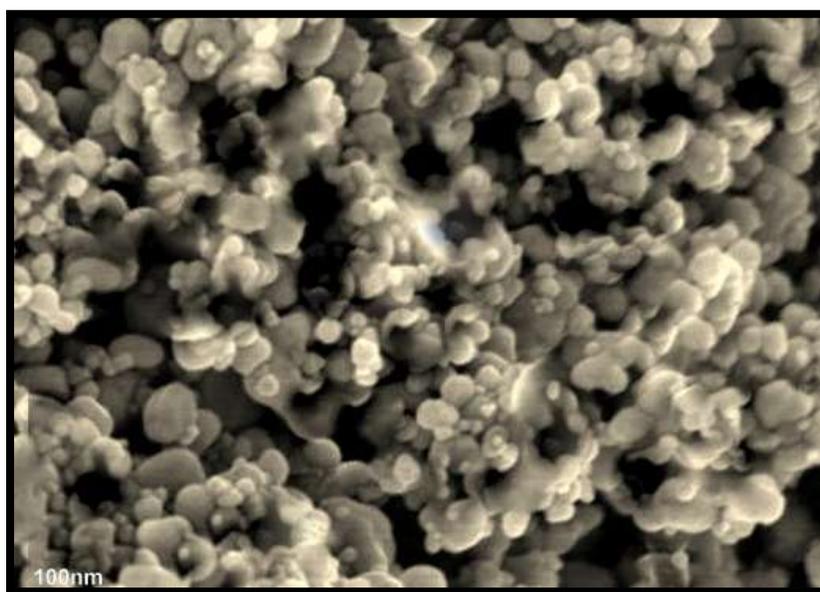


Figure 1: SEM analysis of ESN

TGA-DTA:

The TGA-DTA evaluation of the ESN demonstrated its thermal stability and changes in structure, respective curve is shown in the **Figure 2**. An initial reduction in weight observed around 300°C is linked to the evaporation of moisture and water that is physically adsorbed. A major weight loss occurred between 500°C resulted from the thermal decomposition of CaCO_3 into CaO and CO_2 . The phase, ranging from 300 °C to 500 °C, is linked to the conversion of $\text{Ca}(\text{OH})_2$ to CaO [25].

During this stage, the weight loss reached its peak at 8.2%, as indicated by the DTA curve.

The phase, from 500 °C to 700 °C, is associated with the conversion of CaCO_3 to CO_2 , with a weight loss value of 2.8% [26].

Heating the sample beyond 700°C did not produce any changes, and the weight remained around 90 % of the original sample. The lack of additional weight loss past 700°C suggested the formation of a thermally stable CaO framework. These results confirmed the effective thermal transformation of the eggshell into a robust and catalytically active material, appropriate for high-temperature applications.

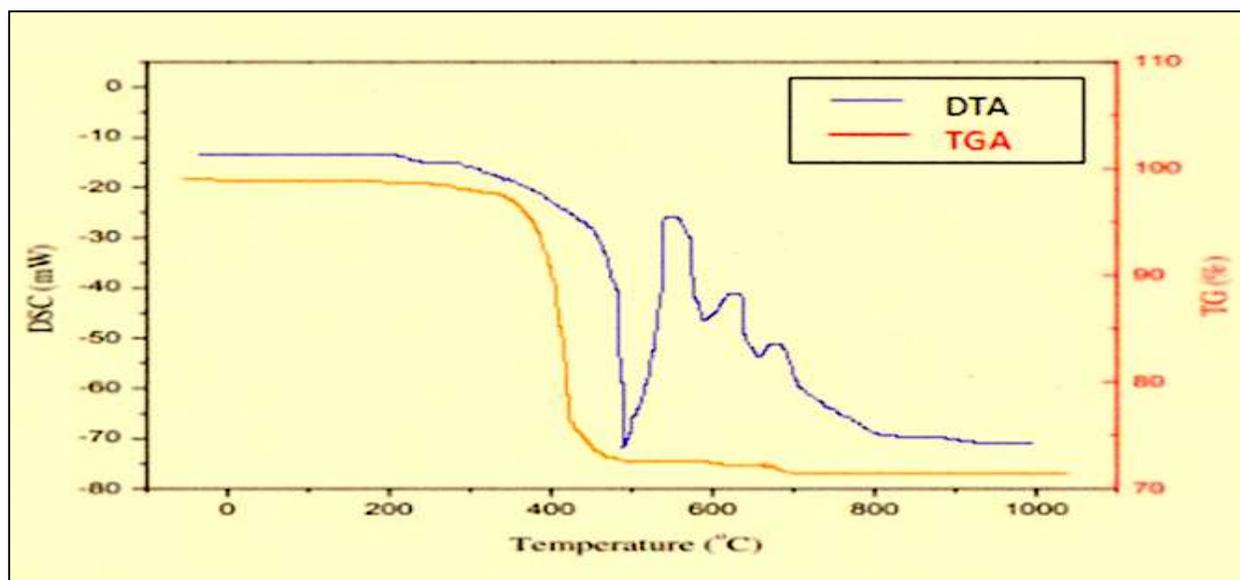


Figure 2: TGA-DTA of ESN

FT-IR analysis of CABD and (CABD)_c:

The FT-IR spectrum for CABD and (CABD)_c is displayed in **Figure 3**. The following is an interpretation of the FT-IR spectrum for

CABD and (CABD)_c: There is no significant differences observed in the peaks of both the biodiesels.

- 3610 cm^{-1} to 3790 cm^{-1} indicated the presence of water impurities.
- 3200 cm^{-1} to 3300 cm^{-1} corresponded to -OH stretching, hydrogen bonding, and water impurities.
- 2895 cm^{-1} , 2990 cm^{-1} and 2845 cm^{-1} represented C-H stretching.
- 2058 cm^{-1} and 2098 cm^{-1} is associated with C=O stretching, indicating the presence of esters.
- 1650 cm^{-1} reflected C=C stretching, confirmed the existence of unsaturated fatty acids.
- 1450 cm^{-1} and 1490 cm^{-1} signified C-H stretching.
- 1115 cm^{-1} and 1195 cm^{-1} revealed C-O bending vibrations, which confirm the presence of esters.
- 715 cm^{-1} and 695 cm^{-1} indicated bending of the C-H group due to the methylene group.
- 675 cm^{-1} -1000 cm^{-1} showed =C-H stretching.

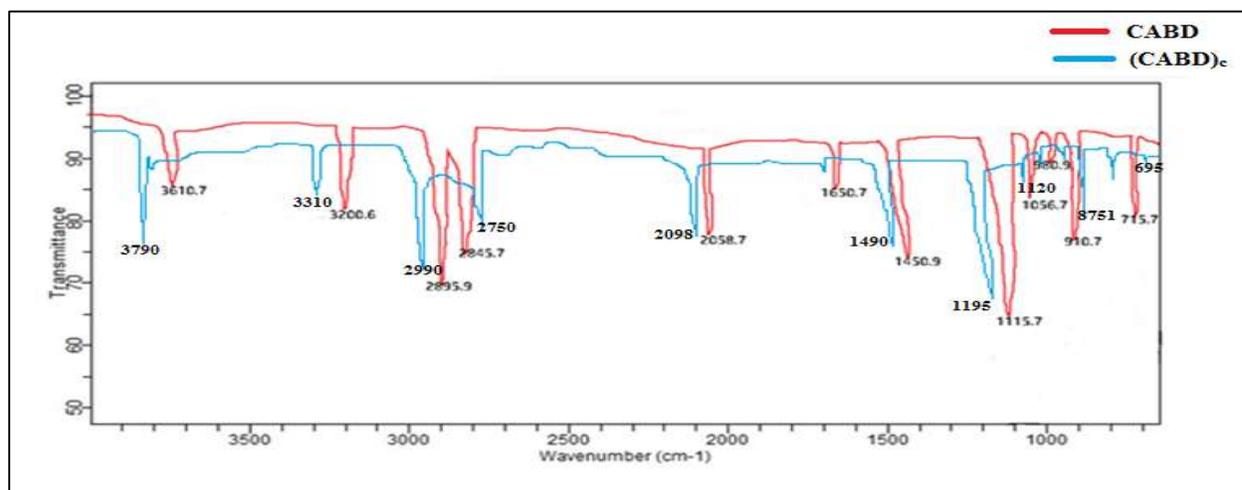


Figure 3: FT-IR SPECTRUM OF CABD and (CABD)_c

GC-MS Analysis of CABD and (CABD)_c:

The CABD and (CABD)_c were analyzed using the GC-MS technique, and the composition of FAMES were examined respective spectrum are shown in the **Figure 4 and 5**. There is no significant difference observed in the percentage of fatty acids composition on the addition of ESN. The

individual peaks in the gas chromatogram were assessed, and the FAME components were identified with the help of an MS database. According to **Table 2**, both the CABD and (CABD)_c fuels generated from CASO feed-stocks exhibited varying proportions of different fatty acids, including palmitic acid (10.2%, 10.1%), stearic acid

(4.5%, 4.2%), oleic acid (25.2%, 25.3), linoleic acid (42.3%, 42.2%), linolenic acid (7.2%, 7.0%), arachidic acid (2.3%, 2.2%), and other acids (8.3%, 8.5%) for the CABD and (CABD)_C respectively. It is observed that oleic acid and linoleic acid are the most predominant fatty acids in the CABD and (CABD)_C fuels analyzed. The amounts of oleic acid and stearic acid found in the CABD and (CABD)_C are slightly greater than those reported for oleic acid (22.51%) and stearic

acid (6.07%) in *Moringa oleifera* by Adedge AA. *et al.* [27]. The level of linolenic acid detected in the CABD and (CABD)_C is marginally higher than that found in waste cooking oil biodiesel (6.82%) according to Chhetri AB. *et al.* [28]. Additionally, the linoleic acid content in the CABD and (CABD)_C exceeds that reported for *Jatropha* (31.6%) and waste cooking oil biodiesel (39.74%) by Augustus GD. *et al.* and Bautista LF. *et al.*, respectively [29, 30].

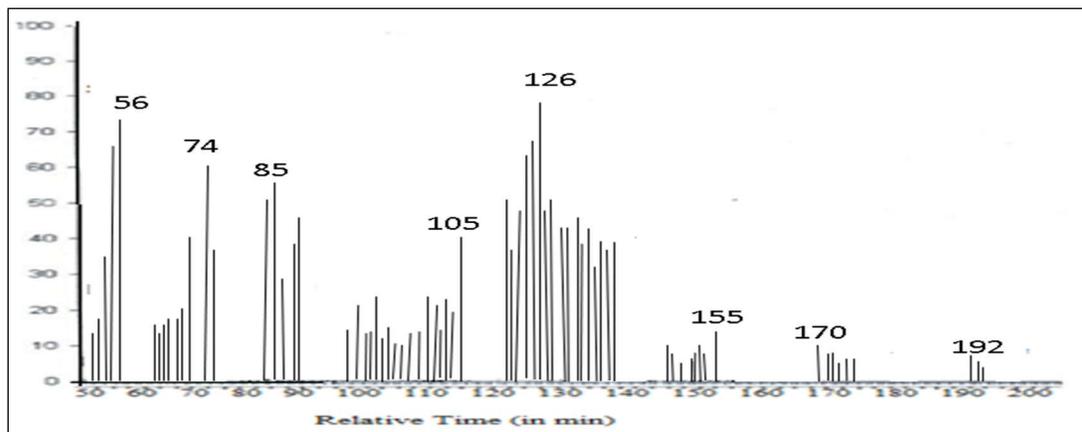


Figure 4: GC-MS spectrum of CABD

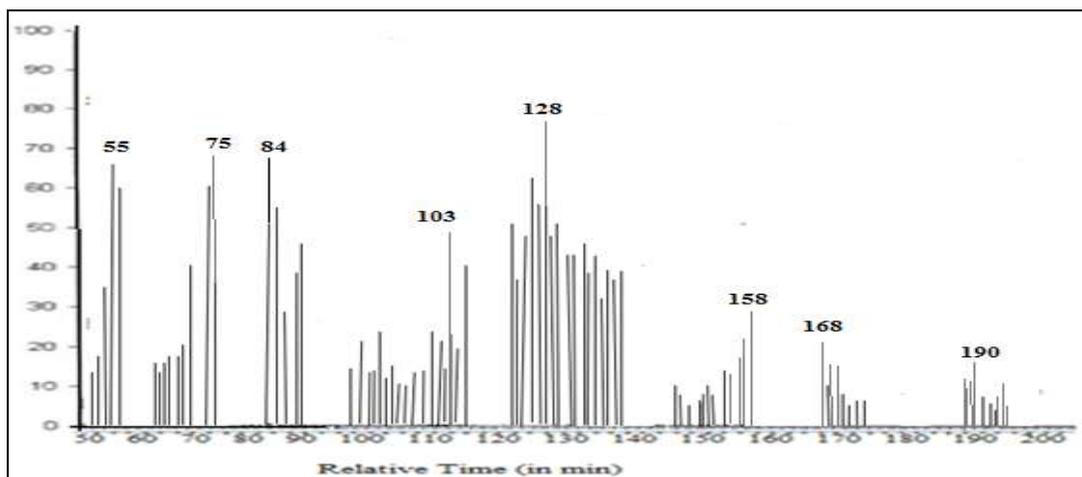


Figure 5: GC-MS spectrum of (CABD)_C

Table 2: Fatty acids composition of CABD and (CABD)_C (uncorrected weight percent) by GC-MS

Fatty acids (%)	CABD	(CABD) _C
Palmitic acid (C _{16:0})	10.2	10.1
Stearic acid (C _{18:0})	4.5	4.2
Oleic acid(C _{18:1})	25.2	25.3
Linoleic acid (C _{18:2})	42.3	42.2
Linolenic acid (C _{18:3})	7.2	7.0
Arachidic acid (C _{20:0})	2.3	2.2
Others	8.3	8.5

Fuel characteristics of CABD and (CABD)_C compared to ASTM D6751 and EN 14214 biodiesel standards [9, 10]

The properties of the CABD and (CABD)_C obtained from CASO were examined and presented in Table 3. In various aspects, both the fuel characteristics closely align with the biodiesel standards [9, 10]. The biodiesel viscosity standard is set between 3.5 mm²/s and 5.0 mm²/s, with CABD and (CABD)_C measuring at 3.9 mm²/s and 3.0 mm²/s respectively, which falls within this range. It was found that (CABD)_C has a comparatively low viscosity. The viscosity of non-edible oil tends to decrease significantly during the transesterification process for biodiesel. A lower viscosity in biodiesel facilitates easier pumping into an engine for the purpose of atomization. Conversely, a high viscosity can lead to inefficient atomization of the fuel spray, resulting in imprecise operation of fuel injectors [31, 32]. Most literature suggests that kinematic viscosity can be influenced by the degree of unsaturation [33-35]. The kinematic viscosity recorded for CABD and (CABD)_C were 4.1 mm²/s and 3.0 mm²/s, and

it is also clear that its specific gravity 0.812 g/cm³ and 0.800 g/cm³ for the CABD and (CABD)_C are more aligned with the biodiesel standard.

For the transesterification reaction to occur efficiently, the AV needs to be lowered to below 2 mg of KOH/g of oil [36]. However, non-edible oils that exhibit an AV exceeding 2 mg KOH/g necessitate a pre-treatment to reduce the AV before conversion to biodiesel. The AV reflects the level of FFA present in a specified amount of oil and provides insight into the extent to which triglycerides in the oil are broken down by lipase into FFA and other physical factors, such as light and heat. This is influenced by the level of rancidity, which serves as an indicator of quality [37]. In this study, the AV for CABD was found to be 1.59 mg KOH/g and for the (CABD)_C was 0.30 mg KOH/g. This noted AV is slightly lower than that of *Ricinus communis* (0.91 mg KOH/g) but significantly lower than that of *Hevea brasiliensis* (202 mg KOH/g), as reported by Obanla OR. *et al.*, 2021 [38]. A high AV suggests a substantial presence of FFA in the oil, making oil with a high AV more prone to

rancidification and unsuitable for biodiesel production. Therefore, the low AV value for the (CABD)_C make it more suitable for the biodiesel production.

The PV serves as an indication of rancidity in oils; therefore, oils with a high PV exhibited a lower resistance to peroxidation during storage [39]. The PV of CABD and (CABD)_C were recorded at 2.4 mEq/kg of oil and 1.2 mEq/kg of oil, complying with biodiesel criteria (the maximum limit). This indicated that both the biodiesels can be stored without significant deterioration. A low PV indicates stability in an oil sample, while peroxide levels ranging between 20 and 40 mEq/kg signify rancidity [40]. The PV of the biodiesels samples examined remained below the maximum threshold, suggesting that the oils would resist oxidative rancidity. The CV of CABD and (CABD)_C were determined to be 39.645 mJ/kg and 45.895 mJ/kg respectively. The CV of any fuel is vital, as a higher calorific value indicates greater engine power output. The (CABD)_C production CV was higher than that of *Jatropha* diesel (42.770 mJ/kg), as reported by Azeez AM. *et al.* [20] The CV of both the biodiesels were higher than those of popular biodiesels like Mahua (22.19 MJ/kg) and Neem (26.9 MJ/kg), according to Mishra RK *et al* [41]. The recorded flash point and fire point for CABD

were 129°C and 134°C, respectively. The recorded flash point and fire point for (CABD)_C were quite higher 135°C and 140°C, respectively. The elevated flash point of (CABD)_C reduced the chances of unexpected fire hazards. After analyzing the cloud point, it was confirmed that CABD and (CABD)_C met the biodiesel standards, which were obtained at -9°C and -3°C for both the biodiesels. Lower levels of ash and carbon residue contributed to reduced carbon buildup on engine components, thereby prolonging engine lifespan. CABD contained an ash concentration of 0.03% and a carbon residue level of 0.04% while (CABD)_C contained an ash concentration of 0.02% and a carbon residue level of 0.01%. Both the ash and carbon concentrations in (CABD)_C were low enough to allow its use in a compression ignition engine without needing modifications. The CN reflects the ignition quality of a fuel and influences the amount of white smoke and combustion roughness. The CN for CABD and (CABD)_C were recorded at 55 and 75. While a much higher CN in (CABD)_C may lead to a shorter mixing time for fuel and air, potentially increasing soot emissions, it can also enhance fuel reactivity and combustion efficiency. The biodiesel yield for CABD and (CABD)_C were found to be 81.80% and 85.90% respectively.

Table 3: Fuel properties of CABD and (CABD)_C compared with ASTM-D6751 and EN 14214 biodiesel standard [9, 10]

Fuel properties	CABD	(CABD) _C	ASTM D6751 ^[9]	EN 14214 ^[10]
Cloud point (°C)	-9	-9	-3 to -12	-----
Pour point (°C)	-3	-3	-15 to -16	-----
Density (Kg/m ³)	859	840	880	860-900
Flash point (°C)	129	135	130	Min. 101
Fire point (°C)	134	140	-----	-----
Kinematic Viscosity (mm ² /s)	4.1	3.0	1.9-6.0	-----
Viscosity (mm ² /s)	3.9	3.0	-----	3.5-5.0
Specific gravity (g/cm ³)	0.812	0.800	-----	-----
Ash content (wt %)	0.03	0.02	0.05max	Max. 0.02
Carbon residue (wt %)	0.04	0.01	0.05max	-----
Acid value (mg KOH/g)	1.59	0.30	0.50 max mg KOH/g	Max. 0.50
Peroxide value (mEq/kg)	2.4	1.2	-----	-----
Cetane no.	55	75	47 min	Min. 51.0
Calorific value (mJ/Kg)	39.645	45.895	-----	Min 35.0
Biodiesel yield (%)	81.80	85.90	-----	-----

CONCLUSION:

The study emphasized the production (CABD)_C utilizing an ESN, which showed improved fuel characteristics in comparison to CABD. The application of the ESN enhanced several critical parameters, including flash point, fire point, kinematic viscosity, specific gravity, ash content, carbon residue, AV, PV, CN, and CV. Specifically, the (CABD)_C had a higher cetane number (75 vs 55), an improved calorific value (45.895 MJ/kg vs 39.645 MJ/kg), and a greater yield (85.90% vs to 81.80%) compared to CABD. These results highlighted the ESN's potential as an environmentally friendly, effective, and sustainable method for improving biodiesel's

Abbreviations:

S. No.	LIST OF ABBREVIATIONS
1.	AV- ACID VALUE
2.	AOCS- AMERICAN OIL CHEMIST'S SOCIETY
3.	ASTM- AMERICAN SOCIETY FOR TESTING MATERIAL
4.	CABD- CASSIA ANGUSTIFOLIA BIODIESEL
5.	(CABD) _C - CASSIA ANGUSTIFOLIA BIODIESEL UTILIZING EGG SHELL NANOCATALYST
6.	CASO- CASSIA ANGUSTIFOLIA SEED OIL
7.	CV- CALORIFIC VALUE

fuel properties, positioning it as a strong candidate for renewable energy applications.

Acknowledgment

We are thankful to the State Government of Rajasthan for providing funding under Rajasthan higher technical education quality improvement scheme (RHTEQIS) and The Head, Department of Chemistry, Jai Narain Vyas University (Jodhpur) for providing necessary facilities.

Conflict of Interest: There is no conflict of interest.

Source of funding:-We have received a financial grant from the State Government of Rajasthan under the RHTEQIS project for the completion of this project.

8.	CN- CETANE NUMBER
9.	EN- EUROPEAN NORMS
10.	ESN- EGGSHELLS NANOCATALYST
11.	FAMEs - FATTY ACIDS METHYL ESTERS
12.	FFA- FREE FATTY ACIDS
13.	FT- IR - FOURIER TRANSFORM INFRARED SPECTROSCOPY
14.	GC- MS- GAS CHROMATOGRAPHY MASS SPECTROMETRY
15.	IV- IODINE VALUE
16.	PV-PEROXIDE VALUE
17.	PUFAs -POLY AND SATURATED FATTY ACIDS
18.	SEM-SCANNING ELECTRON MICROSCOPY
19.	SV- SAPONIFICATION VALUE (SV)
20.	TGA-DTA THERMOGRAVIMETRIC ANALYSIS, AND DIFFERENTIAL THERMAL ANALYSIS

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