



EFFECTS OF Al_2O_3 NANOPARTICLES ON THE PHYSICAL AND THERMAL PROPERTIES OF TRANSESTERIFIED SENNATORA OIL

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Abstract

This study investigated the effects of Al_2O_3 nanoparticles on the physical and thermal properties of transesterified Sennatoro oil, aiming to enhance its combustion efficiency. The study also evaluates the oil's physical and thermal properties, both with and without the addition of Al_2O_3 nanoparticles. The methods adopted in the study include extraction of oil from Sennatoro seeds obtained from Kano, Northwest Nigeria, its purification and the synthesis of trans-esterified Sennatoro oil, and its characterization using Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). The composition of free fatty acids (FFAs) was investigated using Gas chromatography Mass Spectroscopy (GCMS). The results showed an acid value of 3.74mgKOH/g and pH of 5.8 for the crude oil and 0.45mgKOH/g and pH of 6.5 for the esterified oil. The addition of Al_2O_3 nanoparticles has increased pH to 6.9 while the flash point of the oil was observed to have decreased from 225°C to 218°C while the calorific value increased from 17.68MJ/kg to 19.45MJ/g. The major fatty acids contained oleic acid (30.73%), linoleic acid (23.41%), stearic acid (6.52%), and palmitic acid (11.54%). This work underscores the importance of exploring alternative bio-resources like Sennatoro to address global energy and environmental challenges effectively.

Keywords: Global Warming, Nanoparticles, Renewable Energy, Sennatoro oil, Transesterification.

1. INTRODUCTION

In recent times, reliance on the conventional fossil fuel in meeting up our energy demand has brought increasing challenges with underwhelm consequences. This manifests in the occasional increase in the price of petroleum products resulting in a hike in the cost of essential commodities and transport fares (El-Araby, 2024). At domestic level, kerosene and cooking gas have been replaced with firewood and charcoal for cooking by most of our people. The high cost of petro – diesel and insufficient electricity supply have also adversely affected the operation of our industries, agricultural machineries, business, and education sectors in Nigeria. At a global level, energy demand is also on the increase due to the population and industrial growth (Jamo et al., 2023).

The major global energy resources are petroleum, natural gas and coal which are non-renewable and susceptible to depletion. Apart from the fear of their depletion, their usage contributes to emissions of harmful gases leading to global warming (Singh et al., 2022; Abishek et al., 2024). Renewable energy has gained significant attention in recent years due to its potential to replace conventional fossil fuels (Gebremariam and Marchetti, 2017). As a result, there has been significant development in research in areas dealing with renewable energy resources such as those derived from plant, algae or animal waste due to their biodegradability, low cost and environmentally friendly (Nura et al., 2023). The oil extracted from plant seeds to produce biofuel include soya

beans, jatropha, rape seed, coco nut, castor beans, sunflower, palm oil, mustard, flax, and senna tora. It is a matter of concern that majority of the research conducted on senna tora plant focused on the medical or food benefits of the plant (Alam et al., 2021; Zibae et al., 2023) while neglecting its potential as non-edible biodiesel feedstock (Abubakar et al., 2021). Senna tora is a rainy season plant which grows abundantly in parts of Nigeria, China, Afghanistan, India, Pakistan, Nepal and other tropical countries of the world (Abubakar et al., 2021), and its common name is cassia tora under the family of fabacean. It is known as *Tafasa* in Hausa, *Jue Mingzi* in Chinese, *Chakvad* in Hindi, *Chakunda* in Bengali, and *Kawaria* in Gujurati (Raman Ibrahim et al., 2021). Sennatora is a non-edible oilseed crop that has shown promise as a source of biodiesel due to its high oil content and potential for cultivation in arid regions (Zibae et al., 2023).



Figure 1: Sennatora plant



Figure 2: Sennatora seeds

Although biodiesel has emerged as an alternative to conventional diesel fuels due to its low emission of greenhouse gases and sustainability in nature (Atadashi et al., 2013b), conventional biodiesel has some inherent drawbacks, such as high viscosity, low oxidative and thermal stability (Tomar et al., 2019). In recent years, nano particles have shown great promise in enhancing the properties of biodiesel due to their unique physical and chemical properties. In this context, the present study aims to investigate the effects of Al_2O_3 nano particles on the physical and thermal properties of trans-esterified senna tora oil. This study will provide insights into the potential of nanoparticles to improve the

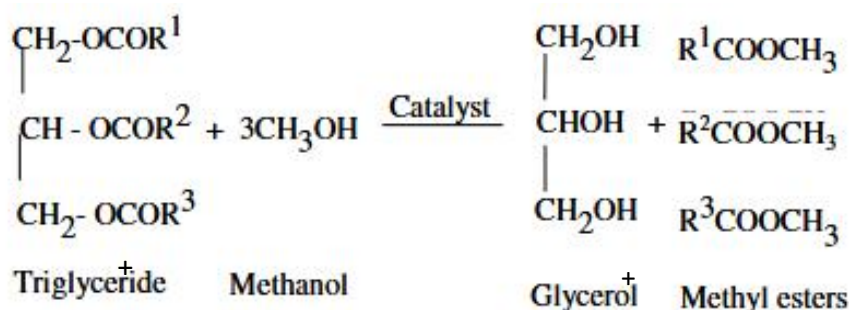
properties of biodiesel, thereby expanding its use as a renewable energy source.

2.0 Literature Review

Biofuel is a renewable energy source that can be obtained from plants, algae or animal waste. The seeds of Senna tora plant have been classified among the twenty-seven potential non-edible biodiesel feedstocks. The global production and consumption of biodiesel have increased, with a

record showing 71.5 million tons of production in 2023 (El-Araby, 2024).

Transesterification – is a process for which fat or oil reacts with alcohol to form esters and glycerol. A catalyst is used to improve the reaction rate and yield. The alcohol used is methanol. Using methanol in the process results in producing biodiesel and glycerol as by-product. Triglyceride is converted into Fatty Acid and Methyl Esters (FAME).



Scheme 1. A typical transesterification reaction.

In the transesterification reaction, the R'' group of alcohol is exchanged with R' group of esters. This is achieved via introduction of a catalyst. Various homogeneous and heterogeneous catalysts, ranging from base, acid to enzyme, have been developed for use. Examples of catalysts for the process include potassium hydroxide, sodium hydroxide, calcium oxide, neon oxide, trifluoroacetic acid, sulfuric acid, and eggshell (Jamo et al., 2023). Potassium hydroxide (KOH) and sodium hydroxide (NaOH) are among homogeneous catalysts and the mostly used (Baskar et al., 2018; Interlenghi et al., 2023). The use of alkaline catalysts has an advantage over acid catalysts in terms of reaction rate to the extent of about 4000 times faster and for every 100 units of biodiesel fuel produced using transesterification method, there will be 11 units of glycerin as by-product (Atadashi et al., 2013a).

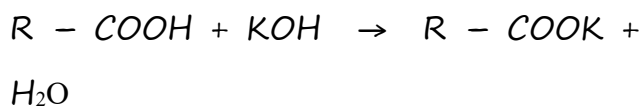
Nanoparticles – Nanoparticles are particles with diameter of the order of nanometers (nm), ranging from 1 to 100nm (1nm = 10⁻⁹m). Nanoparticles can occur naturally or be produced purposefully through engineering to perform a specific function. One of the methods for the production nanoparticles is 'Sol-gel method' (Saravanan and Dubey, 2020). Addition of nanoparticles (with low concentration) to lubricating oils, reduces friction and wear, better efficiency and durability of the equipment (Harsh Gupta and Anand Santosh Kumar Rai, 2021). Specifically, the addition of Al₂O₃ nanoparticles help in reducing viscosity, ignition delay, fuel consumption and carbon monoxide emissions (Kayed et al., 2024)

The addition of Al₂O₃ nano particles improves stability of biofuel over wider range of conditions and enhances engine efficiency by reducing its viscosity and flash point and increasing cetane number.

Cetane number (CN)

The cetane number (CN), is an indication for the time-lapse of compression-ignition engines between fuel injection and ignition (Lin and Wu, 2022). Higher concentrations of saturated fatty acids and longer carbon chains increase CN while higher levels of unsaturated fatty acids tend to lower the CN. Ignition of engine is easier with higher CN in biodiesel.

Acid number (AN) - The acid number test is one of the methods used to estimate the amount of additive depletion, acidic contamination and by-product of oxidation. AN can be measured by the amount of base such as potassium hydroxide (KOH) required to neutralize the acid in one gram of oil sample.



Free fatty acid (FFA) – Free fatty acids are non-esterified fatty acids that are released by the hydrolysis molecules bound to glycerol. Oils with high levels of FFA are more susceptible to oxidative aging. The FFA of oil can be reduced through esterification process by its pre-treatment with methanol/oil ratio (6:1), in the presence of H₂SO₄ catalyst (1.6 v/v) in 93 minutes at 45°C (Morakinyo et al., 2021).

3.0 MATERIALS and METHODS

3.11 Chemicals:

Crude Sennatora oil, ethanol, diethyl ether, phenolphthalein indicator, methanol, 0.1M of potassium hydroxide solution, hydrochloric acid, distilled water, and aluminium oxide powder (Al₂O₃).

3.12 Apparatus: Retort stands with clamp, measuring cylinder, burettes, beakers, conical flasks, sampling bottles, Thermostat Oven DHG-9023A, TS- 200 electronic Compact Scale, Pec Medical magnetic stirrer model 78 - 1, GCMS-

QP2010 Plus Shimadzu Japan, thermometer, funnel, separation funnels, density bottle, filter paper, glass rod, and calorimeter.

3.13 Safety equipment:

Chemical proof hand gloves, eye goggles, face mask, detergent, water, duster, and recycle bin.

3.2 EXPERIMENTAL PROCEDURES

3.21 Determination of acid value

A mixture of ethanol and diethyl ether was used as solvent for the crude Sennatora oil. 15g of the oil was added to the mixture in a conical flask. 5 drops of phenolphthalein indicator were added to the mixture and shaken. A burette was filled with 0.1M of potassium hydroxide solution. Initial volume was recorded. Titration was done against the mixture until the color changed to pink from yellowish. The final volume was recorded. The process was repeated to obtain an average value of the titre (Lin and Wu, 2022).

3.22 Purification process

200 ml of the senna tora oil is measured using measuring-cylinder; then preheated to 70 °C using hot magnet stirrer with thermometer. 14ml hydrochloric acid mixed with 300 ml of methanol also measured and added to the heated oil sample and continuously heated and stirred for one hour at 50 °C. The mixture was then transferred to the vacuum oven and heated at 70 °C for 45 minutes (Jamo et al., 2023). The mixture was poured into separation funnel to settle for 6 hours. The upper layer which contained the purified oil was collected in a beaker. The acid value of 15 g of the purified oil was obtained using the same method for the crude oil. The average value of the titre was recorded.

3.23 Transesterification

100 ml of the purified Sennatora oil was preheated at 50 °C. 2.6 g of KOH pellets are dissolved in 400 ml of methanol. The mixture is

transferred to the preheated oil and stirred using the magnetic stirrer. The heating and stirring continued for one and a half hours at 55°C. The mixture then transferred to the separating funnel for 24 hours to settle and formed two layers. The lower layer was drained in a beaker and the upper layer which contains the biodiesel was collected in a conical flask.

3.24 Nanofluid preparation

2 g of Al₂O₃ nanoparticles powder was dispersed in 250 g of the transesterified Sennatora oil and stirred for 30 minutes at 40 °C using magnetic stirrer.

3.24 Gas Chromatography Mass Spectrometry (GCMS): The sample was analyzed using Shimadzu QP2010 Japan with total flow of 40.8 ml per minute and 1.8 ml per minute as column flow. The column oven temperature was at 70 °C, and 250 °C as injection temperature while the retention time was between 15 and 20 minutes at pressure of 116.9 kPa.

3.25 Fourier Transform Infrared (FTIR)

All spectra were obtained using FTIR-Agilent Technologies with Happ-Genzel apodization.

The range of (4000 – 650) cm⁻¹ were observed at resolution of 8cm⁻¹. (Morakinyo et al., 2021) used ATR-FTIR of Thermo Scientific with a resolution of 4 cm⁻¹ in the range of 4000 – 650 cm⁻¹ to obtain absorption peaks for cassia tora oil.

4.0. RESULTS AND DISCUSSION

Determination of acid value of crude oil is very important before transesterification process. It measures amount of free fatty acids (FFAs) in the oil. Table 1 contained the acid value of the crude sennatora oil, which showed a high value of 3.74 mgKOH/g. High values of FFAs lower quality of biodiesel causing engine corrosion and fuel system damage. Esterification process was used to reduce concentration of FFAs in oil, as shown in Table 2. A burette was filled with 0.1M of potassium hydroxide solution. Initial volume was recorded. Titration was done against the mixture until the color changed to pink from yellowish. The final volume was recorded. The process was repeated to obtain an average value.

Table 1: Determination of acid value for the crude sennatora oil.

| Burette readings | 1 st titration | 2 nd titration |
|---|---------------------------|---------------------------|
| Initial reading/cm ³ | 50.00 | 39.90 |
| Final reading/cm ³ | 39.90 | 30.00 |
| Volume used/cm ³ = (initial – final) | 10.10 | 9.90 |
| Average volume used/cm ³ | 10.00 | |
| Calculation of the acid value | 3.74 mgKOH/g | |
| $Acid\ value = \frac{10.00 \times 0.1 \times 56.1}{15}$ | | |

From table 1, an acid value of 3.74 mgKOH/g was obtained. The value is very high compared to the standard of <0.5 mgKOH/g. The negative effects of oil with high acid value include saponification and difficulty in separating biodiesel from glyceride after transesterification

process (Gomes et al., 2023). There is therefore the need for pretreatment (esterification/purification) before carrying out transesterification process. Table 2 showed reduction in acid value to 0.4488 mgKOH/g which fell within the standard.

Table 2: Determination of acid value for the purified (esterified) oil.

| | | |
|--|---------------------------|---------------------------|
| Burette readings | 1 st titration | 2 nd titration |
| Initial reading/cm ³ | 50.00 | 48.70 |
| Final reading/cm ³ | 48.70 | 47.60 |
| Volume used/cm ³ = (initial – final) | 1.30 | 1.10 |
| Average volume used/cm ³ | 1.20 | |
| Calculation of the acid value | | |
| $Acid\ value = \frac{1.20 \times 0.1 \times 56.1}{15}$ | 0.4488 mgKOH/g | |

Table 3: Characterization of Crude, Purified, Transesterified and Al₂O₃ Nanoparticles Dispersed Oil.

| Sample ID | Acid value/ (mgKOH/g) | Density/ g/cm ³ | pH value | Flash point/ °C | Pour point/ °C | Cloud point/ °C | Cetane number | Calorific value/ MJ/kg |
|--|---------------------------|-------------------------------|-------------|-----------------------|----------------------|-----------------------|------------------|------------------------------|
| Crude oil | 3.74 | 0.987 | 5.8 | | | | | |
| Purified oil | 0.45 | 0.920 | 6.5 | 225 | -7.0 | 0.1 | 48 | 17.68 |
| Transesterified oil | | 0.884 | 6.9 | 219 | -5.0 | 0.2 | 50 | 19.22 |
| 0.4% Al ₂ O ₃ nanoparticles dispersed | | 0.889 | 6.9 | 218 | -5.2 | 0.4 | 52 | 19.45 |
| Standard for biodiesel | <0.5 | 0.860 – 0.900 | 6.5 – 9.5 | 100 – 170 | -1.5 – 16 | -3.0 – 12.0 | 48 – 60 | 37 – 41 |

From Table 3, the acid value of crude oil is 3.74 with 0.884 g/cm³. The flash point obtained mgKOH/g which is far beyond standard value of indicated 218^oC which is beyond the standard <0.5 mgKOH/g for biodiesel. The purification range of 100 – 170^oC. However, the cloud point process (esterification) reduced it to 0.45 of 0.4^oC, pour point of -5.2^oC, and cetane mgKOH/g which falls within standard value. number of 52 obtained fell within standards for The process has also reduced the density of the biodiesel. The addition of 0.4% Al₂O₃ oil to 0.92 g/cm³ and increased pH to 6.5 Other nanoparticles has lowered the pour and flash researchers obtained 2.5 mgKOH/g and 0.38 points but has increased the cloud point, cetane mgKOH/g for crude oil and purified oil number and calorific value. The concentration of respectively (Morakinyo et al., 2021). (Abubakar Al₂O₃ in the nanoparticles from table 4, et al., 2021) obtained a density of 0.848 g/cm³ indicated 92.879% followed by ZnO with for the transesterified oil very close to this work 1.1500% and SiO₂ with 1.015%.

Table 4: XRF OF Al₂O₃ nanoparticles

| Components | Ni ₂ O | Ga ₂ O ₃ | K ₂ O | CuO | ZnO | TiO ₂ | Rb ₂ O | Al ₂ O ₃ | SiO ₂ | MgO | CaO | Na ₂ O |
|------------------|-------------------|--------------------------------|------------------|-------|--------|------------------|-------------------|--------------------------------|------------------|-------|-------|-------------------|
| % Composition | 0.0120 | 0.1129 | 0.0786 | 0.252 | 1.1500 | 0.160 | 0.061 | 92.879 | 1.015 | 0.520 | 2.015 | 0.095 |

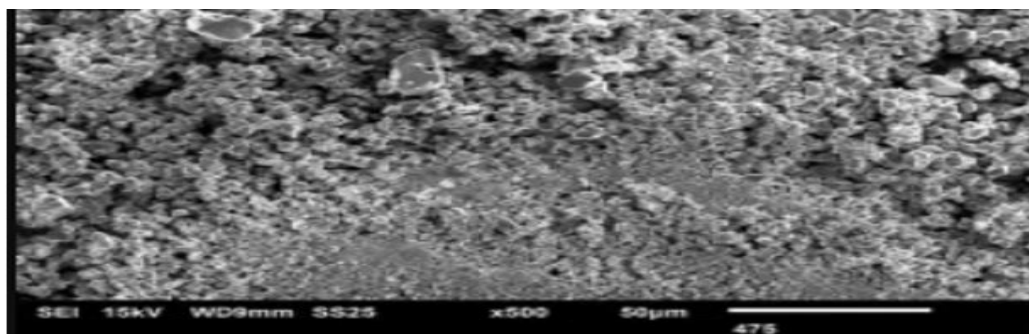


Figure 3: SEM of Al₂O₃

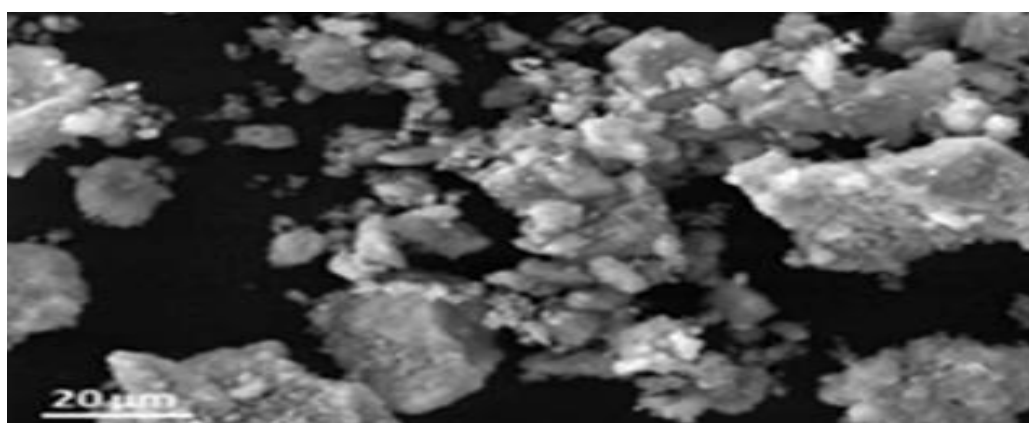


Figure 4: SEM of Al₂O₃ showing oval shape of the nanoparticles.

Gas chromatography mass spectrometry (GCMS) of the oil by separating and quantifying each was used to determine the purity and combustion individual fatty acid esters present. Figure 5 efficiency of oil. It determines the exact profile shows the peaks for biodiesel from sennatora oil.

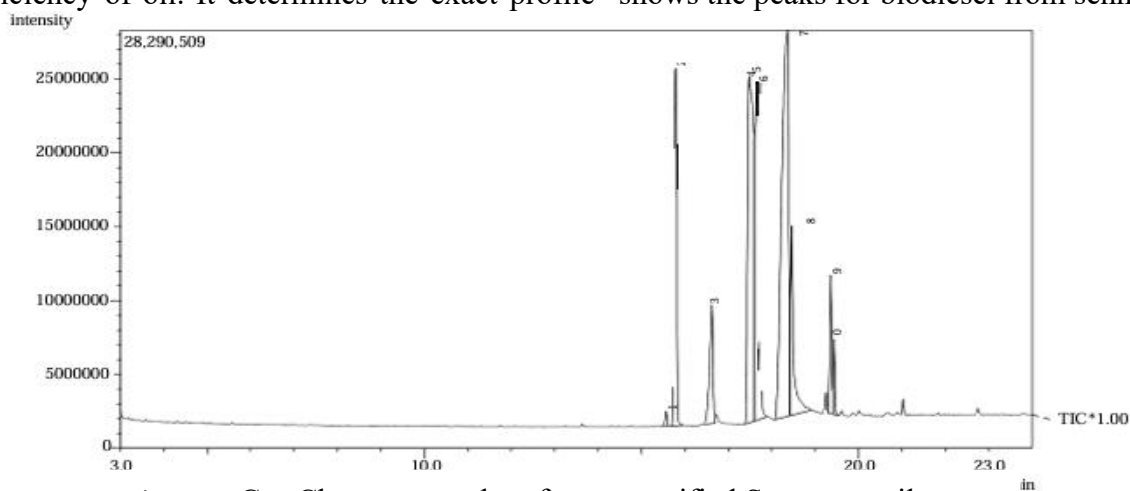


Figure 5: Gas Chromatography of transesterified Sennatora oil

Table 5: Specifications for Gas Chromatography and Mass Spectroscopy of Sennatora oil

| Parameter | Value |
|-------------------------|-------------------------|
| Instrument | Shimadzu QP2010 (Japan) |
| Total flow rate | 40.8 ml/min |
| Column flow rate | 1.8 ml/min |
| Column oven temperature | 70°C |
| Injection temperature | 250°C |
| Retention time | 15 – 20 minutes |
| Pressure | 116.9 kPa |

Table 5 contained specifications used in Total Ion Chromatogram (TIC) and graph plots. obtaining the peaks in figure 5. and Table 6 Peak 2 for palmitic acid, peak 4 for linoleic acid, contained descriptions of the major constituents peak 7 for oleic acid, and peak 8 for stearic acid of fatty acids found using the Peak Report from

Table 6: Analysis for Gas Chromatography and Mass Spectroscopy of Sennatora oil

| Compound name | Chemical formular | Molecular weight | Retention time /min | % peak height | % Area | % Composition |
|-------------------------|--|------------------|---------------------|---------------|--------|---------------|
| Peak 1 Linoleic acid | C ₁₉ H ₃₄ O ₂ | 294 | 17.486 | 15.07 | 23.41 | 23.41 |
| Peak 3 Palmitic acid | C ₁₆ H ₃₂ O ₂ | 256 | 15.792 | 15.56 | 11.54 | 11.54 |
| Peak 7 Oleic acid | C ₁₈ H ₃₄ O ₂ | 282 | 18.368 | 16.78 | 30.73 | 30.73 |
| Peak 8 Stearic acid | C ₁₈ H ₃₆ O ₂ | 284 | 18.459 | 8.25 | 6.92 | 6.92 |

From table 6, oleic acid constituted largest quantity with 30.73%, followed by linoleic acid with 23.41%, palmitic acid with 11.54% while stearic acid constituted least quantity with 6.92%. Figures 6, 7, 8, and 9 represent the structures of fatty acids obtained from the visual TIC graphs

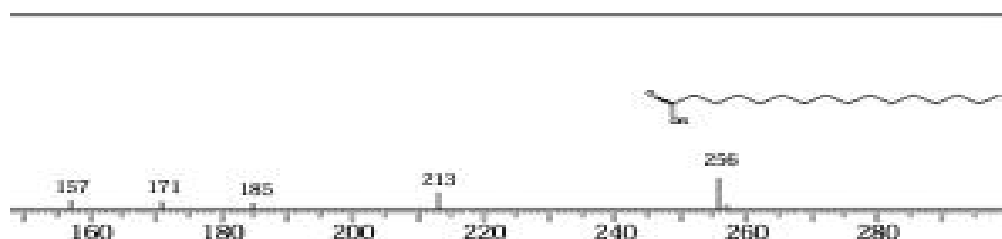


Figure 6: showing structure of Palmitic acid

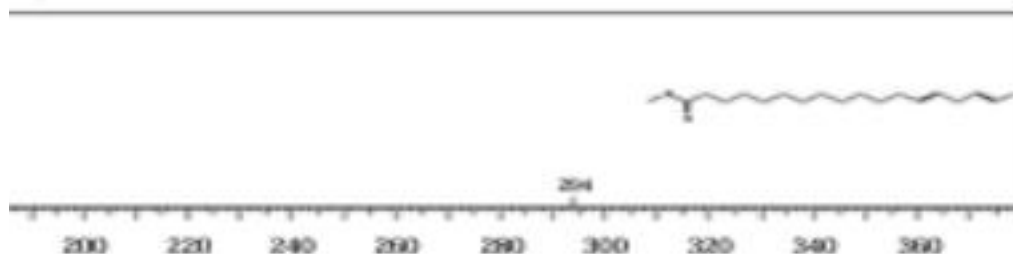


Figure 7: showing structure of Linoleic acid

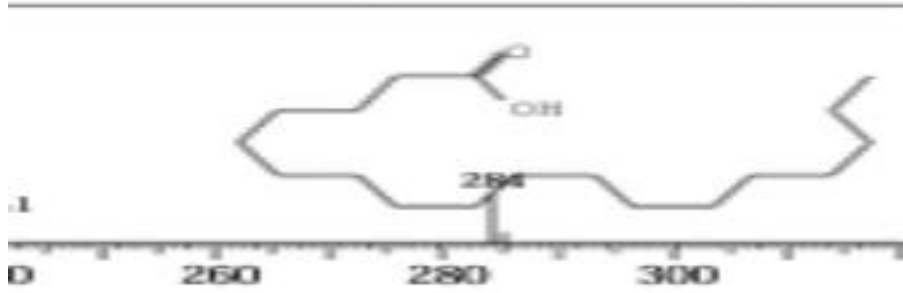


Figure 8: showing structure of Stearic acid

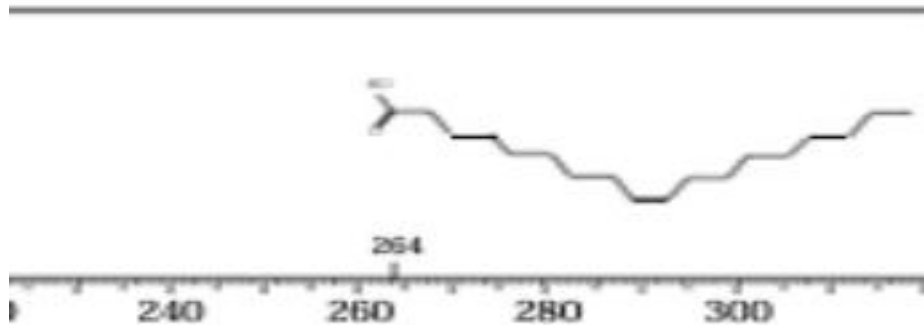


Figure 9: showing structure of Oleic acid

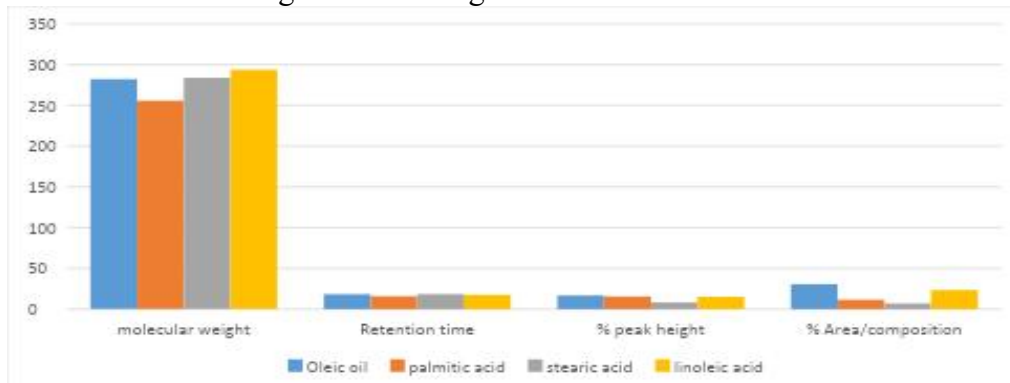


Figure 10: Comparison of molecular weight, retention time, % peak height and % composition of fatty acids.

The above analysis shows that the sample and bioactive phenolic compounds. Fatty acids contains a complex mixture of fatty acid consist of saturated and unsaturated methyl esters (FAMES), known as biodiesel compounds. From Table 7, the unsaturated which is a typical transesterified oil. GC-MS is fatty acids constituted the largest percentage profiles of oil primarily focusing on fatty acids

Table 7: The major fatty acids found in transesterified sennatora oil:

| Fatty acid | Type | % Composition | Biodiesel Standard |
|---------------|------------------|---------------|--------------------|
| Oleic acid | mono-unsaturated | 30.73 | 13% - 43% |
| Linoleic acid | poly-unsaturated | 23.41 | 11% - 30% |

| | | | |
|---------------|-----------|-------|-----------|
| Palmitic acid | Saturated | 11.54 | 12% - 27% |
| Stearic acid | Saturated | 6.52 | 2% - 17% |

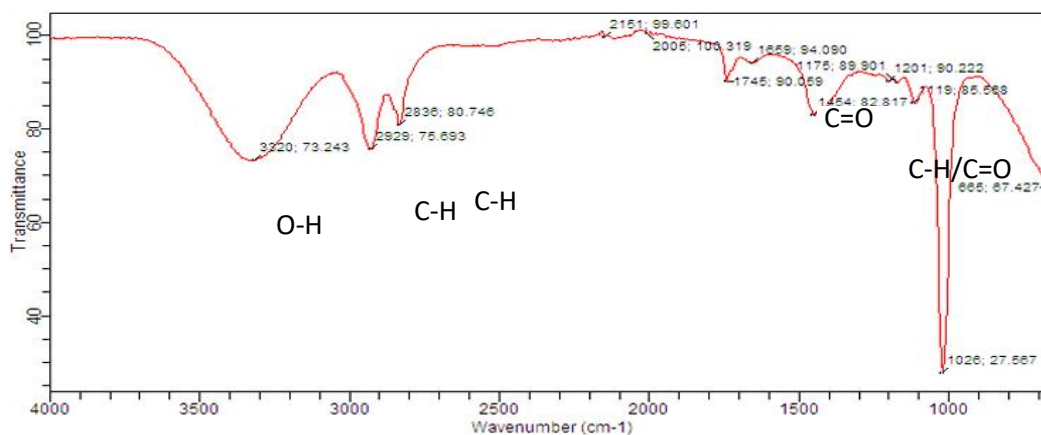


Figure 11: FTIR spectra of transesterified sennatoro oil with addition of Al₂O₃ nanoparticle.

At wave number of 3220cm⁻¹, there is O-H stretching with a broad peak indicating surface hydroxyl of Al-OH group. In 2929 and 2836 cm⁻¹, there are C-H stretching vibrations indicating the presence of 2-methoxyl ethanol while in 1745 cm⁻¹, there is carbon oxygen double bond stretching vibrations which indicate an ester. 1454 cm⁻¹ indicates bending vibrations due hydrogen - oxygen and carbon – oxygen respectively. 1026 cm⁻¹ is the fingerprint showing strongest absorption peak. It is C-O stretching vibrations in ether. 665 cm⁻¹ is low frequency peak representing heavy metal-oxide vibrations or ‘rocking’ motion of the molecule.

CONCLUSION

The present study has demonstrated the potential of Sennatoro plant as feedstock for biofuel. The addition of Al₂O₃ nanoparticles has enhanced the efficiency of transesterified oil in terms of calorific value and combustion capacity. On the physical effects, the results showed an acid value of 3.74mgKOH/g which decreased to 0.45mgKOH/g for the esterified oil while pH of 5.8 for the crude oil increased to pH of 6.5 for the esterified oil and pH of 6.9 with the addition

of Al₂O₃ nanoparticles. On the thermal effects, flash point of the oil was observed to have decreased from 225⁰C to 218⁰C while the calorific value increased from 17.68MJ/kg to 19.45MJ/g with the addition of Al₂O₃ nanoparticles. The major fatty acids found include oleic acid (30.73%), linoleic acid (23.41%), palmitic acid (11.54%), and stearic acid (6.52%). This work emphasizes the importance of exploring alternative bio-resources like Sennatoro to address global energy and environmental challenges effectively.

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