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Renewable diesel synthesis from sesame indicum (bene) seed oil using novel heterogeneous biocatalyst derived from the *Chrysophyllum albidium* seed coat

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ABSTRACT

The synthesis, characterization, optimization and oxidation stability improvement of biodiesel from the seed oil of *sesame indicum* using a novel nano-heterogeneous bio-catalyst derived from the *Chrysophyllum albidium* seed coat (CASC) is reported. The heterogeneous catalyst was produced by calcination and activation at 400 °C, 600 °C and 800 °C using acetic acid. The catalyst was characterized using scanning electron monograph (SEM), Fourier transform infrared (FTIR), and x-ray diffraction (XRD). The seed oil was extracted using mechanical press milling and the biodiesel produced were characterized using AOAC 2019 edition and ASTM D-6151, Fourier transform infrared (FTIR) and gas chromatography (GC) methods. The results indicated that calcined *Chrysophyllum albidum* seed coat contains nano-particles and alkaline elements (75 % graphite). The calcination process improved the size reduction and structural arrangement of the particles. The 600 °C calcination temperature had the highest biodiesel yield of 88 % at 3.0 wt % catalyst concentration, 12:1 M ratio of alcohol to oil and 500 rpm. The reusability of catalyst indicated 71.50 % after fifth (5th) cycle. After 28 days storage duration in the presence of the natural, renewable and eco-friendly antioxidant (1 % turmeric), the oxidation instability of the produced biodiesel was reduced by 50 %. The quality of the biodiesel indicates agreement with standards and literature as well as high potential for effective application in diesel engine.

1. Introduction

Efforts to proffer range of secure and sustainable solutions to overcome the adverse effects that arise from usage of petroleum products as energy sources, and future challenges which could result from their scarcity has continued to pave way for the development of more sustainable alternatives that are renewable and also have less negative environmental impacts. Biofuels including biodiesel is at the centre of the researches to realize the above success since they are obtained from renewable resources. The best method for the utilization of feedstocks in biofuel production and prevent food-fuel crises is through the usage of agro-waste [1]. To a

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Fig. 1. a. The seed, b. The seed coat, c. The ground seed coat, d. seed coat nano-catalyst.

reasonable extent, this has been achieved through the application of animal fats, waste oils and non-edible oils as sources of triglycerides and feedstocks in biodiesel production [2]. However, little efforts have been made in achieving the above cost-effective approach in the application of catalysts for transesterification reaction. Before now, only few efforts have been drawn towards the application of heterogeneous catalysts. Consequently, the research drive by researchers is recently towards the utilization of heterogeneous catalyst [3]. This will definitely complement the efforts already put together by researchers in the application of waste oils and animal fats for biodiesel production.

Heterogeneous catalyst application is a great breakthrough on the challenges of the utilizing homogeneous catalyst due to an overwhelming challenge of emulsification, more energy requirement, generation of excess waste, etc. [4]. Interestingly, heterogeneous catalysis offers a lot of hope as a viable and feasible route due to its characteristics of easy separation, non-corrosiveness, very low sensitivity, environmental friendliness, none-soap formation and high stability. Above all, heterogeneous catalysts have a unique characteristic of reusability [3]. Although some heterogeneous catalysts have recently been sourced from agro-waste (animal and plant) such as animal bones and shells [5], fruit peels and husks [6], trunks and stems [7]. Yet many agro-wastes are yet to be researched on to ascertain their viability and feasibility as heterogeneous catalysts for biodiesel production. Breakthroughs in this area will definitely reduce concentration on food-based feedstocks, overreliance on synthetic and none eco-friendly catalysts and minimize the biodiesel production cost. One of them is *Chrysophyllum albidium* seed shell (or coat). The viability of the seed oil for biodiesel production and the fruit pulp of *Chrysophyllum albidium* seed for bioethanol, have been recently reported [7,8]. However, its seed shell is still been regarded as a waste and have not been found to be of any industrial usefulness. However, there is a great need to assess the viability of the application of the seed coat as a heterogeneous catalyst in biodiesel production using an oil feedstock of established viability such as *sesame indicum seed* oil.

Sesame indicum (Bene seed) is considered to be a crop of great importance due to its high oil content of about 51 % [6]. Bene seed is used as raw material in the manufacture of paints, black ink, margarine, soap and pharmaceuticals. When compared with other vegetable oils, bene seed oil is fairly resistant to oxidative deterioration under good storage conditions. More so, it does not easily turn rancid in hot climates [9]. Based on these properties, bene seed is considered as a potential source of vegetable oil for large scale biodiesel production. Consequently, many researchers have produced biodiesel from bene seed oil but the extensive literature proves that reports on the application of heterogeneous biocatalyst in its seed oil transesterification is conspicuously lacking. Most of the catalyst previously applied are sodium methoxide [10], sodium hydroxide [11,12], and $Ba(OH)_2$ [13]. Therefore, the application of bio-heterogeneous catalyst from the seed coat of *Chrysophyllium albidum* will not only be a first report on this regard but will promote the viability, feasibility and renewability of the biodiesel production process using bene seed.

It is equally worthy of note that the fatty acid composition of bene seed oil among different varieties worldwide is only slightly affected by genotype, agro-climatic conditions, and stages of ripening. However, fatty acids of the oil were found to mainly comprise of unsaturated fatty acids such as: oleic acid (C18:1, 33–54 %), linoleic acid (C18:2, 35–59 %), palmitic acid (C16:0, 8–17 %) and stearic acid (C18:0, 3–9%) [9]. It implies that the bene seed oil requires some level of oxidation stability modifications to maximize its

Table 1
Sesame indicum seed oil biodiesel (SISOB) physico-chemical characterization methods.

Sn	Fuel property	Method
1	Moisture	AOAC-920
2	Kinematic viscosity	ASTMD-246
3	Specific gravity	AOAC 920.212
4	Iodine value	AOAC 920:159
5	Refractive index	AOAC 921.08
6	Pour	ASTM D-97
7	Cloud	ASTM D-93
8	Flash point	ASTMD-2500b
9	Acid value	ASTM D-664
10	Peroxide value	AOAC 965:133

application as a feedstock for biodiesel production. this aspect of research is lacking in the reviewed literature. It is equally very important to realize that although, natural antioxidants have been reported to be biodegradable, non-toxic and more effective than synthetic antioxidants [14], yet it is recently reported that their applications in biodiesel processing is rare [15]. Interestingly, turmeric (Curcuma longa L.) is a highly potential bio-antioxidant due to its high phenolic content. Curcumin as shown in Sketch 1 is the main curcuminoids (59–70 %) in turmeric [16]. This has given it successful applications in many chemical processes as a viable renewable bio-antioxidant. Although, turmeric has been shown to increase the induction period of soy bean biodiesel by 83 % [14], and cotton seed biodiesel [16], many researchers have identified that its application in biodiesel synthesis is rarely reported [14.17].

Consequently, it is the objective of this research to investigate on the application of the seed coat of *Chrysophyllum albidium* as a biocatalyst using *sesame indicum* (benne) seed oil and turmeric as a natural antioxidant. The utilization of bene seed oil as source of glyceride and *Chrysophyllum albidium* seed coat as biocatalyst would globally lead to the availability of commercial quantity and quality biodiesel that do not require further modifications of the diesel engines.

2. Materials and methods

2.1. Materials

2.1.1. Reagents

Reagents used for this study were analytical grade reagents hence, there were no need for any purification.

2.1.2. Catalyst

2.1.2.1. Catalyst collection and size reduction. Modified African star apple waste seed coat (*Chrysophyllum albidium*) was used as catalyst. Its preparation and characterization follow the methods previously reported in the literature [17]. It was thermally activated with acetic acid solution. Bulk seed (Fig. 1a), was carefully cracked to obtained coat of *Chrysophyllum albidium* seed which before being washed and dried in open air. It was later dried in hot air oven at 60 °C for 12 h. The dried seed waste coat (Fig. 1b) was ground using laboratory blender to smooth particle size of 250 μm (Fig. 1c).

2.1.2.2. Treatment with acetic acid solution. Acid activation is commonly a chemical modification of biomass with a hot solution of mineral acid.

Recently some researchers have proven acetic acid a better activation agent than other known agents like $CaCl_2$ and $ZnCl_2$ [18] and in modifying activated carbon for wastewater treatment and separation of CO_2/CH_4 [19]. A known weight of 500 g of the sample was weighed into a 2 L beaker and submerged with 1 L of 10 % acetic acid solution. The mixture was properly homogenized with stirring rode and placed in water bath at 90 °C for 2 h. After the heat treatment, the sample was allowed to drain and subsequently dried in laboratory hot air oven at 120 °C for 24 h before being pulverized prior to activation.

2.1.2.3. Carbonization. The acetic acid treated sample was loaded into porcelain crucibles and fitted with lid to make it air tight and carefully placed in muffle furnace. Samples were carbonized at different temperature regimes (400 °C, 600 °C and 800 °C) each for 4 h. The choice of the variables values is based on preliminary reports from the literature [19, 20, 21]. Each time after carbonization, samples were allowed to stay over-night inside the heating chamber of the furnace to cool properly. The carbonized samples, now catalyst (Fig. 1c), were stored in air tight containers for use and labeled accordingly.

2.1.2.4. .4characterization of the catalyst. Fourier transform infrared (FTIR) spectrometry was done to elucidate available functional groups using Agilent Cary 630" Fourier transform infra-red (FTIR).X-ray diffraction (XRD) was done using Miniflex 600 by Rigaku cooperation Japan model X-ray diffraction was used to ascertain the nature of the synthesized catalyst. Scanning electron monograph (SEM) analysis was carried to ascertain the surface morphology of the raw biocatalyst material and the activated *Chrysophyllum albidum* seed coat catalyst were viewed under a high resolution Phenom ProX by phenom World Eindhovan the Netherlands.

2.2. Oil extraction and characterization

2.2.1. Oil extraction

The seeds of *sesame indicum* were obtained from Nkwagu market in Ikwo, Ebonyi state, Nigeria. The seeds were carefully sorted and winnowed to remove impurities and unwanted materials. It was latter sun dried for 3 days before being subjected to oil extraction by electro-mechanical press using Wenzhou Hongkuo tech co., Ltd mechanical extraction machine model ZY-22B in the Unit operation laboratory of the Chemical Engineering Department of Alex Ekwueme Federal University Ndufu Alike Ikwo Ebonyi state of Nigeria.

2.2.2. Determination of Sesame indicum seed oil (SISO) and Sesame indicum seed oil biodiesel (SISOB) fuel properties

Table 1 contains the fuel properties and the methods used in determining SISO and SISOB fuel properties [20]. The experiments were conducted in triplicates and the average results with their standard deviations were recorded. A U-tube viscometer manufactured by Poulten Selfe and Lee Ltd (PSL ASTM-IP 350) was used to determine the kinematic viscosity while the acid value was determined using ASTM methods. The API gravity was determined from the value of the specific gravity of the product (Equation (1a)) while Diesel index (Equation (1b)) and cetane number determined according to Rajarshi et al. [21], and AOAC [22], (Equation (1c)).

$$API\ gravity = \frac{141.5}{specific\ gravity\ at 60℉} - 131.5 \tag{1a}$$

$$Diesel\ index = \frac{Aniline\ point(℉) x\ API\ gravity}{100} \tag{1b}$$

$$Cetane\ number = diesel\ index - 3 \tag{1c}$$

2.3. Determination of functional group and fatty acids in SISO and SISOB

Fourier transforms infra-red (FT-IR) IR Affinity-1 Shimadzu, model No: 3,116,465 was used to ascertain the functional groups present in SISO and SISOB. It has a maximum resolution of 0.5 cm^{-1} between 400 cm^{-1}- 4000 cm^{-1} region and attached with microlab software. The spectra peaks obtained were identified and interpreted to identify the functional groups in the molecules of the SISO and SISOB in accordance with [23].

The fatty acid constituent of the SISOB was determined using GC on PerkinElmer Claurus 600 model FID in agreement with AOAC official method Ce2-66 [24.26]. The peaks were identified by comparing their retention time with Mass Spectra Library [24]. The amount of methyl ester in percentage as contained in SISOB was ascertained using Equation (2) [25].

$$C = \frac{\Sigma_A - A_{IS}}{A_{IS}} \times \frac{M_{IS}}{M} \times 100 \tag{2}$$

Where.

Σ_A = Summed area under peak for fatty acids C_6– C_{14},(μV/sec).
A_{IS} = Area under thri-internal standard peak,(μV*sec).
M_{IS} = weight of applied internal standard (mg)

M = weight of HSISOB sample (mg).

2.4. Effect of catalyst dosage and calcination temperature on biodiesel yield

For the three activated catalyst forms (400 °C, 600 °C and 800 °C), the effect of catalyst loading on biodiesel yield was studied. As the loading weight is varied at 1 %, 2 % and 3 % weight of sample, other prevailing parameters are kept constant as follows; time is 2 h, temperature 60 °C, methanol and oil mole ratio is 12:1 and agitation speed is 500 rpm. All the process parameters were kept constant to study the effect of the diverse activation temperature treatment (400 °C, 600 °C and 800 °C), given to the catalyst as it affects biodiesel yield. Process conditions were kept constant as follows; time is 2 h, temperature 60 °C, methanol and oil mole ratio is 12:1 and agitation speed is 500 rpm, catalyst loading is 3 % weight of sample.

2.5. Catalyst reusability

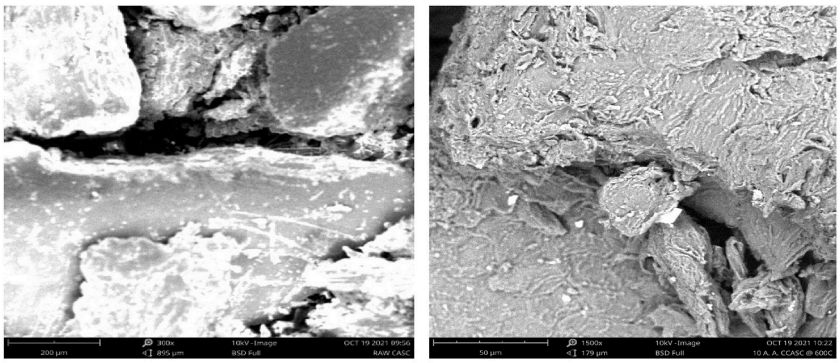
The three catalysts were studied for reusability efficiency. All process conditions were kept constant as catalysts were reused in five cycles (five times). After each production, catalyst is collected by filtration, washed with distilled water and lastly ethanol. It is allowed to dry before reuse.

2.6. Effect of antioxidant on biodiesel yield

The effect of antioxidant on the yield and oxidation stability of the biodiesel from *sesame indicum* seed oil was monitored using 1 %

Table 2
Sesame indicum seed oil properties.

S/N	Properties	Value	Unit
1	Oil yield	43.7 ± 1.41	mgKOH/kg
	Acid value	0.5436 ± 0.25	
2	Free fatty acids	0.2718 ± 0.23	mgKOH/kg
3	Saponification value	167.96 ± 3.26	mgKOH/kg
4	Iodine value	51.35 ± 0.76	Mg/100 g
5	Moisture content	0.04 ± 0.02	% (w/w).
6	Refractive index @29 °C	1.4706 ± 0.003	–
7	Peroxide value	1.40 ± 0.02	Meq/kg
10	Density	0.983 ± 0.007	g/cm^3
11	Kinematic viscosity @ 40 °C	5.063 ± 0.33	(mm^2/s)
12	Molecular weight	1005.27 ± 0.002	g/mol
13	Ester value	99.68 ± 0.21	%



a. b.

Fig. 2. SEM diagram of (a). RCASC catalyst powder (b). ACASC catalyst powder.

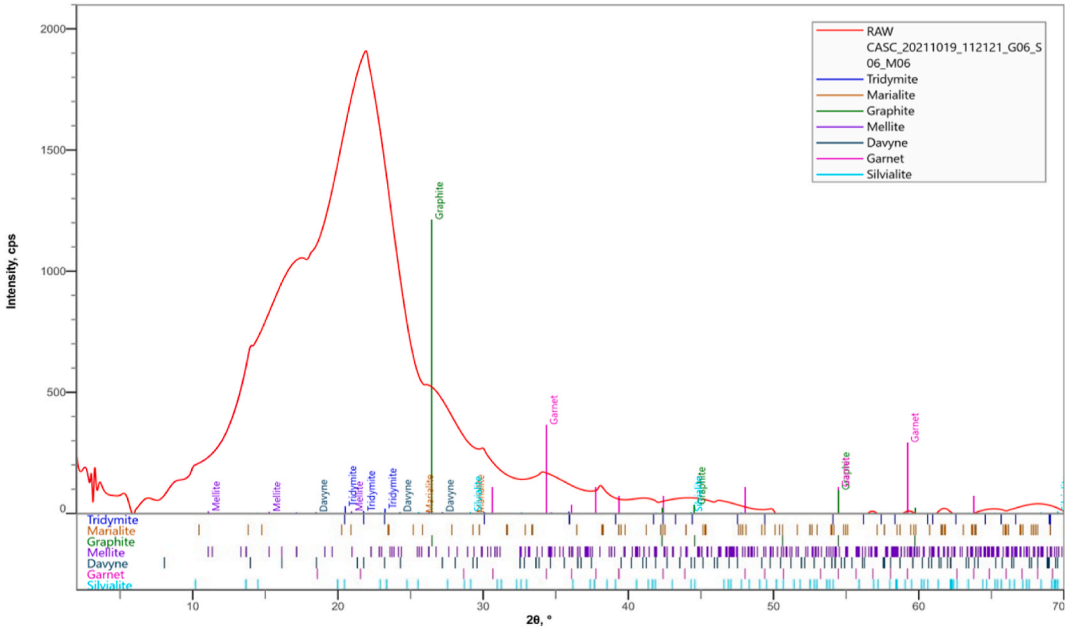
turmeric extract. The change in peroxide value of the biodiesel as an index of rancidity over time was studied for 28 days at room temperature for the three calcinations temperatures of the catalyst.

3. Results and discussions

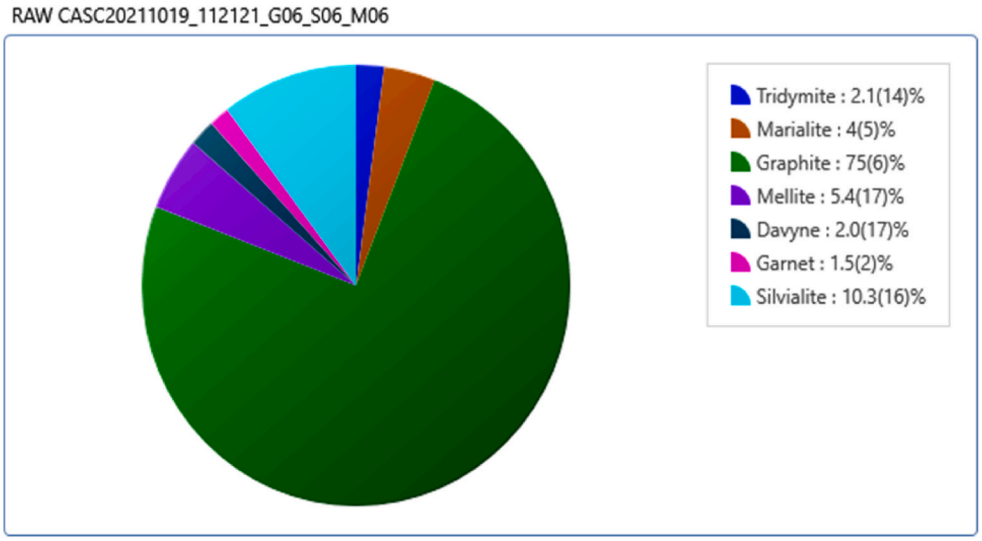
3.1. Quality characteristics of Sesame indicum seed oil (SISO)

3.1.1. Physiochemical properties of the oil

The summary of the physiochemical properties of the oil is presented in Table 2. The results were obtained in triplicates and the average are presented with their standard deviations. The percentage of oil extracted from sesame seed is 43.7 %. Oil content is one of the key factors that determine the choice of plant seeds as potential feedstock for biodiesel and other industrial products. The percentage oil yield from Sesame Indicum seed appears more encouraging for industrial uses as well as biofuel purposes than African star apple seed oil yield of 13.36 %. It quite compares with most oil seed feedstocks (peanut – 50 %, sesame seed – 45 % olive seed – 40 %, castor seed – 50 %, sunflower seed – 35 %) (Esonye et al., 2019b) However, when compared with the results previously obtained from the same plant seed, the results are in close agreement with the results earlier reported (51 %) [14], (46–64 %) [26] and (45.58, 46.79 and 48.13 %) of white, brown and black seed colours respectively [27]. From literature, the saponification value of the feedstock was between the ranges of the standard 175–205 mgKOH/g oil [28]. The saponification values of SISO were found to be 187.96 mgKOH/g oil, this value indicates that the benne seed is a normal triglyceride and suitable for FAME production. The acid value of 0.5436 and the free fatty acid (FFA) value of 0.2718 indicates that the oil requires no esterification since its FFA was significantly low and hence there will be no tendency of losing the oil to soap. The result equally compares well with values reported previously for tiger nut (1.12) and *chrysophyllium albidum* (2.81) [29]. The moisture content of the seed oil (0.4) and researchers have advised low moisture content in oil feedstock (<0.5 %) in order to achieve high biodiesel yield and prevent corrosion of engine metallic components [30]. The moisture content of SISO as obtained in this study would not show the above negative results since it is quite lower than 0.5 %. The kinematic viscosity for the SISO is observed to be 5.063 mm^2/s and already within the 1.9–6.0 mm^2/s standards limit of ASTM. It is lower than 8.08, and 5.6 mm^2/s reported for tiger nut and African pear respectively [24]. Although very low viscous liquid fuels do not provide enough lubrication for accurate fill of engine injection units, the biodiesel derivable from SISO would encourage excellent atomization in the engine because its viscosity is within the tolerable limits (1.9–6.0 mm^2/s) international standards [31]. The iodine value which is an indication of degree of unsaturation of a triglyceride is a very important parameter which is required to be less than 120 gI_2/100 g



a: Phase data view of raw CASC using XRD.



b; Plot of result for raw CASC using XRD

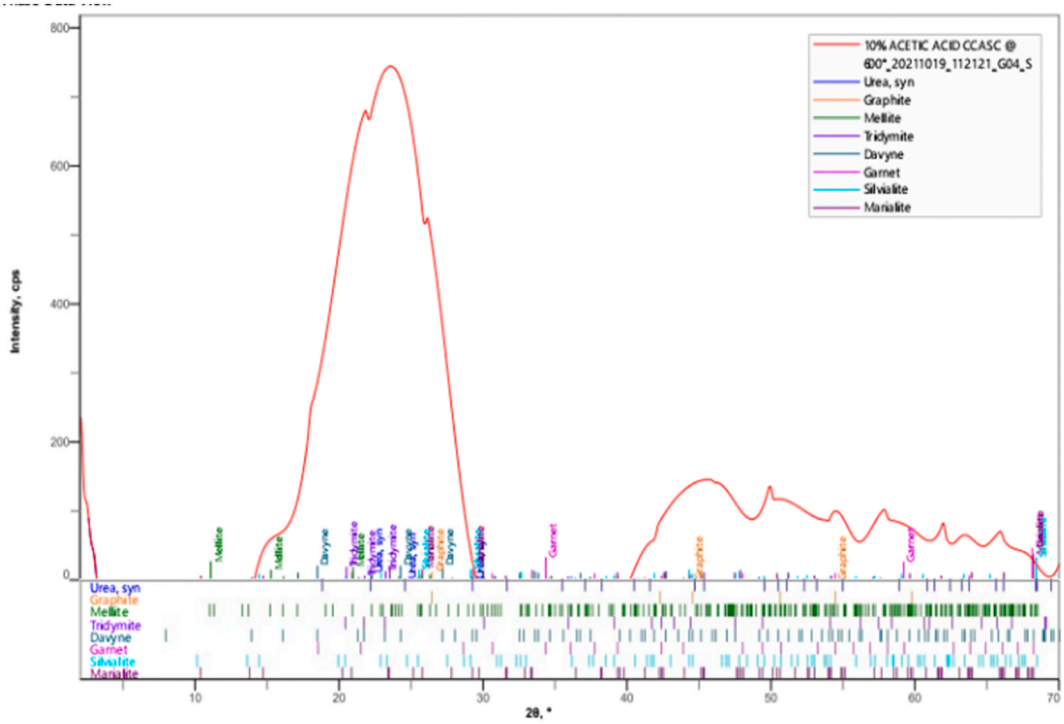
Fig. 3. a Phase data view of raw CASC using XRD. Fig. 3b; Plot of result for raw CASC using XRD.

for good biodiesel feedstock. Therefore, the iodine value of SISO (51.35 gI_2/100 g and peroxide value of 1.5 meq.oxy/g are well acceptable as it will not be susceptible to oxidative rancidity and glycerides polymerization on good storage and handling conditions. Generally, physicochemical properties of the raw oil compare favorably with those of some other oils such as *Pongamia pinnata* [32], *Jatropha curcas* [33] and *Madhuca indica* [34].

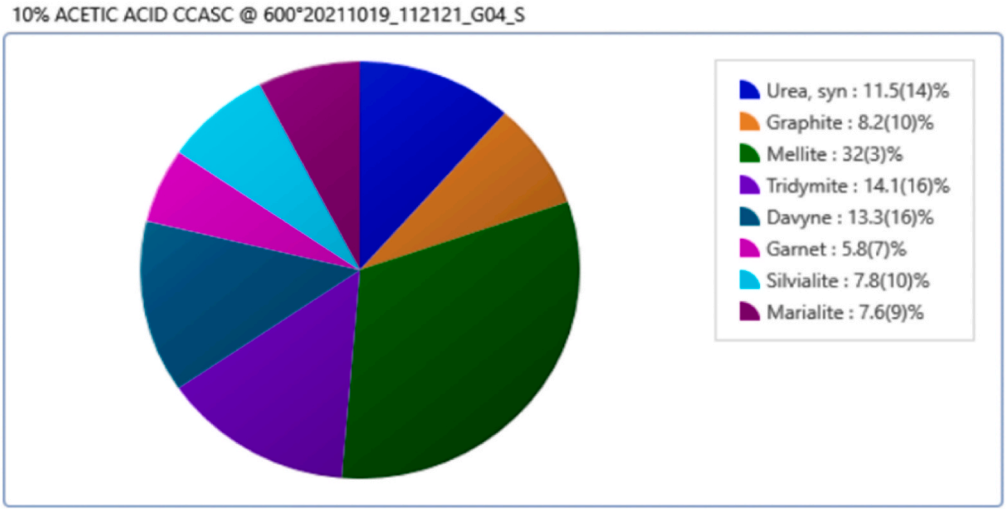
3.2. Characterization of Chrysophyllum albidum powdered catalyst

3.2.1. Catalyst SEM results

The SEM results showing the textural structures of the raw *Chrysophyllum albidum* waste seed powder viewed from the SEM images



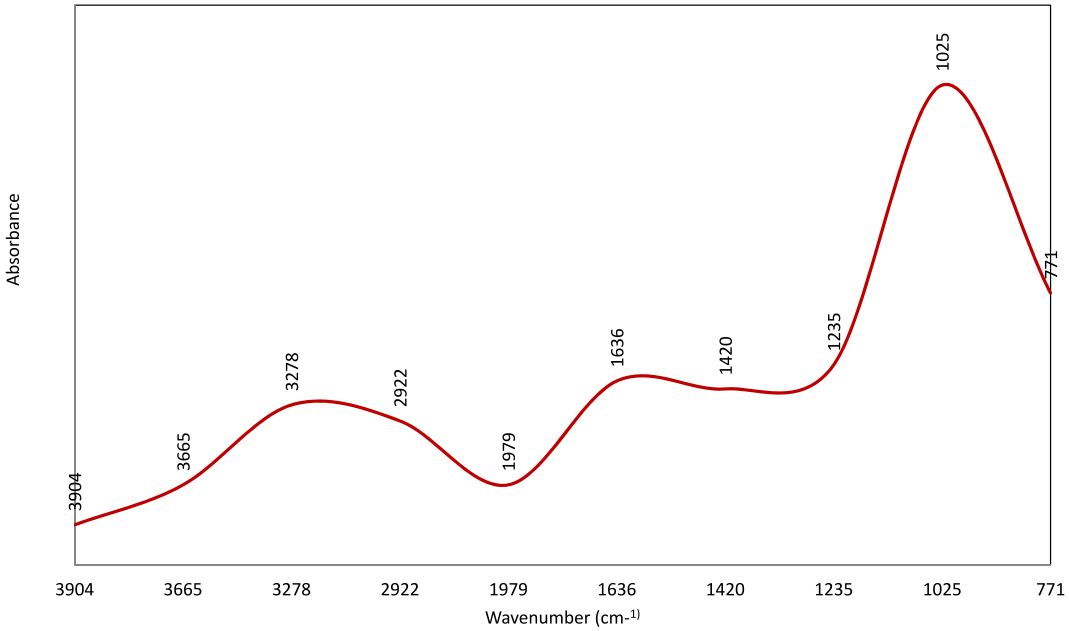
a: XRD Phase data view of acetic acid activated CASC at 600°C



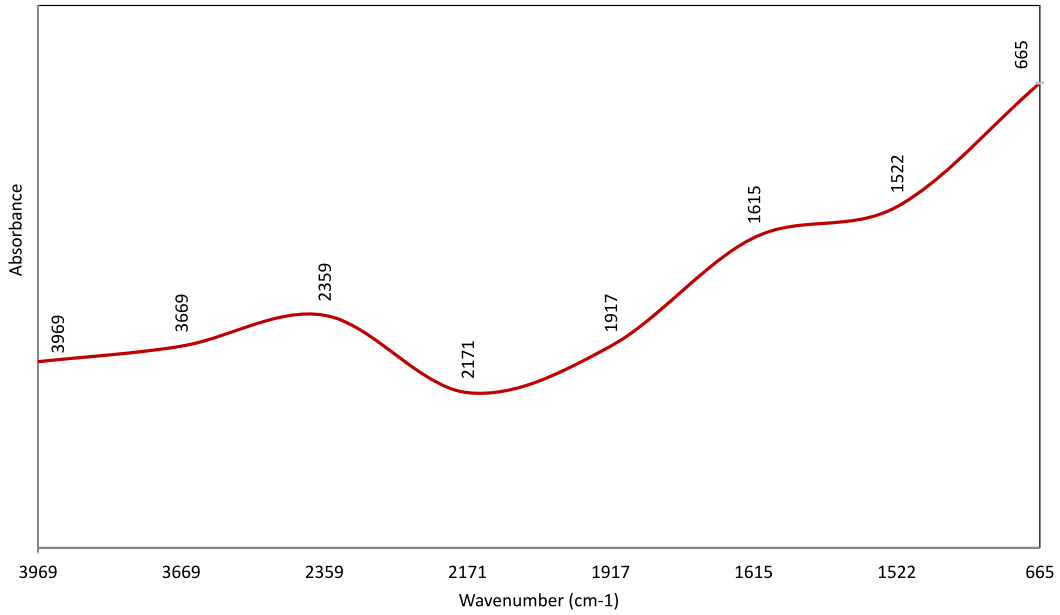
b: Plot of XRD result for activated CASC

Fig. 4. a XRD Phase data view of acetic acid activated CASC at 600 °C, Fig. 4b: Plot of XRD result for activated CASC.

are presented in Fig. 2a and b respectively. It contains the morphology of the sample with irregular pores, shapes and size, varying from one surface to another (50–200 μm of width). Consequently, the image of the raw sample shows that the shape of the sample was formed by tiny crystals in form of water droplet embedded on the large particles; probably due to the heterogeneous distribution in the mechanical properties of the sample shell used which can be regarded as an inherent attribute of high catalytic activity when activated. There is also a slight presence of jointed light-tiny strips with pores. Scanning electron microscope (SEM) analysis on the catalyst shows



a: FTIR spectrum of available functional groups in the raw *Chrysophyllum albidum* seed coat before activation.



b: FTIR spectra of acetic acid activated *Chrysophyllum albidum seed coat powder* at 600ºC.

Fig. 5. a FTIR spectrum of available functional groups in the raw *Chrysophyllum albidum* seed coat before activation. Fig. 5b: FTIR spectra of acetic acid activated *Chrysophyllum albidum seed coat powder* at 600 °C.

a significant change after been activated with acetic acid at a calcination temperature of 600 °C as shown in Fig. 2b. This affects the surface composition and structure of *Chrysophyllum albidum waste seed coat* powder. After activation with acetic acid, the sample shows irregularity in the crystal structure with homogeneous distribution and presence of large porosities was found in the sample.

3.2.2. The X-ray diffraction (XRD) results

X-ray diffraction analysis was used to confirm the purity and stability of the raw, thermal and that of acid activated *Chrysophyllum albidum seed coat*. It shows the diffraction angle or pattern of how the atoms are arranged in their crystals. Fig. 3a and b shows the XRD

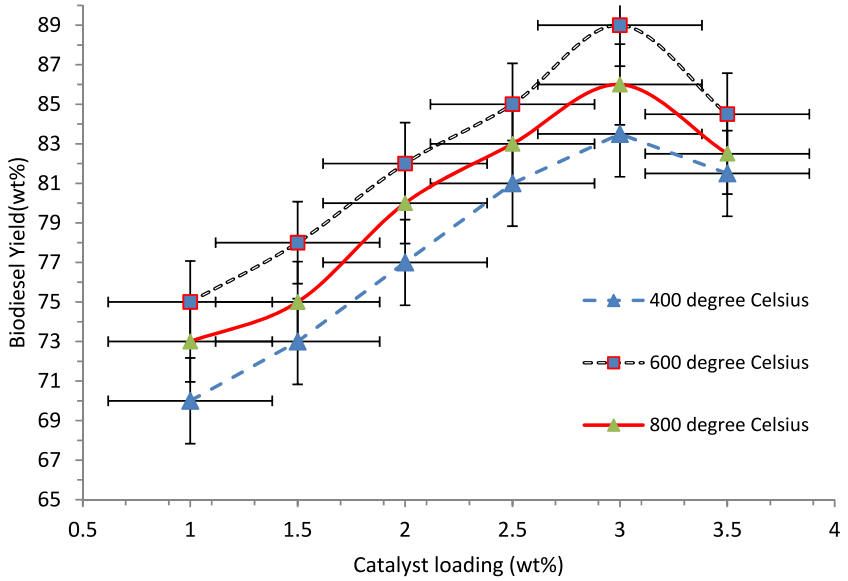


Fig. 6. Effect of catalyst calcined at 400 °C, 600°Cand 800°Cbiodiesel yield.

diffractograms of raw and acetic acid activated waste *Chrysophyllum albidum* seed coat respectively with the broad peak at 2θ. The diffraction peaks show that graphite(C) is the most prevalent in the sample with an occurrence rate of 75 %. There is slight presence of other compounds found in the raw CASC which includes; tridymite $Si(O_2)$ at 2.1 %, marialite $(Na_4Al_3Si_9O_{24}Cl)$ at 4 %, davyne $(K_2Na_4Ca_2A_{16}Si_6O_{24}C_{12})$ at 2 %, garnet 3 (Ca, Fe, Mg)O·(Al, F … etc) at 2 % and silvialite $Ca_4\ Al_6\ (Si\ O_4)_6$ at 2 %. After the catalyst was activated at 600 °C, it is shown in Fig. 4a (XRD Phase data view of acetic acid activated CASC at 600 °C) and Fig. 4b (plot of XRD result for activated CASC) that graphite which was more frequent in the raw sample was been carbonated as shown in the XRD result, thereby reducing its percentage occurrence to 8.2 %. There is presence of other trace compound and element found after carbonization at 600 °C, they include urea (CH_4N_2O) at 11.5 %, graphite (C) at 8.2 %, mellite $C_6(COO)_6Al_2{\cdot}18H_2O$ at 32 %, tridymite (SiO_2) at 14.1 %, davyne $(K_2Na_4\ Ca_2Al_6Si_6O_{24}Cl_2)$ at 13.3 %, garnet 3(Ca, Fe, Mg) O· (Al, Fe) at 5.8 %, silvialite $Ca_4\ Al_6$(Si O4)6 $(SO_4$, C … at 7.8 % marialite $(Na_4Al_3Si_9O_{24}Cl)$ at 7.6 %.

3.2.3. *The Fourier transform infra-red (FTIR) results*

Fourier transform infra-red (FTIR) spectroscope of the raw waste *Chrysophyllum albidum* coat (Fig. 5a) and acetic acid modified waste *Chrysophyllum albidum* seed coat catalysts (Fig. 5b) were done to ascertain the functional groups present in them and the type of vibration. The peak at 3565.2 cm^{-1} is attributed to internally bonded ▬OH group. The phenols (OH) and secondary alcohol and internally bonded ▬OH stretch appeared at lower energy levels of 3904.4 and 3565.2 cm^{-1} respectively in the raw biocatalyst but appeared at higher energy levels of 3969.6 cm^{-1} after calcination. This could be attributed to the conversion of Ca(OH) to CaO due to water loss after high temperature calcination. The 1636.3–1979.2 cm^{-1} range is attributed to the minor inorganic compounds of Mg, P, and S [17]. The occurrences of bands at 1420.1 cm^{-1} and 1025.0 cm^{-1} is attributed to metal oxides of Ca▬O and K▬O respectively, while the peak at 771.6 cm^{-1} is attributed to O▬C▬O stretching peak of carbonate [35]. The amine groups that appeared at 1636.3 cm^{-1} in the raw biocatalyst appeared at a lower energy level of 1615.8 cm^{-1} in the activated biocatalyst. The aromatic esters (▬O) that appeared at 1235.6 cm^{-1} disappeared completely in the activated biocatalyst because of their high volatility. Also, the silicate ion, siloxane or silicone group (Si▬O▬Si) attributed to the bands at 1025 cm^{-1} in the raw biocatalyst disappeared equally in the activated catalyst. Also, the transition metals carbonyls appeared strongly at 1917.7 cm^{-1} in the activated catalyst.

3.3. *Analysis of process variables on biodiesel yield*

3.3.1. *Effects of catalyst loading and calcination temperature on biodiesel yield*

The transesterification reaction was significantly affected by interaction between catalyst concentration (wt. %) and reaction temperature (Fig. 6). The calcination temperature effect on the yield was studied in the temperature range of 400, 600 and 800 °C. The maximum yield of 89.93 wt% was obtained at a temperature of 600 °C. A decrease in yield was observed when the reaction temperatures were above (800°Cand below 400 °C. This implies that 600 °C is the best calcinations temperature. Basically, calcination process plays a vital role in the structural evolution and metal dispersion [36]. Excessively high or low calcination temperature causes poor catalytic performance. The variation of the catalyst performance with temperature could be due changes in the catalyst biomass morphology, coke removal and sintering which can occur above 600 °C (800 °C) while at temperatures below 600 °C (400 °C) less surface area and larger pore diameter. Small pore diameters make the reactants and products to diffuse freely and larger surface area provides sufficient active sites for catalysis. Researchers have observed similar trends in nickel nitrate hexahydrate - ZrO_2 supports

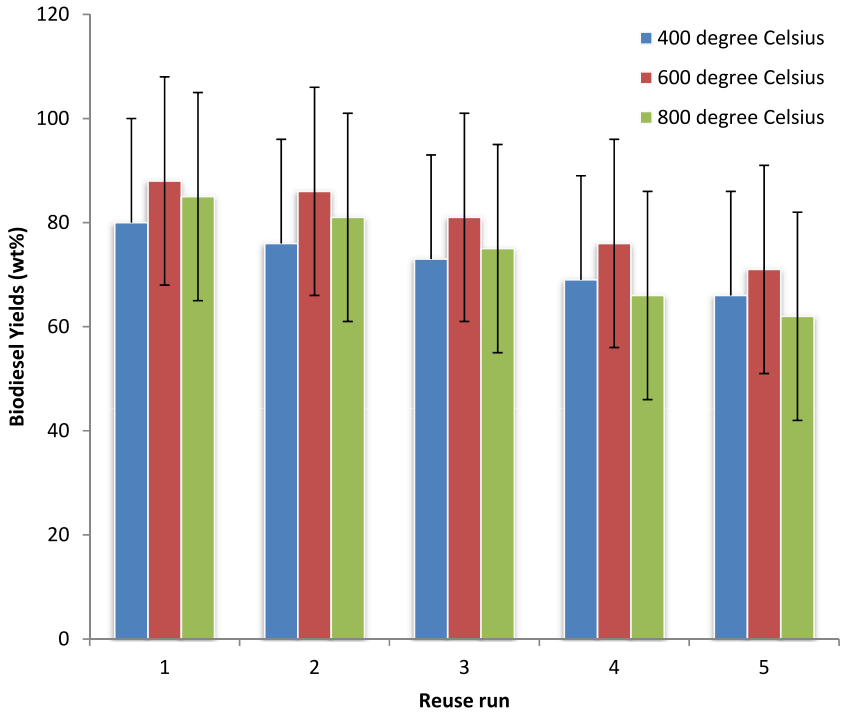


Fig. 7. Catalyst recyclability at different calcinations temperatures.

Table 3
Properties of optimal biodiesel produced from bene seed (*Sesamum Indicum*) oil.

Sn	Property	**400**°C	**600**°C	**800**°C	ASTM D 6751
1	Acid value (mgKOH/g)	0.334 ± 0.19	0.445 ± 0.13	0.333 ± 0.20	0.5 max
2	Density (g/cm^3)	0.9091 ± 0.04	0.9082 ± 0.05	0.9089 ± 0.04	0.860-0.894
3	kinematic viscosity (mm^{2}s-1)	6.145 ± 0.33	6.196 ± 0.32	6.120 ± 0.21	1.9-6.0
4	Flash point (°C)	176 ± 2.5	182 ± 1.7	185 ± 1.5	130min
5	Fire point (°C)	188 ± 2.24	190 ± 1.22	192 ± 1.79	–
6	Refractive index	1.4704 ± 0.007	1.4703 ± 0.009	1.4704 ± 0.006	≤ 0.5
7	Free fatty acid (mg KOH/kg)	0.167 ± 0.027	0.223 ± 0.030	0.166 ± 0.040	0.25 max
8	API gravity	23.59 ± 1.1	23.75 ± 1.2	22.63 ± 2.2	15-45
9	Aniline value (°C)	57.00 ± 2.3	56.50 ± 3.1	56.50 ± 2.2	94 max
10	Diesel index	73.60 ± 2.5	73.60 ± 2.4	73.60 ± 1.3	>50

from China catalyst activity [37] and a series of Cu-mcm-41 catalyst [36].

Also, it was observed that for three calcinations temperatures studied, the biodiesel yield increased steadily from 1 wt% up to 3 wt% catalyst concentrations. Beyond 3 wt% up to 3.5 wt% the yield dropped from 89.93 wt%, 85.62 wt% and 82.55 wt% to 84.65 wt%, 80.43 wt%, and 79.67 wt % for 600 °C, 800 °C and 400 °C of calcination temperatures respectively. It implies that the optimum conditions of calcination temperature and catalyst loading were 600 °C and 3 wt% to realize the maximum yield of 89.93 wt% under fixed conditions of agitation speed of 500 rpm and 12:1 M ratio of alcohol to oil and reaction duration of 2 h.

3.4. Catalyst reusability versus yield of biodiesel

After the first transesterification process, subsequent transesterification runs up to 5th cycle was studied as shown in Fig. 7. About 89.93 wt% maximum conversion of benne seed oil to biodiesel was observed initially before catalyst regeneration and it dropped to 66.20 wt%, 71.50 wt% and 62.65 wt % after the 5th run at calcination temperature of 400 °C, 600 °C, and 800 °C respectively. The initial yields before reuse were 80 wt%, 88.0 wt % and 85 wt % for 400, 600 and 800 °C calcination temperatures respectively. After the third cycle, the yield dropped to 73, 81 and 75 wt% for 400, 600 and 800 °C calcination temperatures respectively. After the 5th cycle, the yield dropped to 66, 71 and 62 wt% for 400, 600 and 800 °C calcination temperatures respectively. Therefore, it is obvious that beyond the 5th cycle, the yield would drop quite below 70 wt% yield indicating that the catalyst has lost significant catalytic activity. This could be due to deactivation resulting from the reduction of the active sites because of the leaching of the active components and as well as the deposition of oil, moisture, and impurities on the surface of the catalyst [17]. Considering other factors

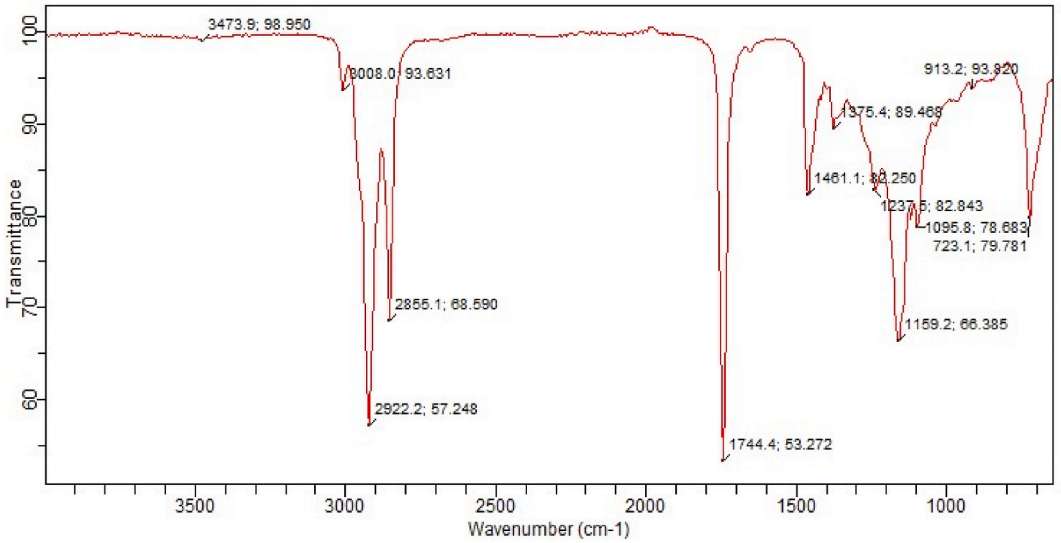


Fig. 8. FTIR spectra of optimal yield biodiesel at 400 ^{O}C.

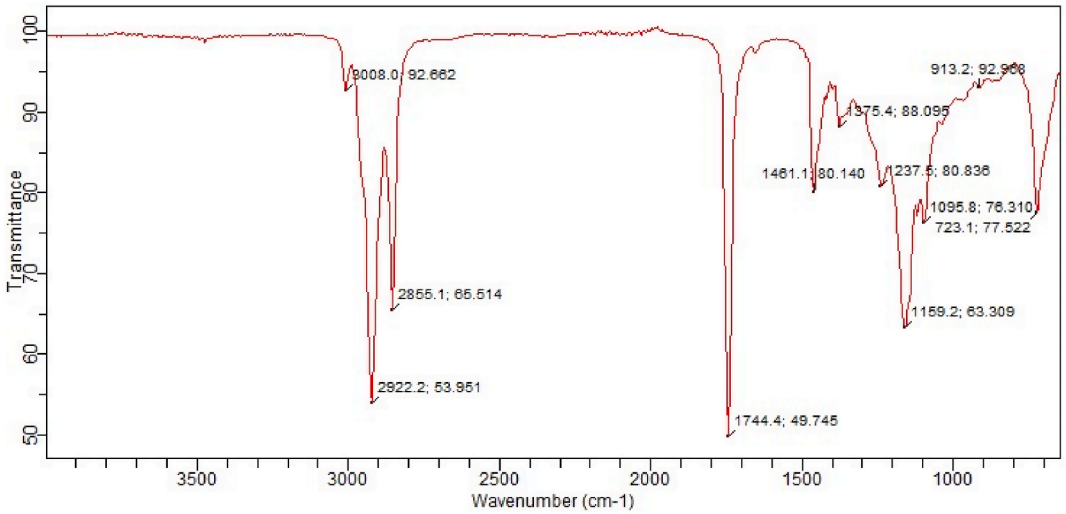


Fig. 9. FTIR spectra of optimal yield biodiesel at 600 ^{O}C.

of production, it might make no further economic sense to run the process below 5th cycle that gave 71 wt % yield.

3.5. Characterization of optimal yield biodiesel

Different properties of the biodiesel produced from bene seed (*Sesamum Indicum*) oil have been estimated with their standard deviations and listed in Table 3. Density is an important fuel property during combustion in IC engines, because injection systems, pumps, and injectors must deliver an amount of fuel precisely adjusted to provide proper combustion [38]. Also, the diesel index of 73.75 is indication of high quality of in the biodiesel. It is an empirical measure of the ignition quality of diesel based on API gravity and aniline point of fuel. The higher the value of the diesel index, the better the fuel quality. However, the value of about 23 indicates that the biodiesel falls within a medium class of (30>API>22). Also, the aniline value of about 57 °C was recorded against the maximum value of 94 °C for the produced biodiesel. The knowledge of aniline value in fuels is very important as a primary point of consideration on storage facility. Aniline point shows the indication of the likelihood for a fuel or oil to damage elastomers such as rubbers when in contact. It therefore provides an approximation for the amount of aromatics present in the fuel. It increases with diesel index. It implies that the produced diesel can be stored in rubber vessels with any tendency of damage. Other properties of the biodiesel indicate good quality for effective performance in diesel engines [20,39].

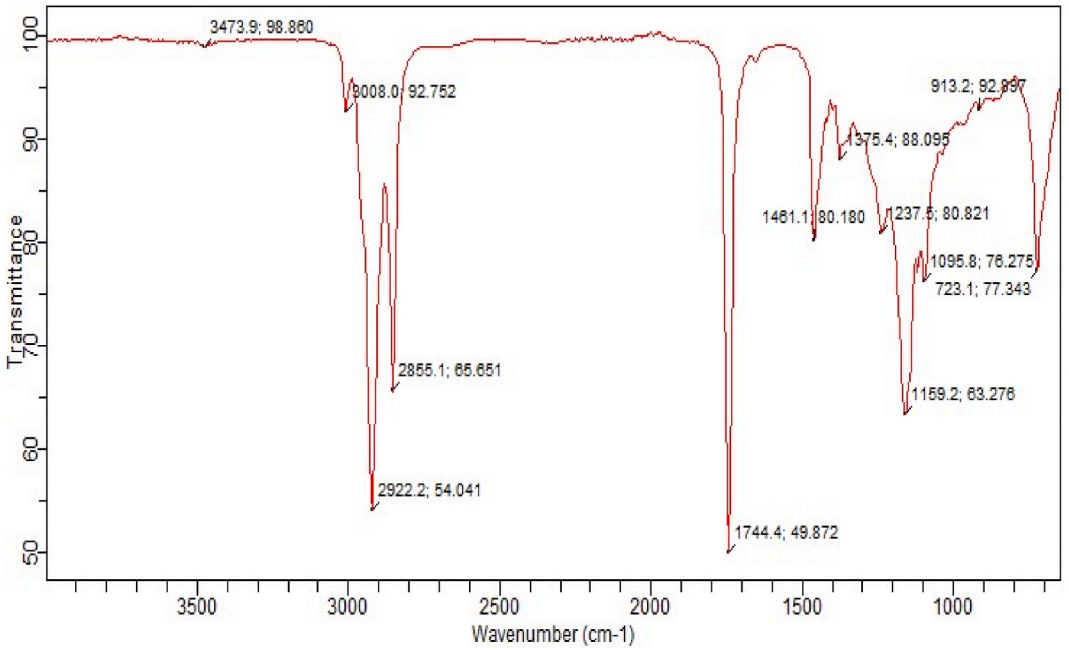


Fig. 10. FTIR spectra of optimal yield biodiesel at 800 ^{O}C.

Table 4
The summary of the fatty acid composition of SISO biodiesel.

Sn	Fatty Acid	IUPAC Name	Structural Formula	Saturation Status	Composition (%)
1	Lauric(12:0)	Dodecanoic	$C_{12}H_{24}O_2$	Saturated	2.91
2	Palmitic(16:0)	Hexadecanoic	$C_{16}H_{32}O_2$	Saturated	3.83
3	Stearic(18:0)	Octadecanoic	$C_{18}H_{36}O_2$	Saturated	31.27
4	Oleic(18.1)	Octadec-9-enoic	$C_{18}H_{34}O_2$	Mono-unsaturated	27.60
5	Linoleic(18:2)	Cis,*cis*-9,12-octadecadienoic	$C_{18}H_{32}O_2$	Poly-unsaturated	11.72
6	Linolenic(18.3)	*Cis*-9,12,15-octadecatrienoic	$C_{18}H_{30}O_2$	Poly-unsaturated	17.80
7	Eicosatrienoic (20:3)	Eicosapentaenoic	$C_{20}H_{34}O_2$	Poly-unsaturated	3.52
8	Eicosadienoic (20:2)	*Cis*-11,14-eicosadienoic	$C_{20}H_{36}O_2$	Poly-unsaturated Poly-unsaturated	3.29
9	Arachidonic(20:4)	*Cis*-5,8,11,14-eicosatetraenoic	$C_{20}H_{32}O_2$		2.06
10	Others	–	–	–	5.99

3.6. Fourier transform infrared spectroscopy analysis of optimal yield biodiesel

A non-destructive technique (FTIR) has been widely applied in the characterization of biodiesel. Biodiesel portrays functional groups with characteristic transmittance bands in the infrared region of the electromagnetic spectrum. Figs. 8–10 show the FTIR spectral view of the biodiesel produced using catalysts thermally activated at 400 °C, 600 °C and 800 °C respectively from sesame seed oil. There was no significant difference in the results of the peak values of the spectrum based on the different calcination temperatures. It implies that the calcination temperature of the catalyst has no influence on the functional groups of the produced biodiesel. Different intense peaks such as the doublet of the - CH_3 and - CH_2 anti-symmetric stretching vibration at 2922.2 cm^{-1} and 2855.1cm−1 were detected. Another peak at 1744.4 cm^{-1} was observed, which is attributed to the stretching of C═O, the peak at 1461.1 cm^{-1} correspond to the asymmetric stretching of CH_3 present in the refined oil spectrum (Soares et al., 2010). The peak at 1374 cm^{-1} can be attributed to the glycerol group O–CH_2 (mono- di- and triglycerides), which is present in the refined oil spectrum. The stretching of O–CH_3, represented by the absorbance at 1156 cm^{-1}. The specific peak at 721.4 cm^{-1} indicates methylene functional group in the biodiesel (-(CH_2) n-). In the FTIR spectroscopy study of the biodiesel produced at 600 °C we observe that there is absence of the amine group (N–H) stretching at peak level of 3473.9cm-1. Absence of this functional group contributes to the yield effect of the biodiesel.

3.7. Fatty acid composition of the optimally produced biodiesel

The study of the GC analysis of optimal yield biodiesel shows the presence of several components as shown in Table 4. It shows the presence of 38.01 % saturated fatty acids and 62.7 % unsaturated fatty acids (27 % mono-unsaturated fatty acid, and 35.1 %

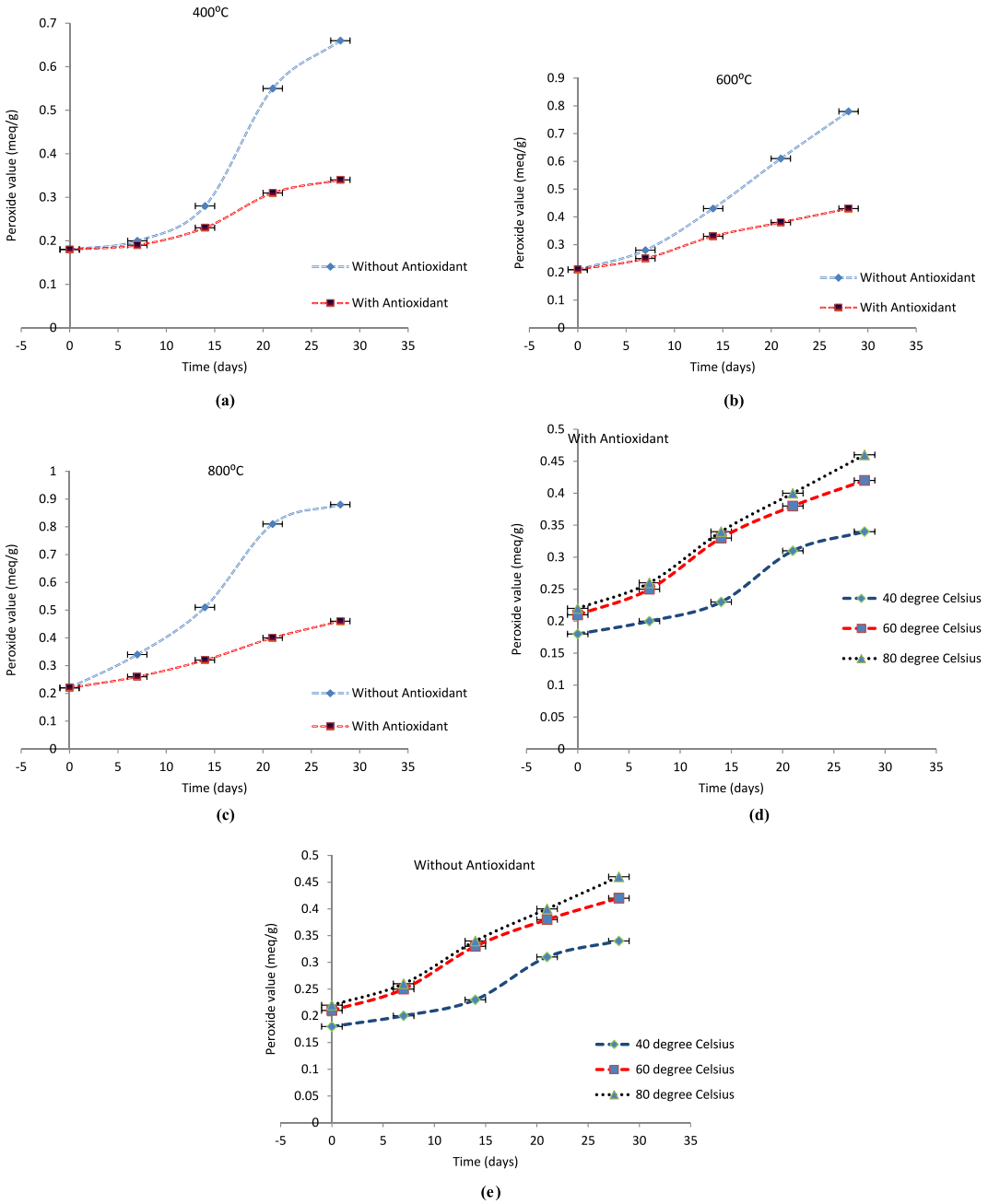
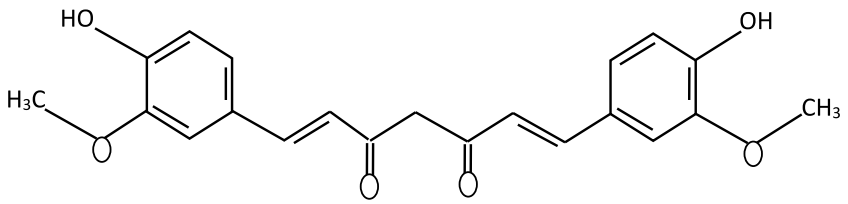


Fig. 11. Oxidation stability analysis at different transesterification temperatures.



Sketch 1. Chemical structure of Curcumin (59–70 %) as the main curcuminoids in turmeric [16].

polyunsaturated fatty acids). It implies that the produced biodiesel is fairly susceptible to oxidation on long term storage. To improve the stability of the biodiesel qualities, antioxidants can be added to the biodiesel.

3.8. Oxidation stability analyses

The result of the oxidative stability analysis of the produced SISO FAME at control conditions (without an antioxidant) and in the presence of natural, renewable and sustainable antioxidant (1 % Turmeric) is shown in Fig. 11. Oxidation stability is one of the most important fuel properties with respect to usage, storage and performance of biodiesel. Unstable fuel can lead to increased viscosity, as well as formation of gums, sediment, and other deposits. Oxidative stability is determined not only by FAME compositional properties, but also by the age of the biodiesel and the conditions under which it has been stored. The oxidation stability was studied for 28 days by studying the peroxide values at intervals of four consecutive 7 days. The initial peroxide value was 0.1875, 0.2105 and 0.2165 mg eq./kg oil for the catalyst samples calcined at 400 °C,600 °C, and 800 °C, respectively as shown in Fig. 11 a, b and c respectively.

The rate of increase in the peroxide values for the SISOB at all temperatures initially were very slow and this is regarded as Lag periods. Lag period of 14 days, 7 days and about 4 days were observed with catalyst calcined at 400 °C, 600 °C, and 800 °C, respectively. This period of time is the equally called the induction time. At corresponding storage times, the values of peroxide values of SISOB in mix with turmeric antioxidant were observed to be lower than those of normal SISOB. This may be attributed to the reduction of the degree of unsaturation of the SISOB by the antioxidant which translated to lower oxidation rates.

After 28 days studied storage duration and temperatures of 40, 60 and 80 °C, the peroxide values of samples without antioxidants were 0.661, 0.784 and 0.830 against 0.344, 0.427 and 0.457 mg eq/I_2 with 1 % turmeric extract as antioxidants (Fig. 11d and e). This indicates that the higher the storage temperature, the less effective the antioxidant and as well as the higher the rate of oxidation degradation [40]. This implies that the presence of the natural antioxidant was able to reduce the oxidation instability of the produced biodiesel by 50 %.

4. Conclusion

A successful development of a viable route for the conversion of sesame indicum seed oil into a renewable and eco-friendly biodiesel using bio-catalyst of waste seed coat of *Chrysophyllium albidum* has been done and reported in this work. The *Chrysophyllum albidium* seed coat biocatalyst was found to possess high purity, stability and sufficient porosity with well distributed homogeneous crystal structure. The potential of *Chrysophyllum albidium* seed coat (CASC) catalyst was confirmed through high reusability and yield of biodiesel (90 wt%) at moderate factor values of calcinations temperature (600 °C), alcohol oil molar ratio (12:1), catalyst concentration (3 wt%), reaction stirring rate (500 rpm) and reaction time (2 h). The properties of the bene seed oil and its biodiesel shows highly potential feedstock and indicates good quality biofuel for effective diesel engine performance. The oxidation stability improvement of the produced biodiesel by 50 % was achieved with 1 % turmeric. The viability of the process shows an ideal process of converting waste to wealth and generating eco-friendly fuel.

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Data availability statement

Data included in article/referenced in article and has not been deposited into any publicly available repository by the authors.

CRediT authorship contribution statement

Esonye Chizoo: Conceptualization, Data curation, Formal analysis, Investigation, Methodology. **Mbonu Felix Okechukwu:** Investigation, Resources, Software, Supervision, Writing – review & editing. **Onukwuli Okechukwu Dominic:** Investigation, Project administration, Supervision, Validation, Writing – original draft, Writing – review & editing. **Ani Amechi Kingsley:** Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation. **Augustine Simon Chimamkpam:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources. **Muodumogu Chiamaka Mariagorretti:** Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization. **Ezeagu Paschal Chinonso:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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