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Extraction And Characterization of Bio-Oil from Waste Kolanut Pod For Biodiesel Production

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Abstract:
The quest for sustainable and renewable energy sources has led to the exploration of novel biofuel feedstocks. Kolanut oil, an unconventional feedstock, was investigated for its potential as a biofuel. This study aimed to evaluate the suitability of kolanut oil for biofuel production by conducting a comprehensive phytochemical analysis, scanning electron microscopy (SEM), and Fourier Transform Infrared (FTIR) spectroscopy. The phytochemical analysis revealed the presence of bioactive compounds, including flavonoids, phenolics, and alkaloids, which are indicative of the oil's potential resistance to oxidative degradation. SEM analysis showed a rough, irregular texture with small pores and crevices, suggesting potential energy storage capabilities. FTIR results confirmed the presence of unsaturated fatty acids, particularly oleic and linoleic acids, and triglycerides, characteristic of biofuel feedstocks. The results of this study suggest that kolanut oil has a high potential to be used as a biofuel due to its chemical composition, physical properties, and resistance to degradation. The findings of this research provide valuable insights into the suitability of kolanut oil as a biofuel feedstock and highlight its potential applications in the energy sector. Further research is recommended to fully evaluate the performance and feasibility of kolanut oil as a biofuel feedstock.

Keywords: Renewable energy, Fourier Transform Infrared (FTIR) spectroscopy, biofuel feedstocks, phytochemical analysis, scanning electron microscopy (SEM)

INTRODUCTION

1.1 Background of the Study

Rudolf Diesel, the inventor of the diesel engine, ran the first diesel engine on groundnut oil at the Paris Exposition of 1900 [1]. This was the origin of the use of vegetable oils as fuel for diesel engines. However, due to the abundant supply of fossil diesel, Research & Development activities on vegetable oil were not seriously pursued. It received attention only recently when it was realized that petroleum fuels were dwindling fast, and environment-friendly renewable substitutes must be identified, the commonest of which is biodiesel. Biodiesel is a clean, smouldering,

renewable fuel that is usually produced from vegetable oils, animal oil or fats, waste cooking oil and tallow [2]. In biodiesel production, many different feedstocks have been used. Biodiesel can be produced by utilizing edible oils and non-edible oils as resources. However, usage of the edible oil can result in competition between usages of food against fuel. This also can increase the condemnation of sustainable edible oils for biodiesel production [3]. Many types of non-edible oils have been used as feedstock for biodiesel production [4]. These include Jatropha *curcas*, *Pongamia pinnata* (Karanja), *Madhuca indica* (Mahua), Linseed, Cottonseed, *Azadirachta indica* (Neem), Camelina, Reutealis Trisperma, Calophyllum Inophyllum, Hevea Brasiliensis, Ricinus Communis, Ceiba pentandra, Schleichera Oleosa, Cerbera manghas and beauty leaf tree [2]. The ultimate goal is to develop specific characteristics and properties of biodiesel through blends and other modifications to have better alternatives to conventional diesel with enhanced efficiency in terms of performance and emissions in internal combustion engines.

1.2 Statement of Problem

Presently, researchers have been preoccupied with finding different sources of biodegradable fuel from biomass (feedstock) as a better alternative to fossil fuel. From the literature, little has been mentioned about the use of waste kolanut pod oil and its blends as a possible source of biodiesel. Consequently, this study aimed at studying the possibility of developing biodiesel using waste kolanut pod oil and its blends with enhanced properties better than, or at best, close to the properties of conventional diesel. The ultimate goal was to find a sustainable, renewable source of fuel that would be beneficial both to manufacturing companies (using diesels as an integral part of production) and the general needs of the transportation sector.

1.3 Aim and Objectives of the Study

This research aimed at extracting and characterizing the material gotten from waste kolanut pod. The specific objectives of this research include the following:

i. Provide and extract oil from waste kolanut pod
ii. Characterise the extract with the use of phytochemical analysis, EDX and FTIR

2. METHODOLOGY

The following methods were adopted for the study:

2.1 Sample collection

Waste kolanut pods were collected from the dried parent fruits purchased from the local dealers at Ubani market in Umuahia, Abia State. After de-husking to remove the kolanut seeds, they were further sun-dried to expel the remaining moisture.

2.2 Oil extraction by Soxhlet Method

Kolanut pods were obtained air dried, sorted to remove impurities and ground using an industrial blender, while 0.1 kg of the ground sample were weighed into a semi-permeable cotton material and placed into the timble of a 0.5 kg Soxhlet extractor as presented in Fig 2.1. After that, 0.4 kg of n- hexane was measured into a 0.5 kg flat bottom round flask and the Soxhlet with the extraction timble containing the sample was connected with the condenser which was fitted to the flat bottom round o flask containing the n-hexane. The Soxhlet extraction system was heated on a hot plate at 60 C for 60 minutes while water was allowed to circulate at the outer jacket of the condenser. The extraction was discontinued when oil was completely extracted from the sample (Fig. 3.2). The de-fatted sample in the semi-permeable membrane also was discarded, while the oil and the n-hexane mixture in the flat bottom flask were separated by distillation.

Fig. 2.1: Oil extraction using Soxhlet extractor

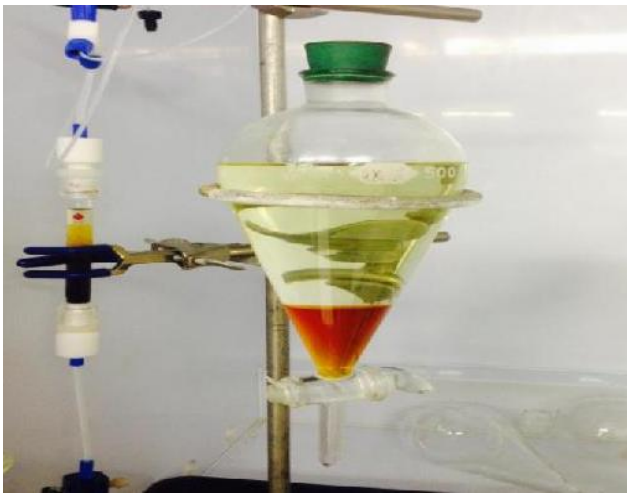

Fig. 2.2: Extracted kolanut pods oil and n-hexane

2.4 Oil characterization

Oil from kolanut pods was characterized to determine its physicochemical properties such as the density and specific density, refractive index, moisture content, saponification value, viscosity, acid value, peroxide value and iodine value, flash and fire points, cloud and pour points, using the following procedures:

(i) Density and specific density:

An empty beaker was weighed and the weight was recorded. After that, 50g of the oil sample was introduced into a 50 ml density bottle and the weight of the density (specific gravity) bottle and sample were taken. Sample weight was obtained by subtracting the weight of the empty density bottle from the weight of the sample and the density bottle. From the sample weight obtained, the density was determined by taking the ratio of the weight of the oil sample to the known volume (50g) according to equation (2.1)

$$Density\ (\rho) = Ws/Vs \quad \text{---------- (2.1)}$$

Where: Ws = sample weight or weight of sample, Vs = sample volume

The specific gravity (Sg) was determined using the 50g capacity pycnometer density bottle. The weight of the empty bottle and that of the 50g bio-oil sample had been taken earlier. Thus, after washing the bottle and drying it, it was filled with water (50g) and weighed. Consequently, the specific gravity was calculated using the expression in equation (2.2).

$$Specific\ gravity, (Sg) = Ws/Ww \quad \text{---------- (2.2)}$$

Where: W_s = sample weight or weight of sample, W_w = weight of water.

(iii) Refractive index:

Refractive index was determined using a digital tabletop refractometer (HI96800) manufactured by Hanna Instruments, Romania adopting the AOAC [8] method in Yau et al. [7]. The device was initially calibrated to zero using distilled water and the sample was placed at the glass prism and read off using the refractive index key.

(iv) Moisture content:

Moisture was determined by the dry oven method used in Carneiro et al. [11] where 5g of the sample was weighed into an already weighed petri dish "a". The sample o in the petri dish was transferred into the oven and left for an hour at 105 C, and thereafter allowed to cool in a desiccator, while the second weight of the sample was taken after oven heating "b", and the final weight "c" after the cooling was recorded. Percentage moisture was thus calculated using equation (2.3)

$$\%\ moisture\ (Sg) = ([(a+b)-c]/a) * 100 \quad \text{-------- (2.3)}$$

Where: (a + b) – c = weight loss and a = sample weight.

(v) Saponification value:

Saponification value (SV) was determined according to the method of Yau et al. [11]. This is the milligram of KOH required to neutralize the fatty acids resulting from the complete hydrolysis of 1g of the sample. SV measures the molecular weight of the fatty acid in the sample. A sample weight of 0.5g was weighed into a conical flask and 50g of 0.5N ethanolic KOH was added to the sample. The mixture was refluxed to saponify the sample. The unreacted KOH was titrated back with 0.5N hydrochloric acid (HCl) using three drops of

phenolphthalein as an indicator. The SV of the sample thus was calculated using equation (2.4).

$$\text{Saponification value, (SV)} = \frac{\text{Tv} \times NHCL \times 56.1}{\text{Ws}} \quad \ldots\ldots\ldots\ldots \text{(2.4)}$$

Where the NHCl = normality of HCl acid = 0.5N; 56.1g/mol = molecular weight (molar mass) of KOH (MwKOH) and Tv = titre value = titre value of the sample, V1 – titre value of the blank, V2.

(v) Viscosity:

U-tube viscosity manufactured by Poulten Selfe and Lee Ltd (PSL ASTM-IP 350) was used to determine the kinematic viscosity. Micropipette was used to introduce 5g of the sample into the viscometer, while sample flow time from the upper to the lower meniscuses was o determined in seconds at 40 C. Consequently, the oil viscosity was calculated using the methods of ASTM D341-93 [10] and ASTM D341-20 (2020) presented in equations (2.5).

$$\text{kinematic viscosity, } (\upsilon) = \frac{Vmsv \times Pa.s}{10(-3)} \quad \ldots\ldots\ldots\ldots\ldots\ldots \text{(2.5)}$$

Where: μ = dynamic viscosity and Vmsv = measured viscosity value of the sample from the viscometer reading in Pa.S.

(vi) Acid or free fatty acid value (ASTM, 2004):

Acid value or free fatty acid (FFA) value is the number of milligrams of KOH required to neutralize the FFA in 1g of the sample. The ASTM [10] method where 0.5g of the sample was weighed into a conical flask, and 20g of ethanol and three drops of phenolphthalein indicator were introduced was adopted for the test. The solution was titrated with constant agitation until a faint, pink endpoint appeared and persisted for thirty seconds, and the volume of the titrant at the endpoint was recorded. From the readings obtained, the AV was calculated using the equation (2.6).

$$\text{Acid value, (AV)} = \frac{\text{Tv} \times NHCL \times 56.1}{\text{Ws}} \quad \ldots\ldots\ldots\ldots\ldots \text{(2.6)}$$

Where, NKOH = normality or concentration of KOH = 0.5N and 5.61 = molecular weight of KOH (MwKOH) divided by 10.

(vii) Peroxide value:

The peroxide value (PV) was determined using the method advocated in Boerlage and Broeze [12]. This measures the peroxides contained in the oil and determines the rancidity of a sample containing fat or oil subject to oxidation. Fresh oils usually have PVs well below 10mEq/kg (milliequivalent/kg) of fat. A rancid taste often begins to be noticeable when the PV is between 20 and 40mEq/kg. For the test, 0.5g of oil sample was weighed into a conical flask and 25g glacial acetic acid and chloroform was mixed in the ratio 2:1. Afterwards, 1g of 10% potassium iodide (KI) was added and vigorously shaken, covered and kept in the dark for one minute. Thereafter, 35g of starch indicator was added, and the whole solution was titrated with 0.02N sodium thiosulphate (Na2S2O3) solution and observed as the solution turned from pale black to white. Also, the titration was made for blank, and the PV was calculated using the equation (2.7).

$$\text{Peroxide Value, (PV)} = \frac{100 \times (V1 - V2) \times \text{N}}{\text{Ws}} \quad \ldots\ldots\ldots\ldots\ldots \text{(2.7)}$$

Where: 100 = milliequivalent conversion factor; V1= titre value of the sample; V2 = titre value of the blank; and N = the normality of the titrant = 0.02 of Na2S2O3 solution.

(viii) Iodine value:

The iodine value (IV) was determined using the method of Boerlage and Broeze [12], and measures the weight of iodine absorbed by 100 parts by weight of the sample. The test was conducted by weighing 0.5g of the sample into a conical flask and adding 15g of chloroform into it. Also, 25g of Wiji's solution thereafter was added and mixed vigorously, covered tightly and placed in the dark for 30 minutes. Consequently, 20g of 10% KI was added and 150g of distilled water was added. The solution was observed as it turned red, and 5g of 5% starch solution indicator was

added and also observed as the solution turned blue-black. The solution was titrated afterwards with 0.1N Na2S2O3 solution until black precipitates appeared in the colourless solution, and titration was also made for blank. Thus, IV was calculated using the equation (2.8).

$$\text{Iodine Value, (IV)} = \frac{12.69 \times (V1 - V2) \times \text{N}}{\text{Ws}} \quad \ldots\ldots\ldots\ldots\ldots (2.8)$$

Where: 12.69 = molecular weight of iodine in g/mol (i.e., 126.9/10) and N = normality of titrant = 0.1N Na2S2O3 solution.

(ix) Flash point and fire point:

The flash point is the lowest temperature at which the fuel ignites when it comes in contact with a light source while the fire point is the lowest temperature at which the fuel combusts continuously when a light source ignites it (Wakil et al., 2012). Flash point and fire point were determined by pouring the diesel sample into a glass petri dish so that the surface of the dish was covered.

A mercury-in-glass thermometer was immersed into the sample in the petri dish so that the tip of the thermometer just touched the diesel sample. The thermometer was held to position using a retort stand and clamp, while the sample was placed on a laboratory heating mantle. The sample was gradually heated and a light source was applied at intervals. The lowest temperature at which the sample just ignited and went off was taken as the flash point, while the lowest temperature at which the sample persistently ignited was called the fire point.

(x) Cloud point and pour point:

Cloud point is the temperature at which the diesel begins to form a mass of cloud at the surface of the diesel while the pour point is the temperature at which the sample solidifies or forms a wax-like mass. For the test, the biodiesel sample was placed in a medium-sized test tube and the test tube with its content was placed in a test tube rack, stored in a refrigerator and monitored. The temperature at which the heavier components formed a mass of colloids was taken as the cloud point, while the temperature at which the sample solidified was the pour point. The temperature was determined using a mercury-in-glass thermometer.

3. DISCUSSION OF RESULTS

3.1 Physico-Chemical Characterization of the Kolanut Pod Oil

Table 3.1 presents the results for the physico-chemical characterization of the kolanut pod oil, including its proximate analysis (moisture, ash, carbon, and volatile matter contents), while Table 3.2 shows the ultimate analyses of the kolanut pod, its pod oil, biodiesel produced and the conventional petro-diesel fuel counterpart analysed.

Parameters	Determined Vales for oil
Refractive index @ 29ºC	1.4584
Moisture (%)	9.68
Density (g/ml)	0.927
Ash content @ 15ºC	0.69
Carbon content	5.53
Kinematic viscosity @ 40ºC (mm^2s^{-1})	32.994
Energy value (J/g)	29967
Flash point (ºC)	245
Fire point (ºC)	257
Cloud point (ºC)	19.90
Pour point (ºC)	14.59
Oil yield (%)	18.63
Acid value (mgKOH/g)	0.5549
Saponification (mgKOH/g	221.10
Peroxide value (meq/kg)	0.77
Iodine value (mg/100g)	18.80
Molecular weight (g/mol)	767.33
Volatile matter	84.10

From Table 3.1, the bio-oil yield produced was 18.63%, which was higher than the value (16%) obtained by Santharam et al. (2013) in the "Biodiesel production from tigernut (Cyperus esculentis) oil and characterization of its blen with petro-diesel". This was attributed to its low FFA (acid) value (0.5549 mgKOH/g) and the peroxide value (0.77meq/kg) compared to those of the tigernut with 8.97 mgKOH/g and 8.33meq/kg respectively. The high energy value and low ash content of the kolanut pod oil indicate that

the fuel sample would serve as a good feedstock for biofuel production since the inherent energy content would increase the combustion efficiency.

Table 3.2: Ultimate analysis of the kolanut pod oil, its biodiesel and the petro-diesel samples

S/n	Element	Determined Value Kolanut pod	Determined Value Kolanut pod oil	Determined Value kolanut pod oil biodiesel	Corresponding value for petro-diesel [1]	Unit
1	Carbon	46.28	42.91	39.90	83.20	% mass
2	Hydrogen	5.59	5.27	5.40	13.39	% mass
3	Nitrogen	0.90	0.45	2.01	1.28	% mass
4	Sulphur	0.10	0.10	0.12	0.14	% mass
5	Oxygen	46.44	47.55	48.88	1.99	% mass

Also, from Table 3.2, the ultimate analysis shows that the kolanut pod, the pod oil and the biodiesel produced from the pod oil have higher oxygen contents compared to the conventional petro-diesel counterpart, while the petro-diesel has higher carbon, hydrogen, nitrogen and sulphur contents than the kolanut pod samples. Consequently, since higher oxygen content supports combustion, it indicates that the pod oil and the associates are better fuel candidates for power plant operation than the petro- diesel fuel.

3.2 PHYSICO-CHEMICAL CHARACTERIZATION OF THE KOLANUT POD OIL BIODIESEL AND BLENDS

The results of the evaluated kolanut pod oil biodiesel, the blends and the petro-diesels are presented in Table 3.3.

Table 3.3: Characterization of the kolanut pod oil biodiesel and blends

S/N	Property	Instrument	Test Method [1]	ASTM Recommended Value[1]	Determined Values for Biodiesel Blends from Kolanut Pod Oil					Petro - diesel[1]	Unit
					B20	B40	B60	B80	B100		
1	Density @ 15°C	Pycnometer	IS:1448	860 – 900	862	865	867	873	880	835	Kg/m^3
2	Specific Gravity	Hydrometer	ISO:12185	0.85 – 0.88	0.88	0.87	0.87	0.86	0.86	0.85	-
3	Flash Point (Closed Cup)	Pensky Marten's Apparatus	D93	≥93	121	125	126	128	130	65	°C
4	Cloud Point	Thermometer	D2500	-3 – 12	6.5	7.3	7.7	8.0	8.5	5	°C
5	Kinematic Viscosity @ 40°C	Red Wood Viscometer	D445	1.9 – 6.0	3.43	3.50	3.55	3.57	3.65	3.4	mm^2/s
6	Pour Point	Thermometer	D97	-15 – 10	3.5	3.8	3.6	4.2	3.3	-35 – 15	°C
7	Heat of Combustion (Calorific Value)	Bomb Calorimeter	IS:1448	37 – 45	42.55	41.84	41.70	41.35	41.18	45.345	MJ/kg
8	Water Content	Weigh/Heat/Weigh	D2709	≤500	330	345	350	358	365	≤500	mg/kg
9	Methanol Content	3/27 Test	EN14110	≤0.2	0.15	0.15	0.16	0.16	0.15	-	% mass
10	Cetane Number	Cetane Number Scale	D613	45 – 65	46.1	47.5	48.5	50.6	51.8	46	-
11	Boiling Point	Thermometer	D1160	315 – 350	316	317	316	319	320	180 – 340	°C
12	Specific Heat capacity	Calorimeter	D6751	≥1.2	1.60	1.64	1.70	1.73	1.76	1.62	MJ/m^{3}K
13	Thermal Conductivity	the photopyroelectric technique (PPE)	D6751	≥0.12	0.147	0.146	0.144	0.142	0.153	0.115	W/m K
14	Fire Point	Thermometer	E14112	≥130	176	187	205	245	251	69	°C

Annual book of ASTM standards, petroleum products and lubricants (I–III), vols. 05.01–05. 03. Philadelphia, 1991.

From the Table, it is seen that all the fuel properties for the kolanut pod oil biodiesel and blends analysed were within the recommended ASTM and EN standard values. They performed better than the conventional diesel fuel in most of the parameters characterized. The specific gravities of 0.88, 0.87, 0.87, 0.86, and 0.86 for the B20, B40, B60, B80, and B100 respectively were within the Standard ASTM range of 0.85 – 0.88 specified for diesel [13]. Since density and other gravities are important parameters for diesel fuel injection systems, their values must be maintained within 3 tolerable limits (0.86 – 0.90 kg/m) to allow optimal air-to-fuel ratios for complete combustion.

Higher-density biodiesel blends outside the standard limits can lead to incomplete combustion and particulate matter emissions [14]. Their flash and fire points also were well above 0 the ≥93 and ≥130 C minimum ASTM recommended values and therefore posed no risk of fire outbreak in case of accidents or crashes. Moreover, the kinematic viscosity, heat of combustion (calorific values), specific heat capacity and thermal conductivity values of the biodiesel and

blends obtained which were within the recommended standard values indicated that their thermal properties were tolerable for diesel plant operations in comparison with the petro-diesel sample.

The biodiesels have higher cetane numbers and lower calorific values compared to the petro-diesel fuel and hence have higher torque and energy delivery. Although the fuel properties of the biodiesel and the blends were very close, the B100 performed marginally well in terms of its cetane number, specific heat capacity, fire point and thermal conductivity perspective [15].

3.3 SEM Analysis of Kolanut Oil

The plate below shows the image for the SEM analysis done on the extracted kolanut oil.

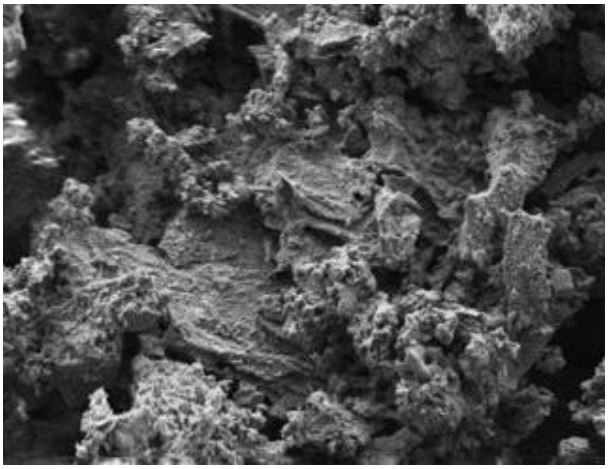

Figure 3.1: SEM image at 1000 magnification for kolanut oil

The SEM (Scanning Electron Microscopy) image shows a rough, irregular texture with numerous small pores and crevices. The pod's surface appears to be covered in tiny ridges and valleys, which may be indicative of the pod's natural defense mechanisms to protect it from external factors such as insects, fungi, and bacteria. The pores and crevices could also be responsible for the absorption of water and nutrients during the pod's growth process. Additionally, the SEM image highlights the presence of small hairs or trichomes on the surface of the pod, which may play a role in repelling insects or other unwanted organisms. Overall, the SEM result provides valuable insights into the intricate structure and surface features of the kolanut pod.

3.5 FTIR Result for Kolanut Pod Oil Extract

The plate below shows the image for the FTIR analysis done on the extracted kolanut oil.

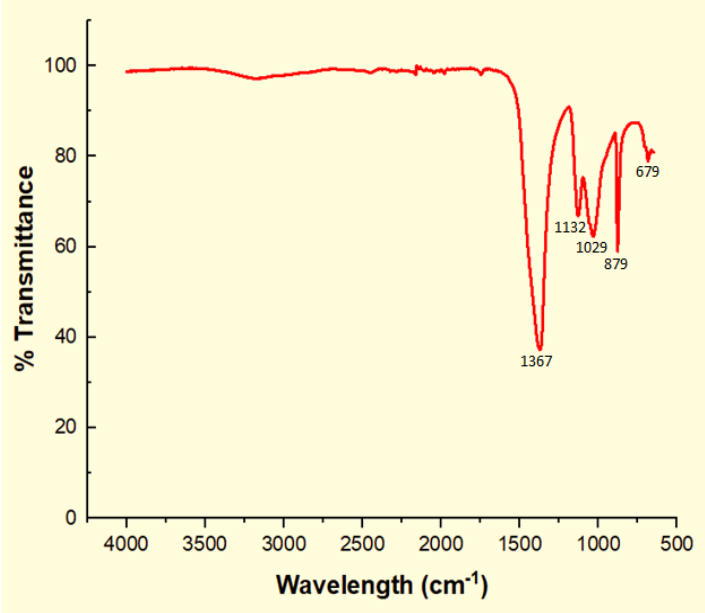

Figure 3.2: FTIR result for kolanut oil

The FTIR (Fourier Transform Infrared) result for the kolanut oil reveals a complex spectrum with several prominent peaks.

The peak at 679 cm-1 is attributed to the C-H stretching vibration of unsaturated fatty acids, indicating the presence of oleic and linoleic acids. The peak at 879 cm-1 is characteristic of the C-C stretching vibration of fatty acid chains, suggesting a high degree of unsaturation. The peak at 1029 cm-1 is associated with the C-O stretching vibration of ester functional groups, which is consistent with the presence of triglycerides in the oil. The peak at 1132 cm-1 is attributed to the C-C-C skeleton of fatty acid chains, while the peak at 1367 cm-1 is characteristic of the C-H bending vibration of fatty acids. Overall, the FTIR result suggests that the kolanut oil is rich in unsaturated fatty acids, particularly

oleic and linoleic acids, and has a high degree of triglyceride content.

4.0 CONCLUSION

Based on the phytochemical analysis, SEM, and FTIR results, the kolanut oil exhibits a promising potential as a biofuel source. The phytochemical analysis reveals the presence of various bioactive compounds, including flavonoids, phenolics, and alkaloids, which are indicative of the oil's ability to withstand oxidative degradation. The SEM analysis shows a rough, irregular texture with numerous small pores and crevices, which suggests that the oil may be suitable for use as a biofuel due to its potential to absorb and store energy. The FTIR results confirm the presence of unsaturated fatty acids, particularly oleic and linoleic acids, and triglycerides, which are characteristic of biofuel feedstocks. The combination of these results suggests that kolanut oil has a high potential to be used as a biofuel due to its chemical composition, physical properties, and resistance to degradation.

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