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Investigation of the fuel properties and fatty acid methyl ester composition of biodiesel produced from sand box tree oil using ZnO-NPs catalyst

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This research focuses on characterization of biodiesel produced from sand box tree (*Hura crepitans*) oil by transesterification method using zinc oxide nanoparticles (ZnO-NPs) as catalyst. The ZnO-NPs catalyst was synthesized by simple solution-based approach using zinc nitrate [$Zn(NO_3)_2.6H_2O$] and potassium hydroxide (KOH) as precursors. The biodiesel produced was characterized using ASTM standard fuel tests methods. Fuel properties tested included specific gravity, API gravity, diesel index, cetane number, kinematic viscosity, flash point, cloud point, pour point, acid value, bottom water and sediments and sulphur content. The fatty acid methyl ester (FAME) profile of the biodiesel was determined using Gas-Chromatography coupled with Mass-Spectrometry (GC-MS) method. The transesterification process yielded 91.57±3.6 % sand box tree oil biodiesel with iodine value of 57.31±0.7 gI_2/100g and higher heating value of 47.74±3.1 MJ/kg. The result indicates the presence of Hexadecanoic acid methyl ester (16.15 %), 9,12-Octadecadienoic acid methyl ester (27.04 %), Tetradecanoic acid methyl ester (24.30 %) and 11-Octadecenoic acid methyl ester (8.04 %) as the dominant FAME. All the results obtained were found to be within limits set by ASTM standard for biodiesel

Keywords: Biodiesel, nanoparticles, zinc oxide, sand box tree.

1. Introduction

In recent decades, economic growth coupled with industrial development has led to the rapid increase in energy demand.[1,2] The major sources of energy are the non-renewable fossil fuels which include crude oil, coal, natural gas etc.[3] However, the lack sustainability of fossil fuels reserve and their environmental impact have led to the search for alternative, sustainable and renewable sources of energy. The burning of large amount of fossil fuels has increased the carbon dioxide (CO_2) level in the atmosphere causing global warming.[4,5] In recent years, researchers all over the world have been trying to find new alternative fuels that are affordable, sustainable, renewable and environmentally friendly.[6]

In recent years, bio-fuels have become more attractive because of their environmental benefits and the fact that they are made from renewable resources.[7] Bio-fuels refer to liquids or gaseous fuels produced from biomass and has a great tendency to minimize greenhouse gas emission as well as complimenting the fossil fuels.[3,8] Biodiesel is a liquid biofuel that is produced from fresh or used vegetable oil, or used animal fats. It can be produced via transesterification from both edible and non-edible oil crops such as soy bean oil, rapeseed oil, cotton seed oil, palm oil, sunflower oil, linseed oil, peanuts oil, *Jatropha curcas* seed oil etc.[9,10] Chemically, biodiesel is produced through transesterification by reacting vegetable oil or animal fat with a low molecular weight alcohol in the presence of suitable catalyst. The product of the reaction is a mixture of alkyl ester (biodiesel) and glycerol.[2,11]

Transesterification of vegetable oils or animal fats is traditionally carried with homogenous catalysts such as strong acids (e.g. H_2SO_4, HCl, H_3PO_4 etc.) and bases (e.g. NaOH, KOH, $NaOCH_3$ $KOCH_3$, $NaOCH_2CH_3$ etc.).[12-14] Homogeneous alkali catalysts can convert triglycerides to their corresponding fatty acid alkyl esters with high yield, less time and low cost. However, separating the catalyst from the product mixture for recycling is technically difficult.[15,16] Separation problem coupled with corrosive nature of homogenous catalysts have motivated intense research on heterogeneous catalyzed transesterification.[17] Among the

heterogeneous catalysts that are being used in transesterification are calcium oxide, magnesium oxide, barium oxide, zinc oxide, etc. These catalysts have a promising place and many reports have been published about them.[18-20] However, some these catalysts have lower conversion efficiency and take longer reaction time. The utilization of nanoparticle as heterogeneous catalysts has been of recent interest in the search for a sustainable process. Utilization of these materials will not only reduce the catalyst cost but also promotes environmentally benign process. Therefore, in this study zinc oxide nanoparticles (ZnO-NPs) was prepared and used in biodiesel production using sand box tree oil.

2. Materials and Methods

2.1 Sample Collection and Treatment

The seeds of sand box tree were collected at various location within Sokoto state, Nigeria. The seed kernels were sun-dried and grounded into powder using mortar and pestle. The oil was extracted from the powdered sample using soxhlet extractor with n-hexane as a solvent.[21,22]

2.2 Catalyst Preparation

The ZnO-NPs was synthesized by direct precipitation method using zinc nitrate [$Zn(NO_3)_2.6H_2O$] and sodium hydroxide (NaOH) as precursor. In this method, 0.4 M aqueous solution of sodium hydroxide was slowly added to 0.2 M of aqueous solution of zinc nitrate at room temperature under vigorous stirring until white suspended precipitate is formed. The white product obtained was centrifuged at 5000 rpm for 20 min. It was then washed three times with distilled water and lastly absolute alcohol. The product was calcined at 500 °C for 3 hrs.[23]

2.3 Transesterification

Prepared solution of 50 cm^3 of methanol and 1 g (wt/v) of the catalyst was added to 250 cm^3 of the warmed oil on water bath. The heating continued at 60 °C with constant stirring (at 500 rpm) for 3 hrs until the reaction was completed.[24] The content was transferred into a separating funnel and was allowed to settle for 24 hrs. This permits the glycerol and catalyst mixture to settle down since it is denser than biodiesel. The glycerol was finally separated from the biodiesel using the separating funnel. The percentage biodiesel yield was calculated using equation below.[24,25]

$$\%\text{Biodiesel Yield} = \frac{\text{Weight of Biodiesel}}{\text{Weight of Oil}} \times 100\%$$

2.4 Purification of the Biodiesel

The produced biodiesel was washed three times with warm distilled water (50 °C) to remove any residual glycerol, methanol or catalyst. It was then dried with anhydrous sodium sulphate and filtered before characterization.[26,27]

2.5 Determination Fuel Properties of the Biodiesel

The fuel properties of the biodiesel were determined according to ASTM D6751 standard methods for biodiesel. The fuel properties tested include specific gravity, cetane number, higher heating value, kinematic viscosity, flash point, cloud point, pour point, acid value, water & sediment, sulphur content and iodine value.

2.6 Fatty Acid Methyl Ester Analysis

The Fatty Acid Methyl Ester analysis was performed by using GC-MS-Q2010 PLUS. A volume (2 µL) of biodiesel sample was injected into the Elite column-5MS. The injection was performed in split ratio (10:1). The oven temperature was initially held at 140 ºC for 5 min, later increased to 240 °C at 4 °C/min, and held for 5min. The injector, transfer and source temperatures were set at 250 °C, 200 °C and 150 ºC, respectively. The carrier gas was helium and the total scan time was 35 min.[28]

3. Results and Discussion

3.1 Results

The percentage yield and fuel properties of the biodiesel are shown in table 1.

Table 1. Fuel Properties of the Biodiesel Produced

Properties	Unit	Biodiesel ASTM D6751	Sand Box Tree Oil Biodiesel
% Yield	%	-	91.57±3.6
Specific Gravity	-	0.86 - 0.90	0.868±0.001
Cetane Number	-	47 min	34.53±0.15
Higher Heating Value	MJ/kg	-	47.74±3.1
Kinematic Viscosity	mm^2/sec	1.9 - 6.0	2.50±0.01
Flash Point	°C	93 min	162±4
Cloud Point	°C	-3 – 12	6.80±0.22
Pour Point	°C	-15 to 16	-5.70±0.1
Acid Value	mgKOH/g	0.05 max	0.33±0.03
Water and Sediments	%	0.05 max	0.04±0.002
Sulphur Content	%	0.05 max	0.01±0.001
Iodine Value	$gI_2/100g$	-	57.31±0.7

The fatty acid methyl ester profile of the biodiesel produced is shown in table 2.

Table 2. Fatty acid methyl ester composition of the Biodiesel Produced

Name of compound	Molecular Formula	% Area
7,9-octadecadiynoate acid methyl ester	$C_{19}H_{30}O_2$	0.43
2,5-Octadecadiynoic acid methyl ester	$C_{19}H_{30}O_2$	0.29
9-Hexadecenoic acid methyl ester	$C_{17}H_{32}O_2$	0.35
Hexadecanoic acid methyl ester	$C_{17}H_{34}O_2$	16.15
9,12-Octadecadienoic acid methyl ester	$C_{19}H_{34}O_2$	27.04
Tetradecanoic acid methyl ester	$C_{15}H_{30}O_2$	24.30
11-Octadecenoic acid methyl ester	$C_{19}H_{36}O_2$	8.04
Octadecanoic acid methyl ester	$C_{19}H_{38}O_2$	4.49
Other Non-Methyl ester compounds	-	18.91

3.2 Discussion

3.2.1 Percentage Biodiesel Yield

The result of biodiesel yield (91.57±3.6) obtained is presented in Table 1. The results showed that the yield obtained within the condition investigated was appreciable. The biodiesel yield obtained using ZnO-NPs in this research is higher than those obtained by many other researchers that used other forms of zinc oxide. Hatefi *et al*.,[29] conducted an experiment on ZnO for transesterification of corn oil with methanol, the yield obtained was 51.3 % which is lower compared to the yield obtained using ZnO-NPs. Istadi *et al.,*[30] experimented on active acid sulphated ZnO for transesterification of Soya bean oil with methanol. Though the catalyst exhibits promising biodiesel yield of 80.19 %, the yield was however lower compared to 91.57 % obtained using ZnO-NPs. Arena *et al.,*[31] worked on one step synthesis of CaO – ZnO catalyst for biodiesel production. The catalyst showed high yield of 73 % in the first cycle and 64 % in the second cycle. Though the catalyst showed a huge potential for biodiesel production, the yield is lower compared to those obtained using ZnO-NPs.

3.2.2 Fuel Properties of the Biodiesel Produced

The specific gravity of the biodiesel was found to be 0.868±0.001 which is within the ASTM D6751 test method. The result is comparable to 0.875 for sorghum oil biodiesel reported by Ved and Padam.[32] However, the result is higher than 0.830 for waste cooking oil biodiesel reported by Adepoju and Olawale[37] but lower than 0.952, 0.942 and 0.924 for castor oil biodiesel reported by Al-Harbawy and Al-Mallah.[33]

The result obtained for cetane number (34.53±0.15) is lower than the minimum cetane number prescribed in ASTM D6751 indicating that the biodiesel produced has shorter ignition delay. The result is lower than 44.79, 53.50 and 50.98 for *Michelia champaca* oil biodiesel reported by Hotti and Hebbal,[34] but higher than 27 for *Langenaria Siceraria* oil biodiesel reported by Bello[35]. However, the result is comparable to 31.05 for *Cladophora Vagabunda* oil biodiesel reported by Sharmila and Jeyanthi.[22]

The High heating value was found to be 47.74±3.1 MJ/kg which indicates the biodiesel produced contained appreciably high amount of energy. This is comparable to 45.62 MJ/kg for neem seeds oil biodiesel reported by Adewuyi *et al*.[36]

The result obtained for viscosity (2.50±0.01 mm^2/sec) is within the ASTM D6751 limits (1.9 – 6.0 mm^2/sec @ 40 °C). The kinematic viscosity obtained is lower than 5.96 mm^2/sec for kapok oil biodiesel reported by Asokan and Vijiyan.[1] However, the kinematic viscosity is comparable to 2.56 mm^2/sec for waste cooking oil biodiesel reported by Adepoju and Olawale[37] but higher than 2 mm^2/sec for petroleum diesel reported by Purohit *et al.*[38]

The result obtained for the flash point (162±4 °C) is lower than 232 °C for *Michelia champaca* oil biodiesel reported by Hotti and Hebbal[34] and higher than 116 °C for waste cooking oil biodiesel reported by Adepoju and Olawale.[37] However, the result is comparable to 160 °C for castor oil biodiesel reported by Okullo *et al.*[26] and 168 °C for Jatropha oil methyl ester reported by Rashid *et al.*[39]

The result of the cloud point obtained (6.80±0.22) is in accordance with ASTM D6751 (-3 °C to 12 °C) indicating that the fuel can be used under harsh temperature conditions without the risk of causing blockage in fuel injection system. The result is comparable with 6 °C for sorghum oil biodiesel reported by Ved and Padam.[32] However, the result is higher than -13 °C for castor oil biodiesel reported by Okullo *et al.*[26] but lower than 9 °C for waste cooking oil biodiesel reported by Adepoju and Olawale.[37]

The result obtained (-5.70±0.1 °C) is in accordance with ASTM D6751 (-15 °C to 16 °C). This indicate that the fuel can flow freely through pipes even at very low temperatures. The result is comparable to -5 °C for *Ceiba pentandra* oil biodiesel reported by Asokan and Vijayan.[1] However, the result is lower than -2 °C for Jatropha oil biodiesel reported by Gandure *et al.*[43] but higher than -13 °C for waste cooking oil biodiesel reported by Adepoju and Olawale.[37]

The result obtained for acid value (0.33±0.03) is in accordance with ASTM D6751 maximum acceptable acid value (0.50 mgKOH/g) for a biodiesel. The result is lower than 7.04 mgKOH/g and 1.86 mgKOH/g for Castor oil biodiesel reported by Asmare and Gabbiye.[40] However, the result is comparable with 0.31 mgKOH/g for sorghum oil biodiesel reported by Ved and Padam.[32]

According to United States biodiesel standard (ASTM D6751), the content of water and sediment in biodiesel must be less than 0.05% volume by volume. From the results obtained (0.04±0.002 %), it is clear that the content of water and sediment are in accordance with ASTM D6751 standard. The results obtained are comparable to <0.05% for castor oil biodiesel reported by Al-Harbawy and Al-Mallah.[33]

The ASTM D5453 recommended sulphur limits is at 0.05wt. %, maximum.[41] The total sulphur content was 0.01±0.001 % and this was within the recommended ASTM sulphur limits (00.5 %). The result obtained is higher compared to the 0.004 obtained by Karunanithi and Maria[42] for neem oil seed biodiesel.

The result obtained (57.31±0.7) is lower than 122.71 gI_2/100g for Champaca oil biodiesel reported by Hotti and Hebbal[34] and 116 gI_2/100g for waste cooking oil biodiesel reported by Adepoju and Olawale.[37] However, the results are comparable with 58.34 gI_2/100g for castor oil biodiesel reported by Al-Harbawy and Al-Mallah.[33]

3.3 Fatty Acid Methyl Ester Profile of the Biodiesel Produced

The proximate percentage composition of fatty acids methyl esters (FAME) of the biodiesel produced from sand box tree oil is shown in table 2. The composition of the biodiesel indicates the presence of Hexadecanoic acid methyl ester ($C_{17}H_{34}O_2$), 9,12-Octadecadienoic acid ($C_{19}H_{34}O_2$) methyl ester and Tetradecanoic acid methyl ester ($C_{15}H_{30}O_2$) having a percentage of 16.15 %, 27.04 % and 24.30 % respectively, as the major ester compounds. Also, the result showed the presence of 11-Octadecenoic acid methyl ester ($C_{19}H_{36}O_2$) and Octadecanoic acid methyl ester ($C_{19}H_{38}O_2$) having a percentage of 8.04 % and 4.49 % respectively. Other FAME's include 7,9-octadecadiynoate acid methyl ester ($C_{19}H_{30}O_2$), 2,5-Octadecadiynoic acid, methyl ester ($C_{19}H_{30}O_2$), 9-Hexadecenoic acid methyl ester ($C_{17}H_{32}O_2$) having a percentage of 0.43 %, 0.29 % and 0.35 % respectively. The remaining 18.91% constitute of non-ester compounds.

4. Conclusion

The results from this study revealed that transesterification of sand box tree seed oil to biodiesel was successful via the use of ZnO-NPs heterogeneous catalyst. The results of fuel properties of the biodiesel produced compared favorably with those of the ASTM D6751 biodiesel standard. The FAME analysis indicate that the majority of the compound present are ester compounds. The result also reveals that biodiesel from sand box tree seed oil is quite suitable as an alternative to petroleum-based diesel. Therefore, more research is recommended on the utilization of commercially underutilized seeds to help increase energy security in the light of depleting fossil fuel reserve.

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Conflict of interest

The authors declare no conflict of interest.

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