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Synthesis and Characterization of Biodiesel from Palm Oil as Alternative Fuel for Diesel Engine Using Potassium Hydroxide as Catalyst

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Abstract

Biodiesel was synthesized from palm oil by alkali-catalyzed trans-esterification using potassium hydroxide as the catalyst yielding palm oil biodiesel of 69.6 %, 72.4 %, 77.1 %, 71.3 % and 75.6 % from five palm oil samples. The biodiesel produced from the five samples was characterized by some of its physicochemical properties such as specific gravity, flash point, cloud point, pour point, aniline point and cetane number. The following are the results of the physicochemical properties determined specific gravity 0.876,0.884, 0.887, 0.884, 0.889, flash point 78^0 C, 79 ^{0} C Cloud point 10.3^0 C, 8.6^0 C,10.6^0 C,10.4^0 C,11.7 ^{0} C, pour point 2.6^0 C1.3^0 C 2.7 ^{0} C, aniline point 17.1^0 C, 20.0^0 C, and cetane number 48, 55, 62, 58, 48 respectively. The GC-MS analysis of the samples shows that four methyl esters were confirmed in two of the biodiesel sample while five methyl esters were confirmed in three of the biodiesel samples. The results obtained were in conformity with American Standard Testing Method (ASTM) standard D6571 and European (EN) standard 14214 and it implies that palm oil can be used to produce biodiesel.

Keywords: biodiesel, palm oil, transesterification, gas chromatography, methyl esters

INTRODUCTION

The increasing global demand for energy and materials, driven by industrialization and population growth, poses significant economic, environmental, and social challenges (Bentley *et al.,* 2007). Fossil fuels serve as the main sources of energy and chemicals globally, are being extracted at a rate faster than they can naturally regenerate. This unsustainable pattern is exacerbated by the concentration of these resources in a few countries, leading to environmental issues with profound global implications (Greene, 2004). Experts concur that greenhouse gas emissions, like CO_2, CH_4, and N_2O resulting from non-renewable energy sources burning and human-driven land utilization alterations, are destabilizing Earth's climate. (Forster *et al.,* 2007). To tackle these issues, there is an increasing agreement on the necessity to shift from geological deposits fuels to sustainable energy sources, which are more widely distributed and pose fewer environmental and social issues. Biomass resources, in particular, hold promise as they are abundant and readily available in many countries.

The evolution of societies has brought about a rise in energy demands, primarily met through combusting fossil fuels like oil, coal, and natural gas. However, these energy sources are non-renewable and contribute to environmental issues. As a result, there has been a push towards

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renewable energy sources such as biofuels, which are non-toxic and biodegradable (Demirbas, 2003).

Biodiesel is a biofuel generated from renewable biological materials like vegetable oils and animal fats, provides potential to lessen dependence on petroleum fuels and mitigate air pollutant emissions. In response to diminishing petroleum reserves, rising fuel costs, and environmental concerns, many countries is increasingly interested in using biodiesel as a fuel for diesel-powered vehicles (Lapueta and Rodriguez, 2005). In recent years, biodiesel production from abundant bio-sources has garnered significant attention from both academics and the industrial sector (Hayan *et al.,* 2010).

Pure biodiesel, known as B100, can be blended with conventional diesel, labeled as BXX, where XX is the percentage of biodiesel in the blend. A typical example is B20, which contains 20% biodiesel mixed with 80% diesel fuel. Biodiesel mainly consists of fatty acid methyl (or ethyl) esters sourced from renewable materials like veggies oils and used restaurant greases. One of biodiesel's appealing traits is that it can be utilized in diesel engines without needing substantial alterations, meaning the engine does not need to be specifically designed for biodiesel use (Tat and Van 2002). When compared to diesel fuel, biodiesel offers several advantages such as reduced sulfur concentration, higher ignition point, lower aromatic compounds, and improved ability to decompose naturally. Additionally, Biodiesel generates fewer emissions compared to petroleum diesel and does not add to the overall concentration of carbon dioxide in the air, thus aiding to the reduction of the greenhouse effect. (Ahmia *et al.,* 2014).

The oil palms (*Elaeis*) belong to the Arecaceae family and consist of two species commonly used in commercial agriculture for palm oil production. *Elaeis guineesis* has pinnate leaves and dense clusters of closely packed flowers. Its vibrant red fruits yield high-quality palm oil (Olorunfemi *et al.,* 2014). Palm Oil naturally appears reddish due to its high β-carotene content. It is among the few vegetable fats that are highly saturated. Palm oil is in partially solid state at ambient temperature because of its triacylglycerol content, primarily palmitic and oleic acids, although it can be liquid in warmer climates. Large-scale industrial cultivation of palm oil began after World War I, drawing on knowledge gained from Sumatra's plantations (USDA, 2009). Oil palm varieties are classified based on their fruit form, color, composition, and leaf shape.

A study by Okpanachi *et al.*(2017) on the physicochemical properties of biodiesel made from waste vegetable oil in Sedi Minna, it was found that the biodiesel met the necessary standards and could be used in diesel engines.

Danlami *et al.*(2022) investigated and he physical and chemical properties of biodiesel produced from Shea butter. The study demonstrated that high quality Shea butter biodiesel was successfully manufactured and the physicochemical properties were within the accepted range for standard biodiesel.

In this research biodiesel was Synthesized from Palm oil using potassium hydroxide as catalyst and the physicochemical properties of the Synthesized biodiesel was determined. The biodiesel was also characterized using GS-MS to detect methyl esters present in the biodiesel. The results shows that Palm oil is a good source for biodiesel production.

METHODOLOGY

Synthesis of Biodiesel

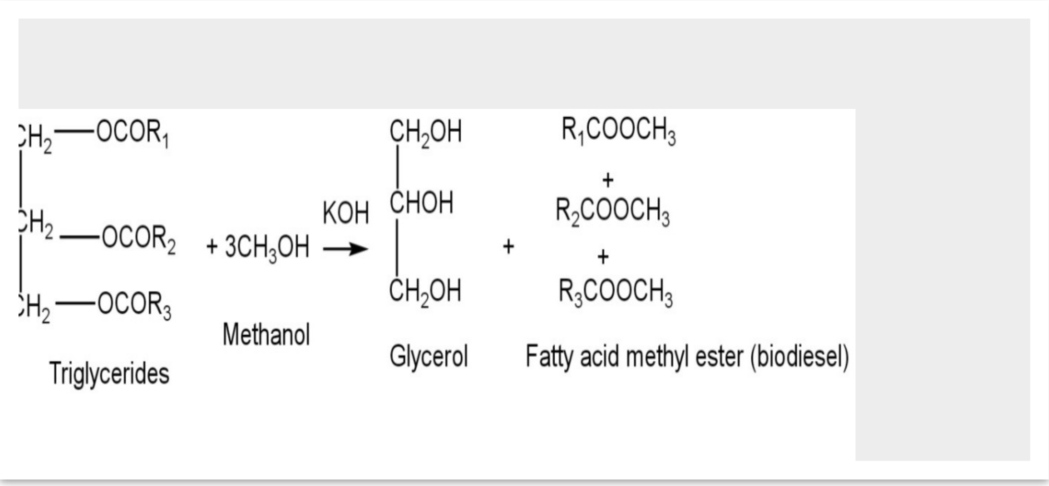
Before synthesizing biodiesel, the palm oil samples must be pretreated with acid to lower their high free fatty acid concentration.

Pretreatment of Palm Oil Samples

The palm oil samples were pretreated through an esterification reaction. First, 150 ml of palm oil was warmed on a hot plate at 60°C. Next, 300 mL of methanol and 2 mL of concentrated sulfuric acid (H_2SO_4) catalyst were added simultaneously. The mixture was heated and stirred with a magnetic stirrer for two hours. After the reaction, it was transferred to a separating funnel and rinsed with distilled water. The mixture was shaken and left to stand for about 30 minutes to separate into two distinct layers. The lower layer of washing water was disposed of, and the upper layer was collected in a glass beaker for trans-esterification.

Trans-esterification Of Palm Oil Using Potassium Hydroxide.

Transesterification is the reaction of vegetable oils or animal fats with an alcohol in the presence of a catalyst to produce alkyl esters and glycerol,the alkyl esters are called biodiesel. The trans-esterification reaction was started by dissolving 4 grams of KOH in 300 mL of methanol and stirring the mixture in a three-neck flask for 30 minutes. Next, 100 mL of oil obtained from the esterification was heated to 105°C, which is above the boiling point of water, for 1 hour. After cooling to 50°C, the oil was combined with the catalyst and methanol mixture and stirred at 60°C for 3 hours. After the reaction was finished, the flask was cooled in cold water, and the mixture was poured into a separating funnel and left at room temperature overnight to allow it to separate into two layers. The lower layer was glycerol, and the upper layer was crude biodiesel.



Equation for transesterification process.

Purification of Biodiesel

The crude biodiesel was purified by washing it with warm distilled water to eliminate inorganic impurities and soaps. Excess methanol was subsequently evaporated by heating the product to 100°C, which is above methanol's boiling point (Aladetuyi *et al.*, 2014). To further purify, Fifty milliliters of hot distilled water were added to 100 mL of crude biodiesel in a separating funnel. The water, which contained dissolved impurities, settled at the bottom, while the washed biodiesel stayed on top. The separated biodiesel was then heated to 110°C to remove any residual water. After cooling, the volume of the purified biodiesel was measured, and the yield (y) was calculated using the given equation below (Alamu *et al.*, 2007; Ribeiro *et al.*, 2014).

$$Yield = \frac{Volume of biodiesel}{Volume of sample} \times 100$$

Determination of Physicochemical Properties of Synthesized Biodiesel

i. Determination of Cloud Point

A 10 cm^3 sample of the synthesized biodiesel was placed in a medium-sized test tube, which was then positioned in a test tube rack and stored in a refrigerator for observation. The temperature at which the heavier components of the biodiesel started to form a mass of colloids, known as the cloud point, was recorded using a mercury-in-glass thermometer (Danlami *et al.*, 2022).

ii. Determination of Flash/Fire Point

The ignition points were measured by transferring the biodiesel sample into a petri dish and covering its surface. A mercury-in-glass thermometer was placed in the sample, with its tip just touching the biodiesel. The thermometer was secured with a retort stand and clamp. The petri dish was then placed on a laboratory heating mantle and heated gradually, with a light source applied intermittently. The flash point was noted as the lowest temperature at which the sample briefly ignited and then extinguished (Dasin *et al.*, 2022).

iii. Determination of Aniline Point

The aniline point was calculated according to ASTM D611. Ten cm^3 of aniline oil was added to a test tube, followed by 10 cm^3 of the synthesized biodiesel. Initially, the two samples did not mix and formed separate layers. The test tube was then set in a test tube rack and stored in a refrigerator. The aniline point was recorded as the lowest temperature at which the aniline oil and biodiesel fully mixed (Reda, 2014).

iv. Determination of API Gravity

The American Petroleum Institute (API) gravity was calculated based on the specific gravity of the biodiesel using the formula provided below (Danlami *et al.*, 2022).

$$API = \frac{141.5}{(SPECIFIC GRAVITY AT 60°F) - 131.5}$$

v. Determination of Cetane Number/Cetane index

The cetane index measures the ignition quality of diesel fuel, with a high index indicating that the fuel ignites more easily in a standard diesel engine. The Cetane index was calculated using the Aniline point and API gravity values of the biodiesel, following equation as outlined by Danlami *et al.* (2022).

$$\text{Cetane number} = \frac{Aniline point (°C) \times API\ Gravity}{10}$$

vi. Determination of Pour point

The same samples used for measuring the cloud point were placed in ice to solidify. Once solidification was confirmed, the test tube was removed and tilted, with close observation until the solidified biodiesel began to flow. The pour point temperature was recorded as the temperature at which the biodiesel began to flow.(Dasin et al., 2019)

Chromatographic Analysis (Using Gas Chromatograhy Mass Spectrometry)

The biodiesel sample was diluted with hexane and analyzed using a Varian gas chromatograph equipped with a flame ionization detector, 30m×0.25mm×0.25mm capillary columns, and a split injection system with a 1:20 ratio. The injector and detector temperatures were set to 280°C and 300°C, respectively. The oven was initially set to 200°C for 45 minutes, then ramped up to 250°C at a rate of 20°C per minute and maintained at that temperature for five minutes. Hydrogen was used as the carrier gas at a flow rate of 2 mL per minute, with the column pressure adjusted to 20 psi. Automatic peak integration was managed by a computer running Star Workstation 6.2, connected to the GC via a Star 800 Module interface. Methyl heptadecanoate was used as the internal standard (Matassoli *et al.,* 2009).

RESULTS AND DISCUSSION

Synthesis of biodiesel

Biodiesel was synthesized by pretreating the palm oil first through esterification procedure to reduce the high free fatty acid to less than 1% followed by the transesterification process to produce biodiesel, which yields percentage results as shown in the table 1

The yield of palm biodiesel synthesized through Trans esterification after being pretreated to reduce the excess free fatty acid to less than 1%. The five samples ranges from 69.6%-77.1% as presented in table 1 in which sample P3 has the highest yield of biodiesel and sample P1 has the lowest yield of biodiesel, all the sample percentage yields are within the limit of the ASTM D6751 standard of yielding of more than 30 %.

Table 1: Percentage yield of biodiesel

Samples	%Yield
P1	69.6
P2	72.4
P3	77.1
P4	71.3
P5	75.6

The physicochemical properties of palm biodiesel, including specific gravity, flash point, cloud point, pour point, aniline point, and cetane number, are detailed in Table 2.

Table 2. Physico - chemical properties of synthesized palm biodiesel.

Samples	Specific Gravity	Flash Point (^{0}c)	Cloud Point (^{0}c)	Pour Point (^{0}c)	Aniline Point (^{0}c)	API Gravity	Cetane Number
P1	0.876	78	10.3	2.6	17.1	29.9	48
P2	0.887	79	8.6	1.3	18.4	28.1	55
P3	0.884	78	10.6	2.4	21.7	28.6	62
P4	0.889	78	10.4	1.9	21.0	27.6	58
P5	0.884	75	11.7	2.7	20.0	28.5	48

The cloud point is the temperature at which waxes in diesel or biodiesel start to form a cloudy appearance. The cloud points for the synthesized palm oil biodiesel ranged from 8.6°C to 10.6°C, as shown in Table 2. This range is higher than the values previously reported by Alang*et al.* (2018), Kalam and Masjuki (2002), and Aldo Okullo *et al.* (2012).

The flash point is a crucial property of fuels, indicating the temperature at which a fuel is prone to accidental ignition, thus reflecting the safety of its storage and handling. The flash point of the palm oil biodiesel in this study ranged from 75°C to 79°C, as shown in Table 2, which is lower than the 93°C and 110°C (Ejeh and Aderemi, 2014; Danlami*et al* 2022). This suggests a relatively high-risk biodiesel.

The specific gravity of the palm oil biodiesel ranged from 0.876 to 0.889, aligning with the specifications set by ASTM D6751 and EN 14214 for biodiesels. This result is consistent with the value of 0.87 reported by Benedict *et al.* (2018).

API gravity, which measures the density of petroleum liquids relative to water, ranged from 27.6 to 29.9 for the palm oil biodiesel samples. This parameter is essential for calculating the cetane number.

The cetane number of the palm oil biodiesel ranged from 48 to 62, which is similar to the findings of Molla and Nigus (2014) but higher than those reported by Kalam and Masjuki (2002). It complies with ASTM D6751 international standards, as it exceeds the minimum requirement of 47.

The pour point is the lowest temperature at which a liquid still maintains its ability to flow. In this study, the pour point ranged from 1.3°C to 11.7°C, as shown in Table 2, which falls within the acceptable range.

The aniline point, which reflects the aromatic content and potential toxicity of a hydrocarbon mixture, ranged from 17.1°C to 21.7°C for the palm oil biodiesel. This is lower than the results obtained by Alang *et al.* (2018) and Danlami *et al.* (2022).

GC-MS Analysis Of The Biodiesel Synthesized From Palm Oil.

The biodiesel were analyzed using GCMS-QP2010 PLUS SHIMADZU JAPAN to recognize chemical composition of methyl esters in the synthesized samples.

The biodiesel characterized by GC-MS identified the methyl esters in the palm biodiesel as shown in figures 1-5. The methyl esters confirmed in P1 biodiesel (fig.1) are methyl 14-pentadecanoate, methyl 15-methylhexadecanoate, methyl decanoate and methyl hexadecanoate methyl esters. Methyl esters discovered in P2 biodiesel depicted in fig. 2 are methyl 14- pentadecanoate, methyl 15-methylhexadecanoate, methyl decanoate, methyl tridecanoate and methyl hexadecanoate. In P3 biodiesel as shown in fig. 3 the methyl esters recognized are methyl 14- pentadecanoate, methyl decanoate, methyl 15-methylhexadecanoate, methyl tridecanoate and methyl hexadecanoate. Similarly for P4 biodiesel the methyl esters discovered are methyl-11-ocyldecanoate, methyl 5-(2-undecyclopropyl) pentanoate, methyl 15-tetracyosenoate, methyl-13,16-octadecanoate and methyl-15-methylhexadecanoate as repreented in fig. 4 and the methyl esters detected in P5 biodiesel as shown in fig. 5 are methyl-11-octadecanoate, methyl 5-(2-undecyclopropyl) pentanoate, methyl-12-(2-octylcyclopripyl) dodecanoate and methyl-13,16-octadecanoate. These results are similar to the work of Alang et al 2018 and Courtney 2020 which confirmed the stoichiometry of biodiesel synthesis.

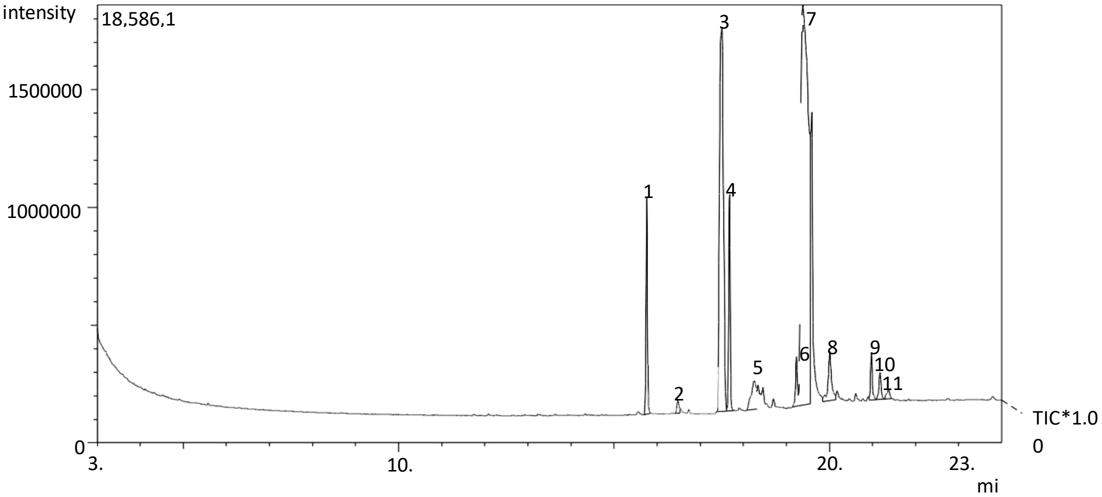


Fig. 1 GC-MS spectrum of P1 biodiesel

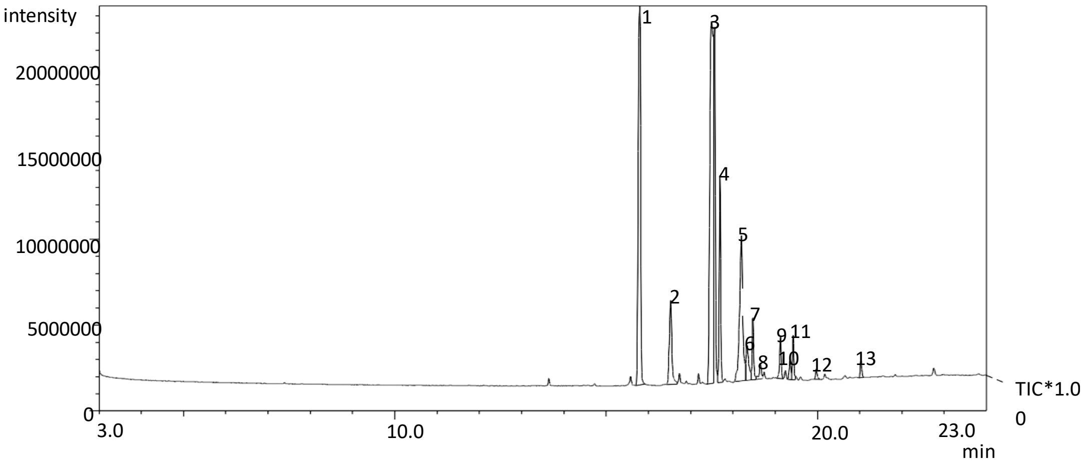


Fig. 2 GC-MS spectrum of P2 biodiesel

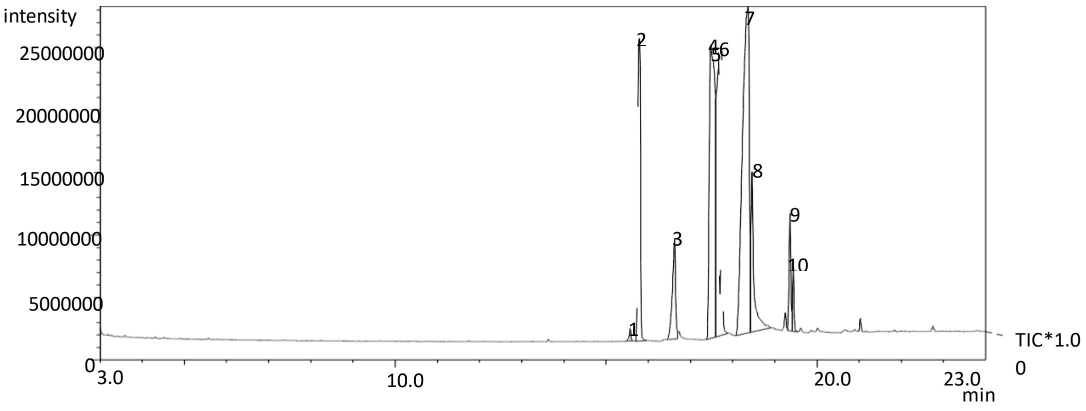


Fig. 3 GC-MS spectrum of P3 biodiesel

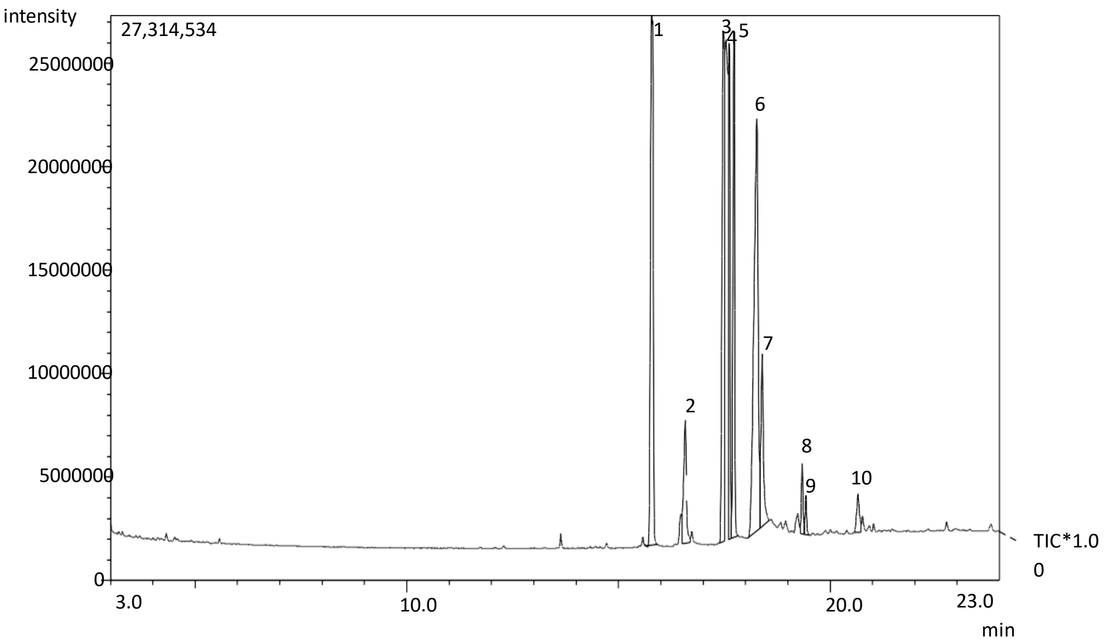


Fig. 4 GC-MS spectrum of P4 biodiesel

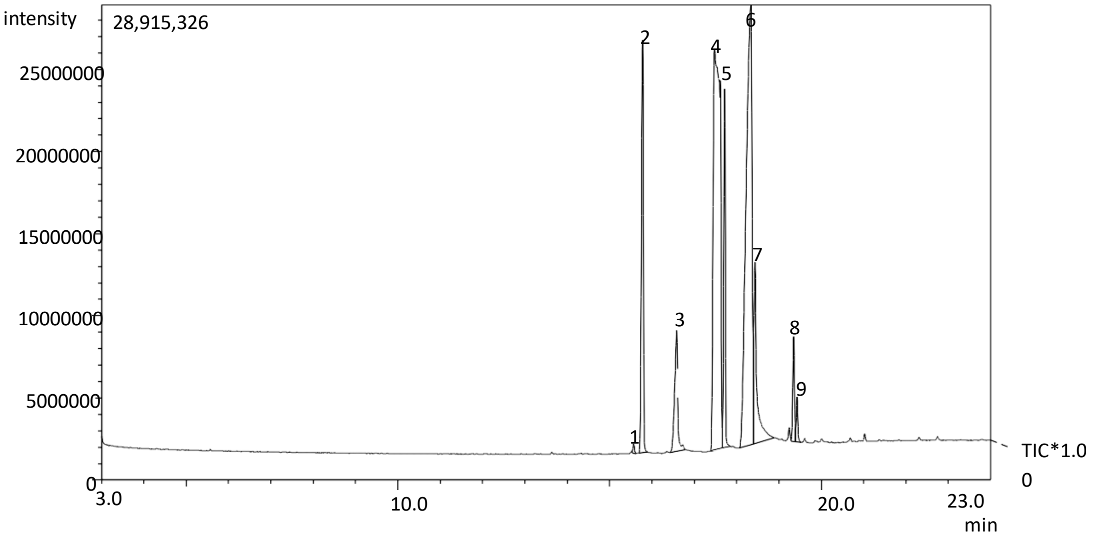


Fig. 5 GC-MS spectrum of P5 biodiesel

CONCLUSION

Palm oil biodiesel was successfully produced through trans-esterification process using KOH as catalyst and methanol at 60°C and also determining its characteristics. The biodiesel yield from the five samples ranges from 69.6 – 77.1% which are higher than the ASTM standard of yielding of more than 30%. Sample P3 gives the highest yield of biodiesel and sample P1 gives the lowest yield of biodiesel. The Synthesized Palm oil biodiesel which was characterized by their Physico-Chemical Properties and the values obtained where within the standard limited of biodiesel except the flash points which are below the standard value for biodiesel. The GC-MS identify four components of methyl esters in two of the palm oil biodiesel and five components of methyl esters in the three of the palm oil biodiesel. This study has shown that most of the properties evaluated from the palm oil biodiesel produced conform to the ASTM D6751 and EN 1421 standard values. Conclusion can be drawn from this study that the biodiesel produced from palm oil can be used as an alternative to diesel.

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