

https://doi.org/10.46488/NEPT.2023.v22i03.047

Vol. 22

2023

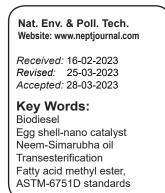
# An Approach for Biodiesel Production from Blends of *Azadirachta indica* and *Simarouba glauca* Triglycerides by Graphene-Doped Calcium Oxide Catalyst and Its Comparative Studies

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# INTRODUCTION

# Energy increases are in demand because of rapid population growth and automation worldwide. The increase in energy demand in the last few years, because of the fast population growth and mechanization all over the country, has made humans much more dependent on non-renewable energy fuel resources. These fuel resources are rapidly depleting, resulting in price instability, decreased global security, stockpile ambiguity to consuming nations, more significant expenses on crude oil fuel imports, and vulnerable economic conditions (Verhoef et al. 2018). Nations depend on nonrenewable energy fuel resources, which has led to price fluctuations, decreased energy security, uncertainty in fuel supply to consuming countries, high costs associated with fuel imports of crude oils, and vulnerable economic conditions affected (Jiménez-Xamán et al. 2019). Additionally, these depletable energy sources cause environmental pollution by releasing toxic emissions (Jacobson 2009). The substantial addition of GHG and SOx to the atmosphere results in global warming and acid rain. Because of these problems, it is necessary to search for other environmentally friendly

#### ABSTRACT

Over the past several decades, people from many nations have adopted and supported using biodiesel energy sources due to their accessibility and advantages in reducing  $CO_2$  and H.C. emissions to the environment. Today, biodiesel is recognized as a sustainable alternative energy source. Commercially, biodiesel was produced by converting homogenous oil treated with a catalyst like NaOH or KOH in Alcohol. These homogeneous catalysts are hazardous to the environment and cannot be recycled. As an alternative, this research article focuses on biodiesel production from a 1:1 blend of *Simarubha glauca* (Laxmitharu in Kannada) and *Azadirachta indica* (Neem) triglyceride via acid-base catalyzed transesterification reaction. The heterogeneous-based graphene-doped CaO was used as a catalyst obtained through the calcination method by doping it with graphene oxide by the hummers' method. SEM, FTIR, and XRD were used to characterize the GaO-CaO catalyst. The results predict that the prepared catalyst yielded a high percentage of ASFAME (94.0%) and meets the quality as per ASTM standards 6751D.

energy resources that fulfill the energy demands (Boldrin et al. 2009). As per the survey, a sustainable renewable resource for replacing conventional fuels in the future, especially in the transport sector, is biodiesel can be considered as an alternative. Both biodiesel and diesel exhibit similar physicochemical properties. They can be used commercially by blending with diesel fuel or biodiesel (B100) in existing CI engines without moderation (Shelke et al. 2016). As plants produce these resources as a source and can absorb  $CO_2$  emitted into the atmosphere, utilizing biodiesel has the benefit of producing zero carbon dioxide emissions. Therefore, biodiesel fuel sources are known to be  $CO_2$ -neutral (Živković et al. 2017).

The feedstock for biodiesel production is from treeborne oil seeds in India; these non-edible oils are obtained from tree-borne oil species (TBO), some of which are still traditionally used as a fuel source in rural areas (Cheng et al. 2016). Tree Borne Oilseeds (TBOs) are cultivated in distributed forest and non-forest areas and wasteland/deserts/ hilly areas concerning agro-climatic conditions (Gomiero 2015). In India, the essential TBOs for biodiesel resources are as follows Neem (Azadirachta indica), Karanja (Pongamia pinnata), Mahua (Madhuca indica), Jatropha (Jatropha curcas), Kusum (Schleichera), Simarubha glauca, Pilu (Salvadora oleoides), Bhikal (Prince piautilis), Surahonnae (Calophyllum inophyllum), etc., The oil content found in these seeds varies between 21 to 60%. Pongamia pinnata, Neem (Azadirachta indica), and Simarubha glauca (Laxmitharu in Kannada) are among the several tree-borne oilseeds (TBOs) available for biodiesel production. These tree-borne seeds are seasonal varieties only accessible for 2-3 months annually. The seeds of neem and simarubha contain a high percentage of oil (40-50 %), while the seed pulp provides a significant source of protein. Furthermore, the resources of the neem and simarubha plants are employed in cancer therapy and bacterial infection. Cultivating TBOs and implementing blended seed oil resources for biodiesel production overcome the problem of raw material scarcity. The consumption of total available nonedible oil resources will reduce the Indian crude oil imports from other countries and also improves the Indian economy (Dhyani et al. 2015). In traditional techniques, biodiesel was synthesized with a homogenous catalyst; however, these homogenous catalysts are allied with problems such as soluble, non-recycling, high percentage of soaps and glycerol during transesterification reaction, resulting in a low yield fatty acid methyl ester (Biodiesel). Therefore, to overcome limitations nowadays, heterogeneous catalysts are used as a catalyst in transesterification reactions. The GaO-CaO catalyst exploited in this research was a heterogeneous catalyst derived from waste eggshells calcinated and doped with graphene oxide. The prepared catalyst is environment-friendly and used in biodiesel production. Furthermore, the crude biodiesel was refined from the novel reefing technique in the presence of areca husk resin(ARH). The biodiesel quality was evaluated according to ASTM Standard 6751 D and compared with conventional methods.

# MATERIALS AND METHODS

#### **Materials**

Azadirachta indica and Simarouba glauca oil were received

from the Biofuel Research Information and Demonstration Centre, JNNCE, Shimoga, Karnataka. The areca husk was obtained from a farmer in the Shivamogg villages, and further, the chemicals utilized in this research were procured from S.D. fine chemicals, Mumbai, India.

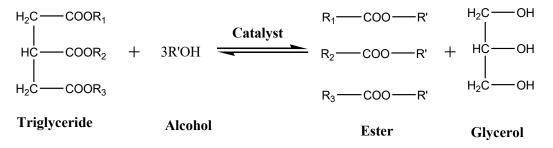
#### **Preparation of GO-CaO Catalyst**

The modified Hummer process was followed to synthesize graphene oxide (GO). Graphite powder (1.0 g) with 20 mL of concentrated sulfuric acid in a clean 250 mL R.B. flask was stirred in the ice bath. The above mixture is treated with NaNO<sub>3</sub> and KMnO<sub>4</sub> at the maintained temperature of  $5^{\circ}$ C. Further, water was added, and the suspension was heated to 98°C. Finally, the obtained graphene mixture was treated with  $H_2O_2$  (30%). The product was filtered, washed with distilled water, and dried. The dried powder was refluxed with derived CaO from waste eggshell at 300°C for 2 h to yield a catalyst. The GO-CaO catalyst was allowed to cool before being stored in an airtight container for future use in biodiesel synthesis.

## Synthesis of Azadirachta Indica-Simarouba Glauca Fatty Acid Methyl Ester (ASFAME)

Transesterification, or fatty acid methyl ester (FAME), was synthesized from mixed triglyceride with an alcohol-containing dissolved catalyst. In recent years, transesterification has been the most accepted for biodiesel synthesis (FAME). The general reaction of the transesterification is shown in Scheme 1 (Fadhil & Saeed 2016)

The biodiesel was produced using acid-catalyzed or base-catalyzed processes, depending on the fatty acid concentration of the feedstock. Acid-catalyzed transesterification processes occur in the presence of acids, the most frequent of which is sulfuric acid(Agarwal 2007). The acid-catalyzed process is the first step before the basecatalyzed transesterification operations; the resulting ASO oil is evaluated for free fatty acid content (FFA). Based on the presence of FFA in ASO oil, the ASO oil is first treated with sulphuric acid for the esterification process, as seen



Scheme.1. Trans-esterification reaction Azadirachta indica-Simarouba glauca fatty acid methyl ester.



Fig. 1: Transesterification setup (a), Illustration of Simarouba glauca and neem fatty acid methyl ester on the top and the glycerol separation (b).

in Fig. 1. The reaction mixture was heated at 60-65°C for around 90 minutes on a hot pallet magnetic stirrer with 900 RPM until the reaction was completed. After 90 minutes, the reaction mixture was transferred to a separating funnel to separate the acid impurities at the top(Yatish et al. 2018); the reduced FFA content of oil was exposed to base-catalyzed transesterification.

## **Base Catalyzed Trans-Esterification**

The recovered ASO oil layer (2.2.1) is transferred in a clean, dry R.B. flask. Again, FFA content was tested for ASO. The catalyst concentration was tuned by combining 0.25, 0.5, 0.75, 1.0, and 1.25 g of produced GO-CaO with a constant amount of methanol. The reaction was set up similarly to an acid-catalyzed reaction, and the reaction mixture was refluxed for roughly 1 h at 60°C. The mixture was allowed to cool to yield Azadirichita-Simaruba fatty acid methyl ester (ASFAME) and crude glycerol at the bottom. The obtained biodiesel was tested for various physicochemical quality parameters such as kinematic viscosity, fire point, Fourier transform infrared spectroscopy (FTIR), relative density, gas chromatography (G.C.), and flash point. Further, the ASFAME yield was optimized.

# **Bioresin Treatment for Refining Crude ASFAME**

The ion exchange column 30 cm<sup>-1</sup> in height and 1 cm<sup>-1</sup> in diameter was pre-filled with Areca husk resin (ARH) with crude ASFAME passed over bio-resin. The eluted refined ASFAME from the ion exchange column with a 30 drops/ minute flow rate, according to Chethan et al. (2023),was collected and stored separately for further study. The physicochemical parameters of refined ASFAME from the ion exchange process and ASFAME refined from the

conventional approach were compared using the ASTM 6751 D standard.

# Characterization of GO-CaO and ASFAME

Thermo Nicolet iS50 FTIR spectrophotometer was used to investigate ASO oil and ASFAME liquid samples. The Thermo Nicolet iS50 FTIR spectrophotometer with 0.2 cm<sup>-1</sup> resolution was used for the FTIR spectral examination in the spectral range between 400 and 4000 cm<sup>-1</sup>. The morphological aspects of GO-CaO and AHR were investigated using the JOEL JSM-IT500. For GO-CaO catalysts with known crystalline characteristics, X-ray diffraction experiments were performed.

# **RESULTS AND DISCUSSION**

# SEM Characteristics of Graphene-CaO Catalyst

Fig. 2(a-d) are the Scanning Electron Microscope (SEM) images of the Graphene coated CaO respectively. SEM provides high-resolution pictures of a sample surface, exposing features as low as 1 nm. GaO – CaO catalyst magnification was observed in SEM at 500X, 1,500X, 3,500X, 7,000X, and 10,000X, respectively (Musharraf et al. 2012a).

X-ray powder diffraction (XRD) analysis confirms the phase identification of a crystalline material and can provide information on unit cell dimensions as per the hkl values. The D/MAX 2200PC diffractometer (Rigaku Corporation, Japan) with copper K $\alpha$  irradiation was used, operating at 40 kV and 30 mA with a scanning rate of 0.2° min<sup>-1</sup> in the 2 $\theta$  range of 0–80° Fig. 3 shows that the GO concentration in Calcium Oxide significantly influenced the variation in characteristic peak intensity and crystal grain size, which

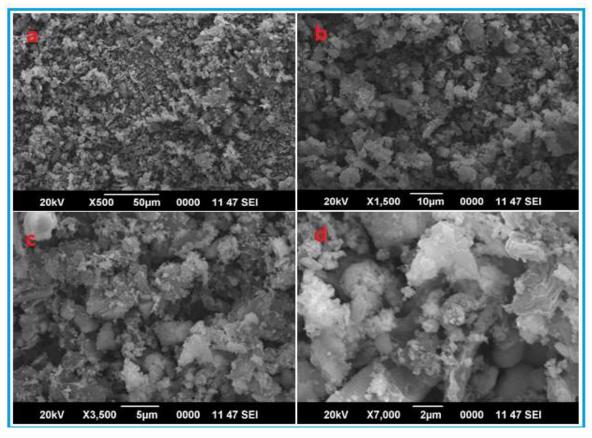


Fig. 2: Scanning electron microscope images of the graphene-coated CaO XRD characteristic of graphene-CaO catalyst.

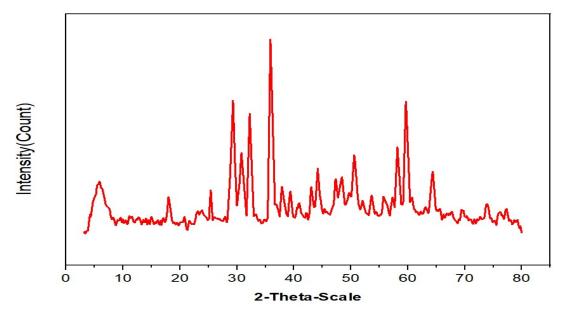


Fig. 3: PXRD of graphene coated calcium oxide.

Sl.No	Samples	GO-CaO in [w/v]	Yield [%]
1.	ASO-1	0.25	61.10
2.	ASO-2	0.5	94.69
3.	ASO-3	0.75	53.40
4.	ASO-4	1	50.50
5.	ASO-5	1.25	44.64

Table 1: Optimization of GO-CaO and ASO Oil.

displayed a characteristic pattern as GO (Sharma et al. 2021) agriculture, forestry, and other land-use practices account for 24% of global greenhouse gas (GHG. The XRD details of GaO-CaO show that CaO is cubic in structure and was confirmed by SEM morphology.

#### Optimization of Catalyst and ASO Oil for Biodiesel Synthesis

ASO oil was pretreated with methanol (0.60 w/w)containing 1% w/w H<sub>2</sub>SO<sub>4</sub> catalyst was refluxed for 1 h at 60°C. in the second step of the reaction, the product from the first step was used for transesterification with GO- CaO catalyst using specified optimization conditions designed (Table 1). The optimization conditions for the second step catalyst concentration with an increase in 0.25 g of catalyst, reaction time for 65 min, and methanol/oil molar ratio were carried out as per Graboski and McCormick (1998). Table 1 results show that as the concentration of the catalyst increases up to 0.5g of Go-CaO results in a high percentage of ASFAME (biodiesel) due to the high reactivity of catalyst with large surface area results in proper separation of ester and glycerol layers (Colombo et al. 2017). Moreover, it is observed that biodiesel yield decreased as an increase above 0.5 g of the catalyst.

#### Physicochemical Properties of the ASFAME

The physicochemical properties of ASO oil, ASFAME, conventional biodiesel method, and diesel were analyzed

as per ASTM standard 6751D. Table 2 results show that the physicochemical of ASO oil were reduced after the transesterification reaction (Alptekin & Canakci 2008). The biodiesel obtained after optimization catalyst at 0.5g of GO-CaO catalyst and methanol, ASO ratio of 0.20:1 ratio yields 94% of ASFAME. Further, it is observed that the percentage yield of ASFAME filtered from AHR resins resulted in a high yield of biodiesel, compared to the percentage yield of ASFAME from the conventional biodiesel refining method (86%). Moreover, table -2 shows that ASFAME refined from novel refining AHR resins are of superior quality, and purity was found to be similar to conventional diesel quality within the limits of ASTM standards 6751D. Hence, the synthesized biodiesel from the newly proposed method is safer and eco-friendly to adopt as a fuel source in present IC engines

#### FTIR Characterization of 1:1 ASO oil and ASFAME

The I.R. spectra show the stretching and bending vibrations of Azadirachta indica and Simarouba glauca oil (1:1) blend and ASFAME (Fig. 4). A narrow peak at 2876 cm<sup>-1</sup> corresponds to -C.H. stretching, showing the oil's presence. The intense peak at 986 cm<sup>-1</sup> is due to the presence of ester groups which was expected for C-O-C stretching. The vibration peaks at 1789 cm<sup>-1</sup> are due to the C=O stretching of the triglyceride ester and the peaks at 1464 cm<sup>-1</sup>. Vibration for C–H bending further observed that the peak at 876 cm<sup>-1</sup> is due to methylene rocking vibration in the ASFAME. The peak of C=O for neem seed oil appeared at  $1674 \text{ cm}^{-1}$ bending, and its intensity corresponds to 3387 cm<sup>-1</sup>. A broad light peak at cm<sup>-1</sup> corresponds to -C.H. stretching. This confirms the presence of the alkane group. A sharp peak at 3855.1 cm<sup>-1</sup> corresponds to -C.H. stretching. This confirms the presence of the alkene group (Silverstein and Webster 1988). A broad light peak at 148.3 cm<sup>-1</sup> corresponds to the -C-O stretching of the carboxylic ester group (Kim et al. 2007). This confirms the presence of the ester group. A broad

Table 2: Physicochemical properties of ASO oil, ASFAME, and conventional diesel.

Sl. No.	Name Of The Analysis	ASO Oil	Conventional Biodiesel refining	ASFAME By ARH Refining	Diesel	Test Method ASTM 6751D
1.	Density (kg.m <sup>-3)</sup>	1.58	0.990	0.864	0.79	D-287
2.	Kinematic Viscosity at 40°C (Cst)	61.87	6.09	5.62	5.20	ASTM D 445
3.	Flashpoint (°C)	219	185	175	74	D93
4.	Fire point (°C)	230	188	180	78	D93
5.	Copper corrosion test at 50°C for 2 h	Complies	Complies	Complies	Complies	D 130
6.	Moisture (%)	0.5	0.05	0.01	0.01	
7.	Calorific value kJ.kg <sup>-1</sup>	41.0	39.0	40.0	43.0	
8.	The yield of ASFAME (%)		86.0	94.0		

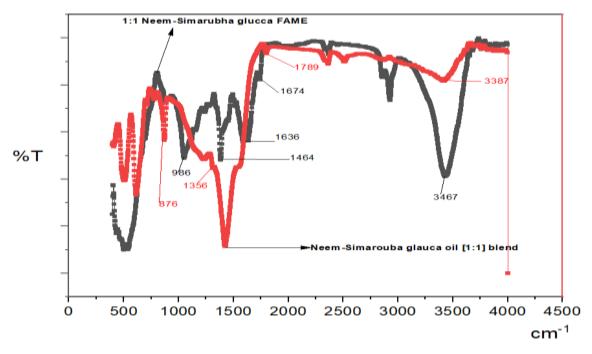


Fig. 4: FTIR spectrum of ASO and ASFAME (biodiesel).

light peak at 1743 cm<sup>-1</sup> corresponds to -C=O stretching. This confirms the presence of the carboxylic ester group. A broad light peak at 1246 cm<sup>-1</sup> corresponds to -C.O. stretching, and its intensity corresponds to 3347 cm<sup>-1</sup> (Sorichetti et al. 2014). This confirms the presence of the carboxylic ester group. A broad light peak at 1435 cm<sup>-1</sup> corresponds to -CH<sub>3</sub> bending. This confirms the presence of the alkane group. The shifts in I.R. peaks confirm the formation of ASO oil into ASFAME.

## Gas Chromatography Analysis of Azadirachta indica and Simarouba glauca Oil

Fig. 5 illustrates a G.C. chromatogram of triglycerides from ASO oil blends. Fig. 6 displays G.C. data from ASFAME, revealing that the separated peaks are free of glycerin, mono, di, and triglycerides (Musharraf et al. 2012b), showing that the triglycerides were transformed into short-chain fatty acid methyl ester. The graph clearly shows that; the produced biodiesel is free from glycerin by comparing the data of standard G.C. of biodiesel.

#### Heat of Combustion

ASFAME (biodiesel) blended with diesel has been discovered to have similar conventional diesel qualities and lower SOx and H.C. emissions during combustion in IC engines. The heat of combustion, also known as calorific value, is the heat created by the fuel within the engine that allows it to accomplish work. According to Freedman and

Bagby, calorific values rose with chain length, and ASO oil, Biodiesel, and diesel were 41.325, 39.345, and 43783 kJ/kg, respectively (1989). Because of the separation of glycerol and other components in the ASO backbone, the calorific value of ASO oil is roughly 8% less than that of diesel; esterification of ASO oil into biodiesel lowers the calorific value of diesel.

#### Wash Water Demand Calculations

The traditional biodiesel refining process uses water. For one liter of crude ASFAME, refining takes roughly 10 to 11 liters of warm water (40°C). It generates an equal amount of contaminated biodiesel-washed water. The Na<sup>+</sup> ion and glycerol in crude ASFAME-washed water were analyzed using Dunstan's and the phenolphthalein test. In the usual approach, the ASFAME yield was roughly 86.0%. With decreased processing time, crude ASFAME refining via developed AHR resins yields 94.0% without warm water (40°C) and fulfills ASTM standard 6751D (Table 2). As a result, the devised technique of refining crude ASFAME in the presence of an ion exchange column packed with AHR resin consumes less water (Sorichetti et al. 2014).

# CONCLUSION

This research aims to study the synthesis of ASFAME using blends of Azadirachta Indica and Simarouba

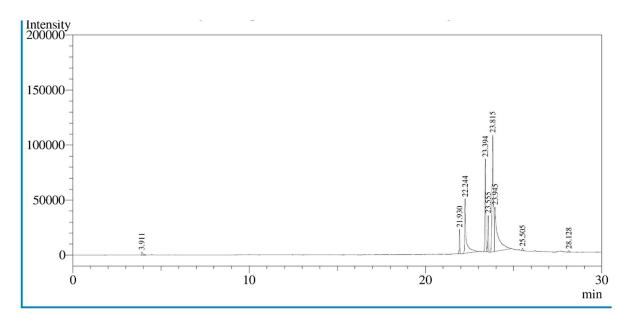


Fig. 5: Gas chromatogram analysis of ASO oil.

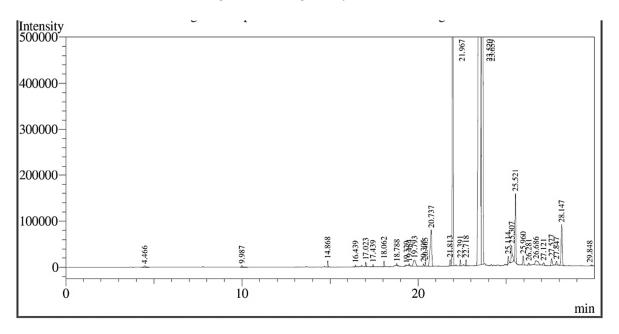


Fig. 6: Gas chromatogram analysis of ASFAME (Biodiesel).

glauca oils (ASO) in the presence of a heterogeneous catalyst, GO-CaO, made from a waste eggshell. The ASFAME yield was optimized using various catalyst concentrations, and it was found that 0.5 g of GO-CaO yielded 94.69% ASFAME (biodiesel). Furthermore, the physicochemical parameters of ASFAME were evaluated and compared with conventional diesel, resulting in high-quality biodiesel that meets ASTM Standards 6751D.

This study helps to meet fuel demand by employing the ecologically friendly heterogeneous catalyst GO-CaO, which can be recovered and reused as a catalytic material. This article helps small-scale biodiesel manufacturers get catalysts at a lower cost and use various residual wastes in synthesizing catalysts. The proposed work helps decrease disposal challenges and ensure sustainability by blending different oils for biodiesel

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