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Synthesis of Sulphonated Carbon- Base Catalyst from *Detarium senegalense* Shell for Transesterification of *Detarium senegalense* Seed Oil

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Abstract

This study focuses on the synthesis of a sulfonated carbon-based catalyst from *Detarium senegalense* shells for the transesterification of *Detarium senegalense* seed oil. The catalyst was prepared by carbonizing the shells at 700°C for 3 hours, followed by sulfonation with 98% sulphuric acid at 150°C for 3 hours. The Box-Behnken Design (BBD) was used to optimize the catalyst synthesis process, yielding optimal conditions of a solid-to-acid ratio of 1:15(w/v), a reaction time of 2.502 hours, and a temperature of 120.523°C. The performance of the catalyst was evaluated in the transesterification of *Detarium senegalense* seed oil. The synthesized sulfonated solid acid catalysts were characterized by acid-base back titration for the determination of total acid density, Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), Scanning electron microscopy (SEM) and X-ray energy dispersion spectroscopy (EDS). Oil extraction from the seeds was optimized using BBD, with optimal conditions determined as 5 hours, 80.001 mL of solvent, and 15g of substrate. The extracted *Detarium senegalense* seed oil was characterized by its acid value (0.57 mg KOH/g), iodine value (70 g/100 g of I_2), FT-IR, and other physical properties, including density, specific gravity, and kinematic viscosity. The catalyst achieved a 78% conversion of free fatty acids in the transesterification reactions. Kinetic studies revealed an oil extraction rate constant of 0.63 and an activation energy (Ea) of 235. kJ/mol.

Keywords: *Detarium senegalense*, Box-Behnken Design, Sulphonated carbon-based catalyst.

Introduction

Limited oil reservoirs and greenhouse gas emissions are major problems related to the use of fossil fuels. These factors motivate the research for alternative sources of energy [1]. In this context, biodiesel has attracted great attention as a promising alternative to diesel in e recent years. Biodiesel is derived mainly from vegetable oils, animal fat or food oil residues and has desirable features, such as non-toxicity and biodegradability [2]. Compared to petroleum-derived diesel, it has similar physico-chemical properties and shows a favorable combustion emission profile [3].

In addition, biodiesel can be used directly or blended with petroleum diesel. The major economic constrain tin biodiesel production is the high cost of raw materials, [4,5] Thus, acid oils, such as non- edible vegetable oils, waste cooking oil, and others, are viewed as promising raw materials because they are cheaper. Furthermore, waste oils offer several advantages: they do not compete with the food market; the process recycles the waste oils and reduces production costs, increasing the economic competitiveness of biodiesel [6,7].

The transesterification reaction carried out with homogeneous alkaline catalysts is the commonly used technology in the biodiesel industry. However, when oils with high acid values are used with basic catalysts, the production efficiency lowers due to saponification reactions [8]. The use of traditional homogeneous acid catalysts has some disadvantages, such as undesirable side reactions, equipment corrosion and a large amount of wastewater for treatment, increasing the environmental pollution. Thus, heterogeneous acid catalysts are better alternative for transesterification reactions when oils with high acidity are used. Besides, they have the advantage of being non-corrosive and can be easily recovered and reused [9].

Several types of heterogeneous acid catalysts have been investigated, including zeolites, Li *et al*.,[10] sulphated zirconia, [11] anion exchange resin,[12] and heteropoly acids [13]. However, most report problems like low stability, low acid density, and stringent reaction conditions. Contrarily carbon-based catalysts have advantages such as low preparation cost and high catalytic performance, also, they are relatively cheap, widely available and easily functionalized, making them interesting catalysts.

Detarium senegalense is a leguminous tree in the subfamily Detarioideae. Unlike most members of the family, it produces globular fruits, its common names include ditax, ditakh, detar, and tallow tree [14]. It is a medium-sized tree that may grow up to 40m tall [15]. Like many trees in the Detarioideae, they have thick, irregularly placed branches. The trunks of mature trees typically range from 60–100 cm in diameter [16]. The fruit is a globular, dark green drupe with fibrous pulp and a single seed [17].

They are similar to the fruits of the tamarind, which belongs to Tamarindus, another genus in the subfamily Detarioideae [15]. The sweet and sour flavour of the fruit is popular and the shelf life is good because of the hard shell and dry pulp [14].

This study is aimed to synthesis of sulfonated carbon- base catalyst from *Detarium senegalense* shell for transesterification of *Detarium senegalense* seed oil**.**

Materials and Method

Materials

Detarium senegalense shells and seeds were obtained from Gwangwan village in Rano Local Government Area, Kano State. In the sulphonation process, 98% sulphuric acid was used. For the transesterification reaction 99.8% methanol, *Detarium Senegalese* oil extracted from *Detarium*

Senegalese seeds. To determine the physicochemical properties of the extracted *Detarium Senegalese* seed oil and trans esterified *Detarium Senegalese* seed oil Titrando 905 model, SX-150 Petroleum Quality Analyzer, and DV2T Touch Screen Viscometer were used.

Preparation of *Detarium senegalense* Shell

The shells were thoroughly washed with deionized water to remove any dirt or impurities. They were dried in an oven at 60°C for 24 hours. The dried shells were crushed into small pieces using a mortar and pestle. The crushed shells were heated in a furnace at 700°C for 4 hours to produce carbon powder. The resulting carbon powder was washed with ethanol and deionized water to eliminate impurities [18].

Synthesis of Sulphonated Carbon-Based Catalyst (Sulphonated Biochar)

A modified method of Adeyi *et al*. [18] was adopted; 10g of biochar (carbon powder) was placed in a beaker, 50 mL of 98% sulfuric acid was added to the biochar. The mixture was heated at 150°C for 3 hours on a magnetic stirrer at 450 rpm. After 3 hours, the mixture was quenched in 200 mL of deionized water. The resulting sulfonated biochar was washed with deionized water until the pH of the wash was neutral. The sulphonated biochar was dried in an oven at 60°C for 24 hours.

Characterization of the Synthesized Sulphonated Biochar

The total acid density of the sulphonated biochar was determined by acid–base back titration Bora *et al*, [19] 0.1g of the sulphonated biochar was dispersed in 20 mL of 0.1 M NaOH solution and agitated for 1 hour. The mixture was centrifuged, and the supernatant was titrated with 0.1 M HCl using phenolphthalein as an indicator. The acid site/strength was determined using equations (1,2,3 and 4).

$$Moles\ of\ HCl = \frac{Molarity\ of\ HCl\ \times volume\ of\ HCl(ml)}{1000} - - - - - - - - - - - - - -(1)$$

$$Moles\ of\ NaOH = \frac{Molarity\ of\ NaOH\ \times volume\ of\ NaOH(ml)}{1000} - - - - - - - - -(2)$$

$$Moles\ of\ HCl\ reacted = Moles\ of\ HCl\ used - Moles\ of\ NaOH\ used - - - - - -(3)$$

$$Acidsite/Acid\ Strength(mmolg^{-1}) = \frac{Moles\ of\ HCl\ reacted\ \times 1000}{Weigth\ of\ sample} - - - -(4)$$

Response surface Methodology (RSM) with a three-level factorial Box-Behnken Design (BBD) of design Expert software version 13.0.5.0 was used to optimize three independent parameters: Substrate amount (X_1) 5–15 g, Extraction time (X_2): 1–5hours, Solvent volume (X_3): 80–160 ml. The software gave fifteen experiments as shown in Table 1. The percentage yield and conversion of free fatty acid was determined using equation (5 and 6).

$$\text{Yield (\%)} = \frac{\text{Weight of suphonted biochar produce}}{\text{Weight of biochar used}} \times 100 - - - - - - - - - - - - (5)$$

$$FFA\ Conversion(\%) = 1 - \frac{AV_f}{AV_i}\ x\ 100 - (6)$$

where AV_i is the initial acid value (fatty acid) and AV_f is the final acid value after the fatty acid transesterification reaction, given in mg KOH per g [20].

X-ray Diffraction (XRD) was Used to determine the crystal structure of the sulphonated biochar. Scanning Electron Microscopy (SEM) was Used to examine surface morphology. Energy Dispersive X-ray Spectroscopy (EDS) was Used for elemental composition analysis. Fourier Transform Infrared Spectroscopy (FT-IR) was Used to confirm the presence of sulphonic acid functional groups

Results and Discussion

Table 1: Acid Density of Sulphonated Biochar/Biochar

Sample	Total acid density (mmol/g)	-SO_3H acid density (mmol/g)
Biochar	0.26	-
Sulfonated Biochar	6.50	6.34

The concentrations of –SO_3H, -OH, and -COOH groups in BC-SO_3H typically range from 0.1 to 4.9 mmol/g, 0 to 0.9 mmol/g, and 0 to 7.8 mmol/g, respectively [20]. While –SO_3H groups are identified as the primary active acidic sites, the presence of -OH and -COOH groups enhances the accessibility of hydrophilic reactants to the –SO_3H groups, thereby improving catalytic effectiveness. Acid-base back titration results indicate that biochar has a lower density of acid sites (0.26 mmolg^{-1}) compared to sulfonated biochar (6.34 mmolg^{-1}), as shown in Table 1 with 78% conversion of FFA.

Table 2: Response (Rate of Sulphonated carbon-based Yield) from the Experimental Design

Ru/ns	A(w/v)	B(hours)	C (degree Celsius)	Yield (%)	**Actual Value**	**Predicted Value**	**Residual**
1	1:10	1	150	25.5	**25.50**	**25.50**	**0.0000**
2	1:15	2	150	24	24.00	24.10	-0.1000
3	1:5	2	120	20.7	20.70	21.40	-0.7000
4	1:10	2	135	23.5	23.50	24.93	-1.43
5	1:5	2	150	20.6	**20.60**	**20.60**	**0.0000**
6	1:10	2	150	25.7	**25.70**	**25.70**	**0.0000**
7	1:5	2	120	22.1	22.10	21.40	0.7000
8	1:15	2	150	24.2	24.20	24.10	0.1000
9	1:5	1	135	20.5	**20.50**	**20.50**	**0.0000**
10	1:15	3	135	24.5	24.50	24.75	-0.2500
11	1:10	2	135	25.7	25.70	24.93	0.7667
12	1:10	2	135	25.6	25.60	24.93	0.6667
13	1:15	3	135	25	25.00	24.75	0.2500
14	1:10	1	120	25.5	**25.50**	**25.50**	**0.0000**
15	1:10	3	150	25.5	**25.50**	**25.50**	**0.0000**

Table 3: Fit summary statistic

Source	**Sequential p-value**	**Lack of Fit p-value**	**Adjusted R^2**	**Predicted R^2**	
Linear	0.0804	0.0457	0.2936	-0.0847	
2FI	0.2694	0.0426	0.3896	-0.5910	
Quadratic	**0.0426**		**0.7848**		**Suggested**
Cubic					**Aliased**

Table 4: Model summary statistic

Source	**Std. Dev.**	**R^2**	**Adjusted R^2**	**Predicted R^2**	**PRESS**	
Linear	1.66	0.4450	0.2936	-0.0847	59.45	
2FI	1.55	0.6512	0.3896	-0.5910	87.20	
Quadratic	**0.9178**	**0.9232**	**0.7848**		*****	**Suggested**
Cubic					*	Aliased

Table 5: ANOVA for quadratic model

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	50.60	9	5.62	6.67	0.0251	Significant
A-solid: acid ratio	10.73	1	10.73	12.74	0.0160	
B-time	0.5339	1	0.5339	0.6338	0.4621	
C-temperature	0.5339	1	0.5339	0.6338	0.4621	
AB	0.2761	1	0.2761	0.3278	0.5917	
AC	0.0003	1	0.0003	0.0003	0.9858	
BC	0.2826	1	0.2826	0.3356	0.5875	
A^2	8.16	1	8.16	9.69	0.0265	
B^2	0.0267	1	0.0267	0.0317	0.8658	
C^2	0.8250	1	0.8250	0.9795	0.3678	
Pure Error	4.21	5	0.8423			
Cor Total	54.81	14				

Table 6: Fit Statistics

Std. Dev.	0.9178	**R^2**	0.9232
Mean	23.91	**Adjusted R^2**	0.7848
C.V. %	3.84	**Predicted R^2**	NA(1)
		Adeq Precision	6.9392

(1) Case(s) with leverage of 1.0000: Pred R^2 and PRESS statistic not defined.

Table 7: Optimization result and validation of the Sulphonated carbon-based yield

Number	Solid: acid ratio	Time	Temperature	Yield	Desirability	
1	**1:15.000**	**2.502**	**120.523**	**25.714**	**1.000**	**Selected**
2	1:15.000	2.530	120.279	25.808	1.000	
3	1:15.000	2.677	122.280	25.703	1.000	
4	1:15.000	2.586	120.833	25.805	1.000	
5	1:15.000	2.972	125.025	25.725	1.000	
6	1:15.000	2.917	121.037	26.327	1.000	
7	1:15.000	2.587	120.360	25.896	1.000	

Response Surface Methodology (RSM) using a Box-Behnken Design (BBD) was employed to enhance the yield of sulphonated carbon biochar (SCB) synthesis. This approach involved 15 experimental runs to evaluate the statistical significance of various parameters, including the solid/acid ratio (A), extraction time (B), and temperature (C) on SCB yield, as detailed in Table 2. The results indicated that varying levels of these parameters produced different SCB yields, suggesting that the selected parameter levels were suitable for optimizing SCB yield. The obtained SCB yields were fitted to a quadratic model equation. Consequently, the predicted model equation was developed based on the regression coefficients, expressed in terms of coded parameters while excluding nonsignificant terms, as follows in Eq. (7):

$$\begin{aligned}\text{yield} &= 24.93 + 1.74\text{A} + 0.3875\text{B} - 0.3875\text{C} \\ &+ 1.24\text{AB} \\ &+ 0.0125\text{AC} - 0.3875\text{BC} - 3.35\text{A}^2 - 0.2000\text{B}^2 \\ &+ 1.15\text{C}^2 \cdots\cdots\cdots\cdots\cdots\cdots (7)\end{aligned}$$

Where: Y is the predicted value of the dependent variable (SCB yield). The coefficients of A, B, and C are the main linear effects of the independent process variables; solid acid/solvent ratio, time and temperature respectively, in coded units. AB, AC, and BC represent the linear interaction effects between solid acid/solvent ratio, time and temperature respectively, while A^2, B^2, C^2, are the quadratic effects of the respective process variables (solid acid/solvent ratio, time and temperature). This equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. The equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. From the table 3, Model **F-value** of 6.67

implies the model is significant with p-value (0.0251) <0.05. There is only a 2.51% chance that an F-value this large could occur due to noise for SCB extraction. **P-values** less than 0.0500 indicate model terms are significant. In this case A, A^2 are the significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The **Lack of Fit F-value** of 0.0457 and p-value 0.0804 for SCB synthesis implies the Lack of Fit is not significant relative to the pure error. There is an 8.04% chance that a Lack of Fit F-value this large could occur due to noise. Since the model is required to fit, a non-significant lack of fit is good. **Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Table 6 ratio of 6.939 indicates an adequate signal. This model can be used to navigate the design space. Based on solving the predicted polynomial model using Design-Expert software version 13.0.5.0, optimal parameters for SCB extraction were determined: a solid acid ratio of 1:15, an extraction time of approximately 2.502 hours, and a temperature of about 120.532°C, resulting in a predicted yield of approximately 25.714%. Further triplicate experiments under these optimal conditions yielded an average oil yield of approximately 25.650%. This validation confirms that the model is appropriate for predicting.

Effect of parameters on the yield

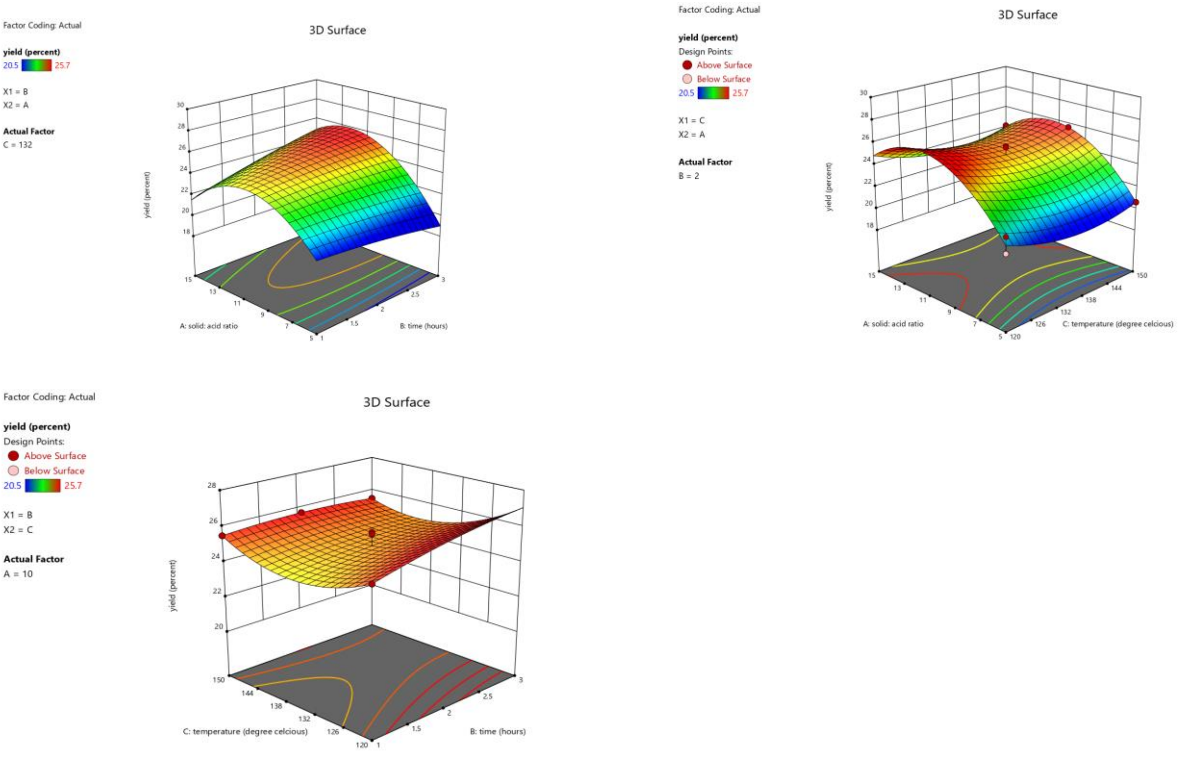

Figure 1 :3D plot for the effect of time, solid/acid ratio, and temperature

From figure 1 it is evident that the sulfonation process is dependent on time but significantly influenced by temperature and solid/acid ratio, corroborating findings by Zhang *et al.*, (2015) and Niu *et al.*, (2018).

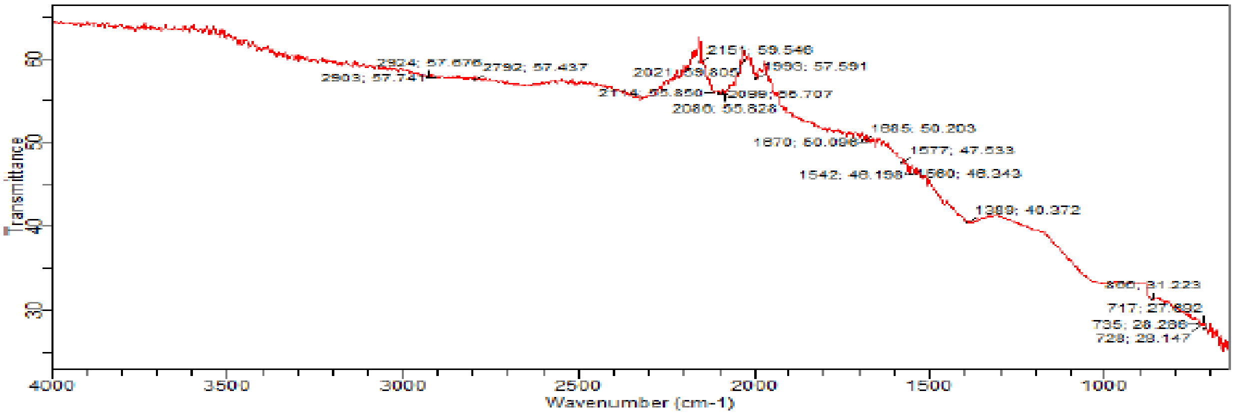

Figure 2A: FTIR Spectra of biochar derived from *Detarium Senegalense.*

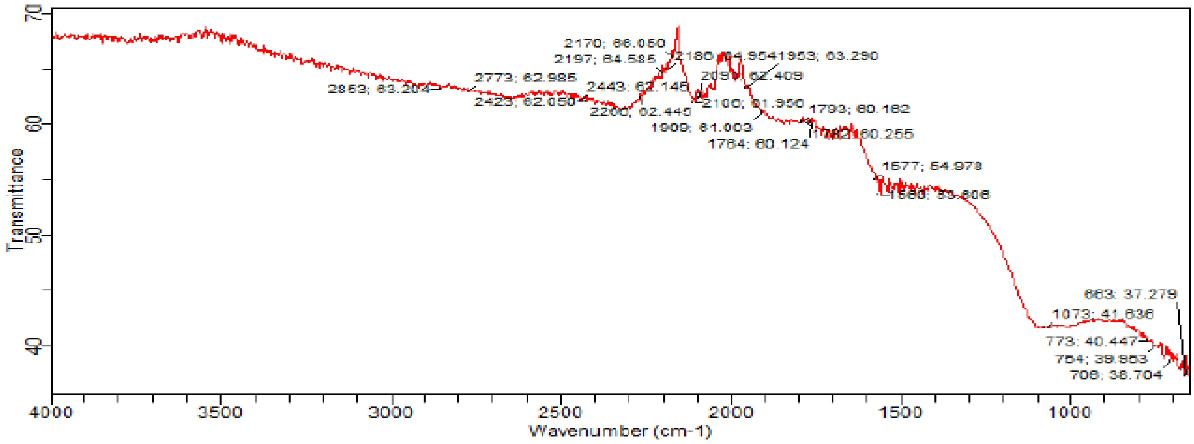

Figure 2B: Sulphonated biochar (SBC) derived from *Detarium Senegalense*

From Figure 2A the peak at 2924-2903 cm^{-1} (C-H Stretch) Indicates the presence of aliphatic hydrocarbons,2792 cm^{-1} (Aldehyde C-H Stretch) Suggests the presence of aldehyde groups.2114-2086cm^{-1} (Alkyne C≡C Stretch) Reflects unsaturation in the carbon structure 1993 cm^{-1} (Isothiocyanate N=C=S Stretch) Indicates potential nitrogen and sulphur interactions.1685-1670 cm^{-1} (C=C Stretch) Shows unsaturation typical in carbonaceous materials.1577-1560 cm^{-1} (Aromatic C-C Stretch) Confirms stable aromatic structures.1542 cm−1 (Nitro Compound N-O Stretch): Indicates nitrogen-containing groups.1389 cm^{-1} (C-H Bend): Further confirms aliphatic hydrocarbons.

From Figure 2B the peak at 2853 cm^{-1} (O-H Stretch): Indicates hydroxyl groups for carboxylic, 2773cm^{-1} (O-H Stretch): Another indication of hydroxyl for phenol presence, 2443-2423 cm^{-1} (S-H Stretch): evidence of thiol groups introduced by sulphur functionalization.2266 cm^{-1} (C≡N Stretch): Suggests additional nitrogen functionalities.2197-2106 cm^{-1} (Alkyne C≡C Stretch).2091 cm^{-1} (Isothiocyanate N=C=S Stretch): Indicates sulfur incorporation through isothiocyanate groups.1953-1909 cm^{-1} (Aromatic C-H Stretch): Shows stability of aromatic structures despite modifications.1793, 1782, 1764 cm^{-1} (C=O Stretch): Peaks indicating carbonyl functionalities from sulfonation.

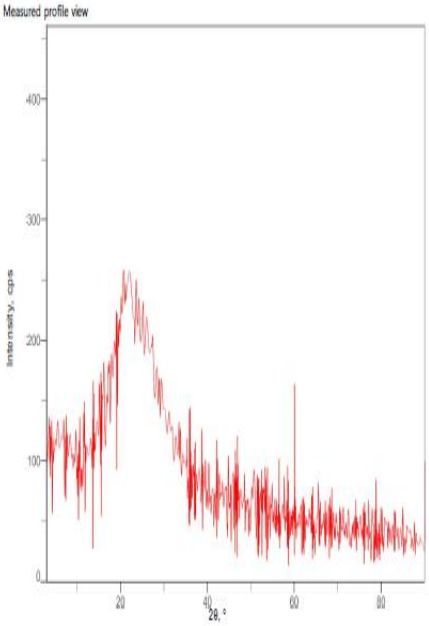

Figure 3a: XDR of Biochar

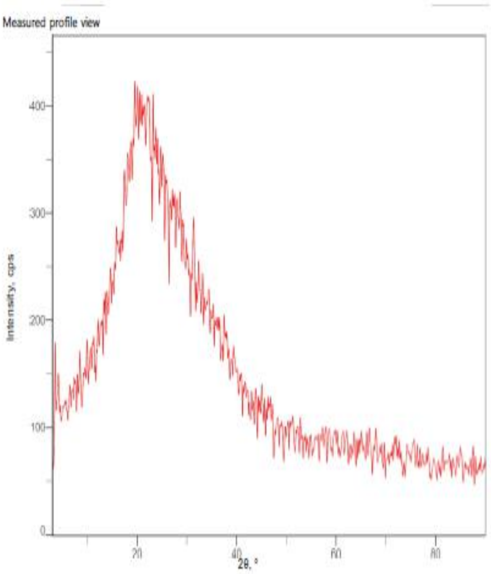

Figure 3b: XDR of Sulfonated Biochar

Figure 3a shows that D*etarium senegalense* Biochar; Displays layered structures with ordered crystalline regions. Largest d-spacing (~15.8 Å) reflects a layered biochar material. Peaks at 2θ = 21.05° (d ≈ 4.22 Å); Indicates graphitic or semi-graphitic content. Peak at 2θ = 74.7°; Highest normalized intensity, signifying highly oriented crystalline region. Figure 3b XDR of Sulfonated Biochar (SBC); indicate that Sulfonation introduces structural expansion (increased interlayer spacings) and enhances crystallinity. Peaks at: - 2θ = 15.54° (d ≈5.70 Å), 24.01° (d ≈ 3.71 Å), and 74.43° (d ≈ 1.27 Å): Show increased intensity and sharpness, indicating improved order and potential incorporation of sulfonic acid moieties. 2θ = 77.94° (d ≈ 1.23 Å): Signifies highly ordered domains formed post sulfonation.

SEM micrographs of Biochar And sulfonated Biochar

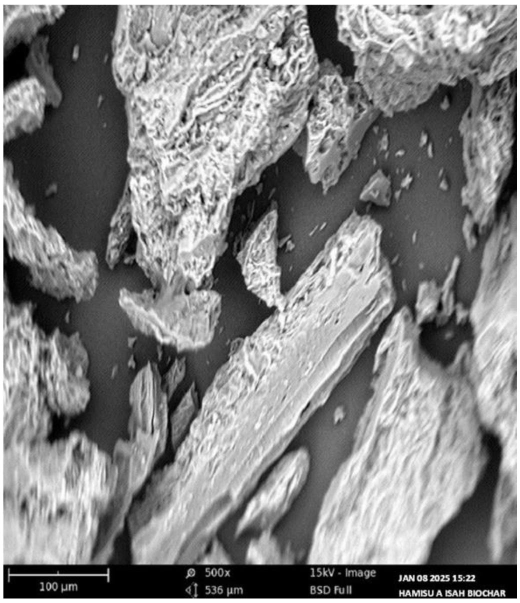

Fig. 4a

Fig. 4b

Fig. 4a presents SEM images of biochar and fig. 4b presents SEM image of sulphonated biochar, biochar in figure 4a display an irregular and heterogeneous surface with a prominent porous structure, consistent with carbonized organic waste [21]. In contrast, the SEM images of the sulphonated biochar Fig 4b) show reduced porosity, likely due to small cracks, oxidation, condensation, and partial destruction of the porous structure caused by the sulfonation process [3]. The analysis suggests that the partial blockage of pores is a result of -SO_3H group adsorption on the biochar, indicating successful sulfonation [19].

EDS analysis of Biochar And sulfonated Biochar

The EDS analysis reveals that both materials have a lot of carbon (Fig. 5a), which comes from the pyrolysis process. After the sulfonation process, the sample has 10.84% sulfur (Fig. 5b), showing that sulfonic groups were added successfully. Also, the oxygen content in the biochar rose from 32.73% to 36.31% after functionalization, indicating that using concentrated sulfuric acid for sulfonation helps create more oxygenated groups.

Table 8: Elemental composition of Biochar

Element Number	Element Symbol	Element Name	Atomic Conc.
6	C	Carbon	54.12
8	O	Oxygen	32.73
14	Si	Silicon	10.02
19	K	Potassium	0.29
26	Fe	Iron	0.29
20	Ca	Calcium	1.52
11	Na	Sodium	0.84
12	Mg	Magnesium	0.19

Table 9: Elemental composition of Sulfonated Biochar (SBC)

Element Number	Element Symbol	Element Name	Atomic Conc.
6	C	Carbon	46.50
8	O	Oxygen	36.31
14	Si	Silicon	2.02
19	K	Potassium	0.36
26	Fe	Iron	0.11
20	Ca	Calcium	2.99
16	S	Sulfur	10.84
12	Mg	Magnesium	0.87

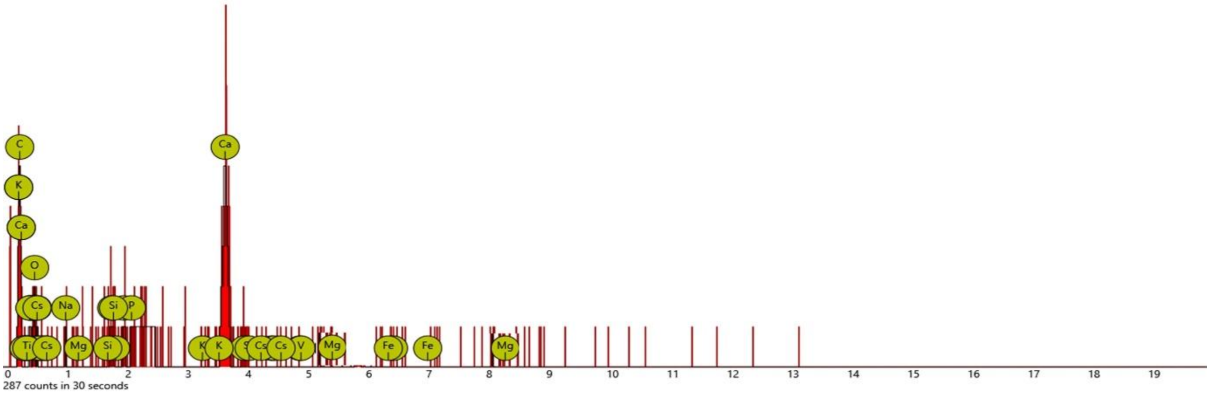

Figure. 5a: EDS Spectra of biochar derived from *Detarium Senegalense.*

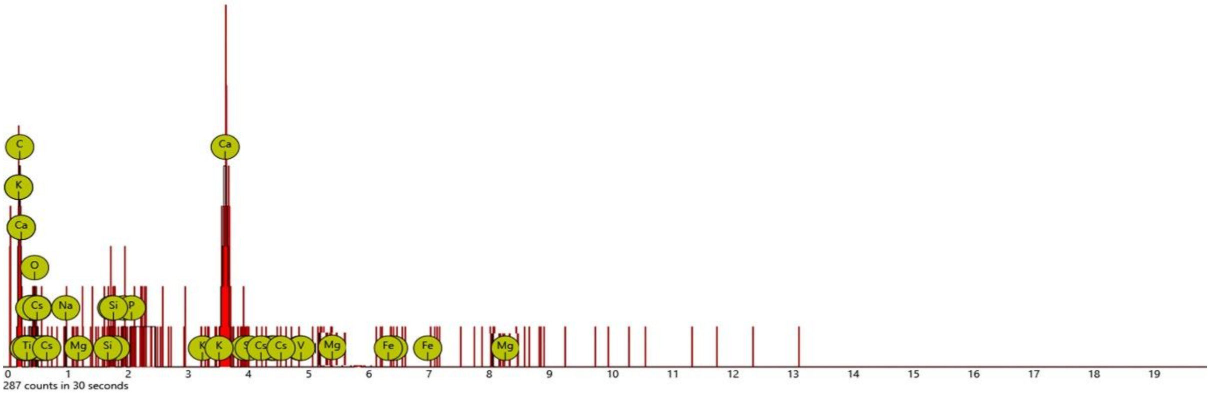

Figure. 5b: EDS Spectra of Sulfonated biochar (SBC) derived from *Detarium Senegalense.*

Conclusion

The study successfully demonstrated the synthesis of a sulfonated carbon-based solid acid catalyst from *Detarium Senegalense* shell, a bio-waste material. The catalyst showed excellent properties for heterogeneous catalysis and proved to be a cost-effective alternative to commercial catalysts. The catalyst was applied to transesterification, and the reaction parameters were optimized using the Box-Behnken Design (BBD) of RSM. Optimum reaction conditions for catalyst synthesis were determined to be a solid/acid ratio of 1:15, reaction time of 180 minutes, and temperature of 120.5°C, yielding 25.1% conversion. Temperature was identified as the most significant reaction parameter, followed by solid/acid ratio and time. The sulphonated carbon-based catalyst can significantly contribute to sustainable transesterification processes, reducing reliance on expensive catalytic materials.

Conflicts of interest

There are no conflicts of interest to declare

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