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SYNTHESIS OF SULFONATED CARBON-BASED CATALYST FOR TRANSESTERIFICATION OF NEEM SEED OIL

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Abstract

This study examines the synthesis of sulfonated carbon-based catalysts made from neem seed residue for the transesterification of neem seed oil, which produces biodiesel. The catalysts were synthesised via pyrolysis and sulfonation with conc. H_2SO_4 . Biodiesel yields were recorded by adjusting the catalysts' sulfonation temperatures (140–200 °C). The maximum yield (95.9%) was attained at a sulfonation temperature of 200 °C. The catalyst's characterisation revealed that the catalyst possessed good surface area, pore volumes, and pore sizes for reaction during transesterification. The density, viscosity, specific gravity, cetane number, and calorific value of the resultant biodiesel were in compliance with ASTM D6751 criteria. This study shows how neem seed oil and residue could be used as sustainable feedstocks for effective biodiesel synthesis, supporting renewable energy sources and valuing agricultural waste.

Key words: Pyrolysis, synthesis, sulfonate catalyst, neem seed oil, biodiesel,

INTRODUCTION

The biodiesel synthesis process involves the transesterification of feedstock oils with alcohol in the presence of a catalyst. Common feedstocks include both edible oils (e.g., palm, soy bean, canola, sunflower, and groundnut oil) and non-edible oils (e.g., karanja, jatropha, castor, tallow and neem seed oil). The alcohol acts as a solvent, facilitating the reaction, while the catalyst accelerates the process and enhances biodiesel yield (Hamza et al., 2021).

There are three types of catalyst employed for transesterification reactions: enzymes, homogeneous catalysts and heterogeneous catalysts. Enzymatic catalysts are highly efficient but impracticable for industrial applications due to their high

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cost and slow reaction rates (Christopher et al., 2014; Tran et al., 2013). Homogeneous catalysts, which include base and acid variants, are widely utilised because of their affordability and well understood-properties (Akpan et al., 2023). While homogenous base catalysts generally yield higher biodiesel output, their tendency to form soap during reaction complicates separation and purification (Akpan et al., 2023). Conversely, acid homogeneous catalyst can simultaneously catalysed both esterification and transesterification without soap formation but require large quantities of water for purification, making them corrosive and less efficient (Hamza et al., 2021).

Heterogeneous catalysts offer solutions to these challenges by remaining in the solid phase, simplifying recovery, reducing impurities, and eliminating soap formation (Talha & Sulaiman, 2016). They are further classified into acid and base heterogeneous catalysts, with base variants exhibiting higher catalytic power (Veluru et al., 2022). However, their high cost increases biodiesel production cost. To mitigate this issue, researchers have explored biomass-derived catalysts, which are cost effective, environmentally friendly, and highly efficient (Inayat et al., 2021). These green catalysts, sourced from biomass, offer advantages such as high reusability, increased active reaction sites, and reduced production cost (Chutia & Phukan, 2024; Ribeiro et al., 2023).

The goal of this work is to produce a sulfonated carbon based catalysts obtained from residue of neem seed post oil extraction for the production of biodiesel via transesterification method using neem seed oil. Given its abundance in northern Nigeria, neem (*Azadirachta indica* A. juss) is a promising feedstock for the production of sustainable biodiesel. Neem seed oil contains a diverse fatty acid composition, while neem seed residue- rich in carbohydrates, proteins, fiber, and essential minerals-serves as an excellent precursor for carbon based catalyst synthesis (Aransiola, 2012; Bakthavatsalam, 2016). The catalyst preparation process involves two primary stages: carbonization (or pyrolysis) and activation. The carbonisation stage transforms the biomass into biochar, which is subsequently modified using activating agents such as KOH, H₂SO₄, HNO₃, K₂CO₃, ZnCl₂, CaO, ZnO, (Chutia & Phukan, 2024). While previous studies have explored biomass-catalyst from materials such as cocoa pod husk (Betiku et al., 2017), palm seed cake (Akinfalabi et al., 2017), neem seeds residue (Inayat et al., 2021), rubber seed kernel (Iriany et al., 2022), coconut mesocarp (Espinoza-Montero et al., 2024)

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and bean husk (de Araújo Sobrinho et al., 2025), no published research has evaluated the catalytic performance of sulfonated neem seed residue catalysts for transesterification of neem seed oil. Therefore this study aims to synthesise carbon-based catalyst from neem seed residue via sulfonation with concentrated sulfuric acid to produce biodiesel.

MATERIALS AND METHOD

Oil Characterisation

The neem seed oil was sourced after it was extracted mechanically in National Research Centre for Chemical Technology (NARICT), Bassawa-Zaria, Kaduna State Nigeria. The oil was characterised based on the method reported by (Ogunwale, 2015).

Catalyst Preparation and Characterisation

The pretreated neem seed residue was charged into a pyroliser and heated to temperature of 450 °C for 1.5 hr with a heating rate of 4.72 °C per minutes to obtain activated carbon. Distilled water was used to wash the ZnCl₂-impregnated activated carbon to remove the residual zinc and chloride. Further, the neem residue activated carbon (NAC) was dried in oven at 110 °C for 12 hr. The resultant solid was charged into a pyroliser and heated to temperature of 450 °C for 1.5 hr with a heating rate of 4.72 °C per minutes to obtain activated carbon. Distilled water was used to wash the ZnCl₂-impregnated activated carbon to remove the residual zinc and chloride. Further, the neem residue activated carbon (NAC) was dried in oven at 110 °C for 12 hr.

After weighing 10 g of carbonized carbon, 100 ml of concentrated sulfuric acid (H₂SO₄) was added. Under continuous agitation, the sulfonation reaction occurred in a flat-bottom flask that was attached to a hot plate and condenser. After allowing the resulting sulfonated neem activated carbon (SNAC) cool to room temperature, excess sulfuric acid was removed from the SNAC catalysts using distill water until the pH of the wash water matched that of the original distill water. The resulting sulfonic acid functionalized neem activated carbon (FNAC) catalysts were then dried in an oven set at 110 °C for 12 hr. The catalyst was then ground and sieved to a particle size of 150 µm. At temperatures of 160, 180, and 200 °C, the sulfonation process was repeated; however, the 4 hr sulfonation time was

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maintained. The catalysts synthesized were subjected to Brunaur Emmett-Teller (BET) analysis to determine the surface areas, pore volumes and pore sizes.

Catalytic Performance of the Sulfonated catalysts

The catalytic activities of the sulfonated catalysts were evaluated by transesterification of the esterified oil. 33.8 g of neem oil was preheated in a flat bottom flask fitted with a thermocouple and condenser. 1.5% weight of catalyst produced at the sulfonation temperature of 140 ° C was premixed with 10 ml methanol in a glass beaker, stirred, and then added to the preheated oil in the flat bottom flask and heated at 65 ° C for 2 hr under a constant stirring speed of 350 rpm. The reaction temperature of 65 ° C, time of 2 hr, and catalyst weight of 1.5% were selected based on the work of (Inayat et al., 2021). When the transesterification time was completed, the mixture was separated by filtration and purified by washing. The yields of the biodiesel were determined. The biodiesel yield was calculated from Equation 1.

$$\text{Biodiesel yield} = \frac{\text{weight of biodiesel produced}}{\text{weight of oil sample used}} \times 100\% \quad (1)$$

Characterization of Biodiesel

The properties of biodiesel, including viscosity, density, specific gravity, cetane number, and calorific value, were assessed and compared against the standards set by the American Society for Testing Materials (ASTM).

Viscosity Determination

Viscosity was measured using a digital rotational viscometer. A 2.0 ml tin sample container was filled with biodiesel up to 3 mm from the top rim of the depth gauge. The sample was sheared using spindle number 2, with the temperature maintained at 40 °C. The container with the biodiesel sample was positioned in the processing ring on the spindle, and the lever was pulled down to automatically activate the motor. The disc was allowed to run until a steady state was achieved, which took approximately two minutes. After this duration, the viscosity was recorded. Each sample underwent three separate determinations, and the mean value was subsequently calculated.

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Determination of density

The densities of the biodiesel were determined by ASTM method D-129-12b (2017). The sample was brought to a temperature of 40° C, and a test portion was transferred to a density bottle of known weight brought to approximately the same temperature. After temperature equilibrium had been reached, the mass of the biodiesel plus density bottle was taken using analytical weight balance. The mass of the density bottle was then subtracted from the mass of biodiesel plus the density bottle. The mass of the oil was then divided by the volume of the density bottle to determine the density of the biodiesel based on Equation 2.

$$\text{Density (kg/m}^3\text{)} = \frac{\text{mass of biodiesel}}{\text{volume of biodiesel}} \quad (2)$$

Determination of specific gravity

The specific gravity of the biodiesel was calculated based on the formula given in Equation 3

$$\text{Specific gravity} = \frac{\text{density of biodiesel}}{\text{density of water}} \quad (3)$$

The density of water at 25 °C (997.05 kg/m³) and the densities of the biodiesel produced at the various sulfonation temperatures of 140 °C, 160 °C, 180 °C and 200 °C were used to calculate the specific gravities.

Determination of cetane number

The cetane number was calculated based on the Krisnangkura empirical equation, which relates the iodine and saponification values to the cetane number. The correlation is given in Equation 4.

$$\text{Cetane number} = 46.3 + \frac{5458}{SV} - 0.225 \times IV \quad (4)$$

Where IV is the iodine value in mg I₂/100g, and SV is the saponification value in mg KOH/g.

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Determination of calorific value

The calorific value of the biodiesel was estimated based on its relationship between the iodine and saponification values. It was calculated using the empirical Equation 5.

$$\text{Calorific value} = 49.45 - 0.015 \times \text{IV} - 0.015 \times \text{SV} \quad (5)$$

Where IV is the iodine value in mg I₂/100g, and SV is the saponification value in mg KOH/g

RESULTS AND DISCUSSION

Characterisation of the Synthesized Catalysts

The Brunaur Emmett-Teller (BET) of the biochar and the catalysts produced were carried out and the results are presented in Table 1.

Table 1 BET and acid properties of biochar and sulfonated catalysts

Sample	Surface area (m ² /g)	Pore volume (cm ³ /g)	Pore size (nm)
NSB	257.7	0.158	2.11
C140	190.5	0.1120	2.11
C160	204.4	0.1431	2.13
C180	314.2	0.1891	2.12
C200	382.0	0.1392	2.12

Where, NSB represents Neem seed residue biochar. C140 represents sulfonated catalysts at 140 °C, C160 represents sulfonated catalyst at 160 °C, C180 represents sulfonated catalyst at 180°C, and C200 represents sulfonated catalyst at 200 °C.

The parent NSR biochar exhibited a BET surface area of 257.7 m²/g, pore volume of 0.1580 cm³/g and average pore diameter of 2.112 nm. These values fall within the typical range of 200-300 m²/g and narrow micropores (aprox 2nm) often reported for biomass-derived carbon supports (Yusuff et al., 2022). At 140 °C, the BET surface area decreased by 26 % relative to the parent biochar. Pore volume

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dropped by about 29 % while average pore diameter remains constant (2.11 nm). This reduction is consistent with the well-known phenomenon of pore blockage by -SO₃H insertion especially in microporous channels. An eucalyptus bark sulfonated at 150 °C, recorded a BET drop from 217-31 m²/g (Yusuff et al., 2022). At 160 °C, the sulfonated neem seed residue biochar rebound in both BET (204 m²/g) and pore volume (0.143 cm³/g) likely reflects partial removal of tar and volatile surface residues during sulfonation. Literature from ordered mesoporous carbon indicates that between 120-160 °C, surface area often decreases slightly due to functional group coverage (Na et al., 2019).

The sulfonation process using concentrated H₂SO₄ as reagent tends to increase the surface area and pore volume (Ngaosuwan et al., 2016). This was observed in this study at 180 °C and 200 °C with an increase in the surface areas and pore volumes of the catalysts. The surface area increase is attributed to acid etching and micropore formation, aligning with sulfonated rice husk biochar (300-400 m²/g) under the same conditions (Majid et al & panchalbalaji, 2024). The partial drop in pore volume (0.1392 cm³/g) of the catalyst sulfonated at 200 °C suggest partial pore collapse as reported for sulfonated catalyst (Saidiet et al 2023).

Characterisation of the Cude neem Oil

The physicochemical properties of crude neem seed Oil is presented in Table 2

Table 2: Physicochemical Properties of Crude Neem Seed Oil

Property	Neem seed oil	ASTM Standard
pH	6.4	–
Refractive Index	1.464	1.5000
Density (g/cm ³)	0.8687	0.8-0.90
Specific Gravity	0.8882	0.90
Viscosity (mm ² /s)	23.23	25-35
Acid value (mgKOH/g)	8.34	10
Free Fatty Acid	4.17	25MAX
Saponification Value (mgKOH/g)	178.1175	189-198
Iodine Value (gI ₂ /100 g)	123.03	123
Peroxide Value (meq/kg)	7.4	< 9

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The physicochemical properties of the oil are shown in Table 2. The pH value of 6.4 indicates slight acidity, though there is no specific ASTM-recommended value for neem seed oil. The measured density (0.8687 g/cm³) and specific gravity (0.8882) fall within the ASTM standards. However, the viscosity of 23.23 mm²/s is below the specified ASTM range. The acid value of the neem oil is 8.34 mg KOH/g, which aligns with ASTM recommendations. The free fatty acid content, calculated from the acid value, is 4.17 and falls within the ASTM standards. The saponification value was determined to be 178.1175 mgKOH/g, which is lower than the ASTM specification. The iodine value (123.03 mgI₂/100g) and peroxide value (7.4) meet ASTM standards.

Biodiesel Yields

The biodiesel yields based on the four catalysts are presented in Table 3

Table 3: Yields of biodiesel produced at different sulfonated catalysts

catalyst, °C	Biodiesel yield, %
A	84.4
B	88.0
C	90.5
D	95.9

Where,

A represents sulfonated catalyst produced 140 °C,

B represents sulfonated catalyst produced 160 °C

C represents sulfonated catalyst produced 180 °C

D represents sulfonated catalyst produced 200 °C

The biodiesel yields presented in Table 3 were 84.4 %, 88.0 %, 90.5 % and 95.9 % at catalysts sulfonation temperatures of 140 °C, 160 °C, 180 °C and 200 °C respectively. This trend indicates that higher sulfonation temperature leads to increased biodiesel yield. This effect occurs because higher temperatures enhance sulfonic acid incorporation and acid density, thereby improving catalytic performance until an optimal sulfonation temperature is attained (Kumawat & Rokhum, 2022; Zhang et al., 2021). Because sulfonating neem seed biochar

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between 140 and 200 °C increases sulfonic acid absorption, the acid density and catalytic performance rise proportionately to the sulfonation temperature.

The highest percentage yield observed at 200 °C aligns with the findings of (da Luz Corrêa et al., 2020) who studied sulfonated carbon-based catalyst derived from murumuru kernel shell for esterification reaction.

The yield of 95.5 % achieved in this study, exceeds the reported optimal biodiesel yield of 93.9 % from neem seed oil by Inayat et al. (2021) using neem seed residue support catalyst impregnated with KOH but is slightly lower than the 97.6 % obtained from soybean oil using bean seed husk- derived catalyst, as investigated by de Araújo Sobrinho et al. (2025).

Biodiesel Properties

The following biodiesel properties were analysed as shown in Table 4.

Table 4: Properties of biodiesel produced at different sulfonated catalysts versus petro diesel

Catalyst, °C	Properties	A	B	C	D	Biodiesel	Petro diesel
Fuel Standard						ASTM D6751	ASTM D975
Density, kg/m ³		855.0	871.46	864.46	863.54	860-900	820-845
Kinematic viscosity, mm ² /s(@40 °C		2.47	2.26	2.45	2.40	1.9-6.0	1.3-4.1
Specific gravity		0.88	0.89	0.87	0.88	0.86-0.9	0.80-0.85
Saponification number		179	179	179	179	-	-
Iodine value		120.3	126.2	124.1	123	1230Max	-
Cetane number		48.20	52.00	50.10	49.30	48-65	40-45
Calorific value, MJ/kg		44.91	44.91	44.91	44.91	39-41	42-46

Table 4 compares the fuel characteristics of biodiesel with the requirements for petroleum diesel and biodiesel, ASTM D975 and ASTM D6751, respectively.

The density is a critical parameter for biodiesel, ensuring proper fuel injection, combustion, and engine efficiency while serving as a quality control measure. The densities of the biodiesel produced using the four synthesised catalysts were 855.0 kg/m³, 871.46 kg/m³, 864.46 kg/m³ and 863.54 kg/m³. Except for the biodiesel obtained at 140 °C all values fall within ASTM D6751 standards, confirming their suitability for diesel engines. The close similarity among these density values indicates that catalyst sulfonation temperature does not significantly impact biodiesel density. The biodiesels obtained in this study agree with densities

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reported for biodiesel produced from neem seeds oil by (Aransiola, 2012; Inayat et al., 2021; Karmakar et al., 2012).

Besides density, viscosity is another important property of biodiesel that influences engine performance, fuel quality, and stability. Table 3.2 indicates that the viscosities at 40 °C were 2.47 mm²/s, 2.26 mm²/s, 2.45 mm²/s and 2.40 mm²/s, all in close agreement with ASTM D975 specifications for biodiesel. These viscosities exhibit close agreement with the ASTM D6751 and ASTM D975 specifications for biodiesel and petroleum diesel. Like density, viscosity appears unaffected by sulfonation temperature. These results strongly agree with findings on neem seed oil biodiesel by Aransiola et al. (2012). However, other studies have reported differing viscosity values, including 5.3 mm²/s (Dash et al., 2021), 4.99 mm²/s (Taiwo, 2020), and 4.3 mm²/s (Banik et al., 2018). These variations may be attributed to differences in reaction conditions and feedstock parameters.

The specific gravity is also a crucial property of biodiesel in influencing engine performance, compatibility and biodiesel blending with petroleum diesel. From table 3.2, the specific gravities of the biodiesel obtained were 0.88, 0.89, 0.87 and 0.88. The values are within the standard recommended by ASTM D6751 and also agree closely with a value of 0.9 by (Taiwo, 2020).

As shown in Table 4, the cetane numbers are within the ASTM D6751 specification for biodiesel. These values are however, less than the cetane number of 59 for neem oil noticed by Inayat et al. (2021), 55 for cotton seed oil reported by Naemezie (2021), and 76.8 for *citrillus lanatus* as reported by Jauro & Adams (2020). Despite this, the neem seed oil biodiesel demonstrates superior combustion quality (ignition capacity) to the other biodiesel sources examined.

The calorific value is the amount of heat produced by complete combustion of a given mass of a substance. The calorific values obtained by this study are higher than ASTM D6751 specification for biodiesel. A fuel with a higher calorific value has more energy per unit mass or volume than the required amount. However, the calorific values align with the recommended range 42-46 MJ/kg (ASTM D975) for petroleum diesel. Banik et al.(2018), and Dash et al.(2021) reported a lower calorific values of 35.2 MJ/kg and 38.34 MJ/kg respectively from the investigations of production of biodiesel from neem oil.

CONCLUSION

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Four different catalysts were synthesised from neem seed residue. The catalysts were characterised using BET, and the results show that the catalyst at 180 °C had the largest pore volume, 0.1891 (cm³/g), but a lesser surface area compared with the catalyst sulfonated at 200 °C (382 m²/g). Catalytic performance test showed that the catalyst sulfonated at 200 °C gave the highest biodiesel yield of 95.9 %. The fuel properties of the biodiesel meet the ASTM standards. These results establish the viability of sulfonated neem seed residue as a sustainable catalyst for biodiesel production.

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