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Research Article

Polyoxovanadate Catalyzed Synthesis of Biodiesel (Methyl Esters) from Waste Cooking Oil

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Abstract

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Biodiesel, Waste cooking Oil (WCO), Polyoxometalates, Polyoxovanadate, Transesterification spurred research interest in alternative and renewable energy sources. This study aimed to investigate the transesterification of waste cooking oil using Polyoxovanadate (POV) as a catalyst. The waste cooking oil was first acquired and subjected to physicochemical analysis for characterization purposes. Subsequently, transesterification of the oil with methanol was performed using Polyoxovanadate (POV) synthesized from Ammonium metavanadate (NH_4VO_3) solution at pH. The synthesized POV underwent characterization using techniques including UV-visible spectrometry, Fourier transform infrared (FTIR) spectrophotometry, X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). The resulting biodiesel was also characterized using FTIR, Gas Chromatography-Mass Spectrometry (GC-MS), and its fuel properties were determined using ASTM Methods. The process proved successful, resulting in a biodiesel yield of 76.44 % when employing a catalyst loading of 0.8 g, a 1:9 oil-to-methanol ratio, and conducting the reaction at 60 °C for 90 minutes. The biodiesel obtained predominantly consisted of saturated fatty acid methyl esters. Furthermore, biodiesel exhibited favorable fuel properties that met the standards established by ASTM for diesel engines. Notably. Its low sulfur content highlighted its environmentally friendly nature in comparison to petroleum diesel.

Depleting fossil fuel resources and the steady increase in energy consumption have

Nomenclature and units

1.0 Introduction

The primary sources of energy consumed globally are nonrenewable fossil fuels, including coal, petroleum, and natural gas. These sources account for 86 % of the world's energy use, and the transportation industry relies entirely on them (Boakye & Boakye, 2013). Despite their widespread use, fossil fuels are not sustainable or renewable energy sources and their combustion releases harmful pollutants such as Sulfur oxides, carbon oxides, particulate matter, and other gases that contribute to air pollution (Patil et al., 2012). The danger of this pollution cannot be discussed exhaustively as it affects the environment, human health, and the survival of other living organisms. The depletion of the ozone layers which result from the emission of greenhouse gases from fossil fuel is now a major global challenge as it affects the climate. The rise in the ocean water level, low agricultural vield, and also extinction and endangering of some plants and animal species. However, world petroleum reserves depletion and increased environmental concerns have recently stimulated interest in alternative sources for petroleum-based fuels (Meng et al, 2008). It is crucial to explore alternative sources of energy that are renewable and sustainable, such as hydro, biomass, geothermal, hydrogen, nuclear, wind, and solar power, to reduce reliance on exhaustible energy sources (Boakye & Boakye, 2013). Fuels derived from biological feedstock (biomass) are generally called biofuel and they include; biogas, biokerosene, biodiesels, etc. Biofuel in recent years has attracted the attention of researchers and has given rise to numerous renewable energy technologies.

The production of biodiesel is typically costlier than that of conventional fossil fuels because the expense of raw materials accounts for over 80 % of the overall production cost (Haas et al, 2006). The use of expensive edible plant and vegetable oils as raw materials for biodiesel production is a significant obstacle that hinders its widespread use.

The cost of using edible plant and vegetable oils as feedstock for biodiesel production is prohibitively high, accounting for 60 - 80 % of the total cost (Dahlquist & Gardy, 2010), making it economically uncompetitive. One cost-saving strategy is to use low-qualify feedstocks, such as waste or used vegetable oils, non-edible oils, and soap stock (a by-product of vegetable oil refinery), which are abundantly available and make for attractive feedstock options for biodiesel production Canakci, M. & Sanli, H. (2008).

According to Energy Information Administration (EIA), the United States produces roughly 100 million gallons of waste cooking oil daily, with each person generating about 9 pounds of waste cooking oil per year (Patil et al. 2012). The management of waste cooking oils and fats is a significant challenge due to the problems of disposal and potential contamination of water and land resources ((Boakye & Boakye, 2013). Although some of the KJSET | 158

water oil is repurposed for soap production, the majority is discharged into the environment, contributing to environmental pollution. The conversion of waste cooking oil (WCO) into biodiesel is helpful for the reduction of pollution and will be a valuable addition of energy to the existing energy grid (Sadaf et al., 2018).

The role of catalysts in the production of biodiesel is of great importance as they are given lots of concentration due to their ability to increase yield and low cost and time of production. Biodiesel production can be facilitated by both homogeneous acid and base catalysts (Dahlquist & Gardy, 2010). Homogeneous base catalysts, including sodium, potassium hydroxides, and alkoxides, are preferred to homogeneous acid catalysts due to their high catalytic efficiency and low corrosive properties (Kawashima et al., 2009 in Dahlquist & Gardy, 2010). However, the use of homogeneous base catalysts can lead to an unwanted reaction between the catalyst and free fatty acids in the waste and frying oils, resulting in soap formation and reduced biodiesel conversion (Dahlquist & Gardy, 2010).

Polyoxometalates (POMs) are a remarkable category of nanoscale metal-oxo clusters known for their diverse structures and oxygenenriched surfaces (Li et al., 2014). Polyoxovanadates (POVs) have been receiving growing attention not only for their significance in bioinorganic and magnetochemistry but also for their potential applications in medicine, as well as homogeneous catalysis (Wang, 2015). With the availability of polyoxovanadate salts and the large surface area it offers, it can be utilized in the transesterification process for biodiesel production. In a recent study by Xie et al (2020), a vanadium-substituted polyoxometalate (POM) acid catalyst was utilized for transesterification of low-quality oil. The researchers achieved a conversion rate of 92.2 % at 140 °C, using an oil–methanol ratio of 1:30 for 8 hours. The catalyst exhibited exceptional stability and reusability properties.

Although there is available literature on the use of Polyoxometalate as a catalyst in the production of biodiesel from waste oil, there is a lack of evidence regarding the application of Polyoxovanadate in the transesterification of waste oils.

2.0 Materials and Method

2.1. Materials

The waste cooking oil was collected from Houses in the Badariya area beside ABI Private School Badariya, Birnin Kebbi, Kebbi State Nigeria. All required glassware was soaked in dilute nitric acid for 24 h and then rinsed in distilled water before experiments. The chemicals, reagents, and solvents used in this research work were analytical and general purposed grade. except for Ammonium meta vanadate (NH_4VO_3) purchased from Sigma Aldrich.

2.2 Sample Pre-treatment

The waste cooking oil was heated at 90 °C for an hour and was allowed to cool before filtering using filter paper to remove suspended impurities and moisture.

2.3 Determination of physicochemical properties of Waste Cooking Oil (WCO)

The following physicochemical properties of the waste cooking oil (WCO) were determined: Specific gravity was determined using ASTM D1298 (ASTM, 2002), while the Free Fatty Acid value, Acid value, Saponification value, and Iodine value, and Iodine value were determined using AOCS methods (AOCS, 1980; 2003).

2.4 Catalyst Preparation and Characterization

The Catalyst was prepared by dissolving a specific amount of ammonium metavanadate (0.8 g) in a few drops of distilled water, then mixing it with 45 ml of methanol. The mixture was stirred until it dissolves. The solution was then adjusted to pH 2 using a few drops of dilute HNO₃ and NH₄OH solutions. The POV solution was characterized using a **UV-Visible** spectrophotometer. In addition, the POV solution was dried at 100 °C and heated at 500 °C in an air oven before characterization. The Fourier transform infrared (FTIR) spectra were acquired on a KBr disc using an Agilent spectrophotometer from 500 - 4000 cm⁻¹. The crystal phase of the catalysts was identified using X-ray diffraction (XRD) measurement with Cu K α (0.154) radiation at 40 kV and 15 mA. The scanning range was between $3 - 90^{\circ}$ at a scan rate of 4°/min. The Morphology of the catalysts was obtained using scanning electron microscopy (SEM).

2.5 Transesterification Process

Exactly 5 ml of the waste cooking oil was poured into a twonecked round bottom flask fitted with a reflux condenser and heated to a temperature of 60 °C under stirring. A solution of polyoxovanadate catalyst (0.8 g) in 45 ml of methanol acidified with HNO₃ at pH 2 was added to the heated oil and stirred for 90 minutes using a magnetic stirrer as shown in Fig. 1. After the reaction time, the mixture was allowed to stand overnight in a separating funnel. The produced biodiesel at the top of the separating funnel in excess methanol was separated from the other two layers of glycerol and catalyst at the bottom. The excess methanol was removed from the biodiesel (methyl esters) via refluxing. In addition, water a byproduct of the reaction is removed from the biodiesel via drying using silica gel and filtered on a filter paper containing Sodium sulfate. The biodiesel was kept in an airtight bottle for further characterization.

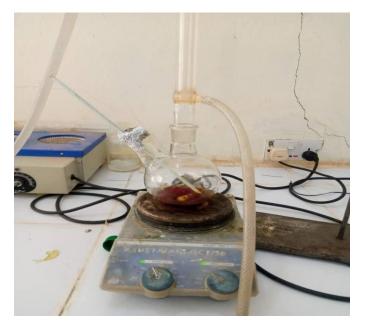


Figure 1 Transesterification experimental setup

The best parameter for optimum yield was used to produce a large quantity of biodiesel.

The yield was calculated using the equation below;

Biodiesel yield (%) =
$$\frac{weight of Biodiesel}{weight of WCO} x 100$$

Where the weight of biodiesel = weight of oil - the weight of glycerol.

2.6 Biodiesel Characterization

The produced biodiesel was characterized using Shimadzu 2010. Gas chromatography-mass spectroscopy (GC-MS) MS- QP 2010 SE and Fourier-transform infrared (FTIR) on the fuel.

2.7 Fuel Test

The following fuel test will be carried out on the produced biodiesel using ASTM methods:

- 1. Flash point
- 2. Viscosity
- 3. Specific gravity
- 4. Cloud point
- 5. Pour point
- 6. Sulphur content

3.0 Results and Discussion

3.1 Result and Discussion of the physicochemical properties of waste cooking Oil Feedstock

Table 1 below depicts the physicochemical properties of the waste cooking oil (WCO). The WCO is brownish in color and liquid at room temperature. It reveals a Specific Gravity of 0.925 g/cm³, with Acid, Free Fatty Acid, Saponification, and iodine values of 0.69 mg KOH/g, 0.34 %, 181.9 \pm 0.25 mg KOH/g, and 105.48 \pm 1.0 mg iodine/g respectively.

Table 1. Results of physicochemical properties of the waste cooking oil.

Parameters	Value
Color	brownish
State at room temperature	liquid
Specific Gravity (g/cm ³)	0.925
Acid value (mg KOH/g)	0.67
Free fatty acid (%)	0.34
Saponification value (mg KOH/g)	181.9 ± 0.25
Iodine value (mg iodine/g)	105.45 ± 1.0

The results are mean of triplicate determination

Specific gravity

The Specific gravity of the Waste cooking oil 0.925 g/cm^3 falls within the range of most oils. This value affects the use of waste oil directly as fuel in diesel engines.

Acid Value

The waste cooking oil was found to have acid and Free fatty acid values of 0.67 mg KOH/g and 0.34 % respectively as shown in Table 1. According to Leung and Guo (2006), oils with elevated acid values tend to form soap with acid catalysts which deactivate the catalysts causing a low yield of biodiesel. The low acid and Free fatty acid of the waste cooking oil indicate that the Polyoxovanadate (POV) catalyst is suitable for the transesterification reaction of WCO.

Iodine Value

The Iodine value in Table 1 indicates the waste cooking oil has 105.48 mg/g which is within the AOCS acceptable limit, this value reveals the unsaturation level of the oil and oxidative stability of the oil. However, the iodine value is lower than 115g/I2 100 g set for dry oils (Abdulkareem et al, 2011).

Saponification Value

The waste cooking oil had a saponification value of 181.9 mg KOH/ g, which fall within the specified limit set by AOCS. The saponification value serves as an indicator of the oil's propensity

to produce soap during the transesterification reaction, which could reduce biodiesel yield.

3.2 Catalysts Characterization

UV-Visible determination of polyoxovanadate species

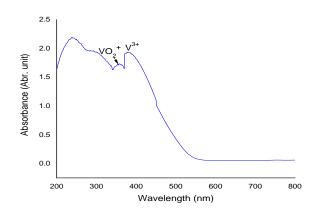


Figure 2 UV-Visible spectra of Vanadate species in Methanol at pH2.

The UV-visible spectrum of the acidified POV at pH 2 reveals a peak at 382.3 nm and a shoulder peak at 355.5 nm. These peaks may be ascribed to a combination of Vanadium species V^{3+} and VO_2^+ , representing polymeric (dimer) and monomeric Vanadium oxide species, respectively. These observations align with the various Vanadium species documented by Brooker et al. (2015). However, Gumerova and Rompel's (2020) findings indicate that solely VO_2^+ is present at a pH below 2.

FTIR Spectra of POVs Catalysts

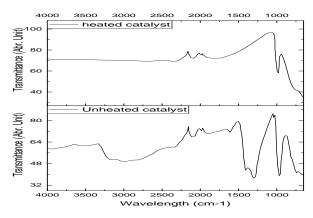


Figure 3 FTIR Spectra of Unheated and Heated POVs Catalysts.

Figure 3 shows the FT-IR spectra of the Polyoxovanadate catalyst at a pH of 2. The unheated catalyst reveals fingerprints at 965, 1300 - 1400, 1615, 1881, and 2094 cm⁻¹, which could be attributed to Vanadyl oxygen stretching vibration "v(V=O), Vanadyl oxygen bending vibration " $\delta(V=O)$, Vanadyl –carbonoxygen stretching in a vanadyl complex "v (V-C-O), Carbon – oxygen stretching in vanadyl complex, and vanadyl in-plane bending, respectively.

These vibrational peaks are completely absent in the heated catalyst at 450 C, except for peaks at 975, 1793, and 2105 which could be assigned to Vanadyl oxygen stretching vibration "v(V=O), Vanadyl oxygen bending vibration " $\delta(V=O)$, and Vanadyl out-of-plane bending vibration. These findings are in agreement with those reported by Barbosa (2005). The presence of the V=O center in both catalysts is accepted as the active site for catalyzing the transesterification reaction.

XRD analysis of POVs catalysts

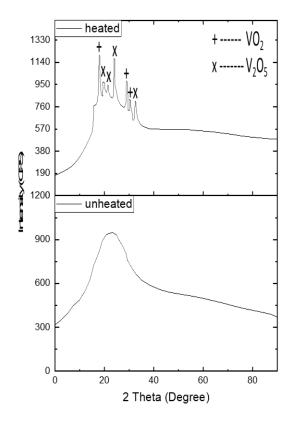


Figure 4: XRD Diffractogram of POVs catalysts.

Figure 4 depicts a typical XRD diffractogram of both heated and unheated POV catalysts. The broad peak of the unheated catalyst spectra centered at 22.63°. This indicates an amorphous nature of the unheated catalyst with no defined crystal structure. The heated catalyst at 450 °C reveals peaks at diffraction angles of 17.5°, 28.9°, and 30.53° assigned to the VO₂ crystal phase, while the peaks at 19.14°, 21.39°, 23.85°, and 32.58° are attributed to V₂O₅. This finding conforms to works reported by Zhang (2016), Zhao et al (2014), and Batista et al (2008). In addition, the XRD diffractogram of the Polyoxovanadate Phase corroborates the UVvisible spectra in Figure 2 above.

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Scanning Electron Microscopy (SEM)

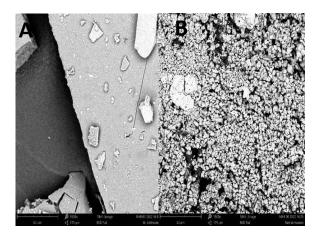
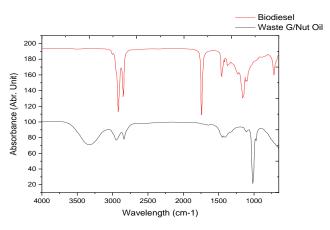


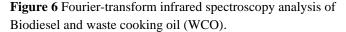
Figure 5 SEM Image of POV (A) Before heating, and (B) after calcination at 450 °C.

The SEM images in Figure 5 reveal the unheated catalyst A indicating a composite of amorphous material, while the calcined catalyst at 450 °C B reveals a crystalline grain of POV with sizes between 200 - 500 nm. A similar observation was reported for nanocrystalline vanadium oxides by Batista (2008). This finding supports the XRD data of the POV catalyst in Figure 4.

3.3 Characterization of Biodiesel

FTIR Characterization of Biodiesel





Biodiesel (black) and Waste G/Nut Oil (Red)

FT-IR spectra of the waste cooking oil and the biodiesel produced from the oil are illustrated in Fig. 6 Revealing peaks at 2924 cm⁻¹ and 2853 cm⁻¹ represent the antisymmetric and symmetric stretching vibrations of C-H in CH₂ and CH₃ groups respectively. The strong peak present at 1743 cm⁻¹ is attributed to the presence of C-O stretching vibration of carbonyl groups that is present in the triglycerides of the waste cooking oil. The peaks at 1400- 1200 cm⁻¹ region confirmed the bending vibrations of CH₂ and CH₃ aliphatic groups for both the waste cooking oil and the biodiesel. Similarly bending of HCH takes place at 1373 cm⁻ ¹ and CH₂ and scissoring at 1466 cm⁻¹ respectively. The peaks present around the region of 1120-1090 cm⁻¹ presented the stretching vibration of C-O ester which is pronounced on the biodiesel spectra. Moreover, the peak at 1742 cm⁻¹ confirmed the stretching vibration of C=O present in the esters and peaks present in the range of 1300-1000 cm⁻¹ conforming to that of the C-O stretching vibrations. The FT-IR spectra of the waste cooking oil and biodiesel produced are comparable to each other because of the presence of triglycerides and esters. However, very small differences were observed where the peaks that appeared at 1743 - 876 cm⁻¹ in the waste cooking oil were shifted to 1742 - 884 cm⁻¹ in the biodiesel respectively. So, the disappearance of the peaks from the spectrum of the waste cooking oil at about 1543 and 1198 cm⁻¹ and the formation of new peaks at 1430 cm1 and 1024 cm⁻¹ in the produced biodiesel confirm the conversion of waste oil into biodiesel. Though, the presence of a peak in the region of 3100-3500 cm⁻¹ proposes that the biodiesel synthesized from waste cooking still contains some traces of methanol and water which can be removed by refluxing and drying with silica gel.

GC-MS characterization of the biodiesel obtained from waste cooking oil (WCO)

The GC-MS analysis shown in Fig. 7 depicts the profile of fatty acid methyl esters composition of the waste cooking oil (WCO). Table 3.2 reveals that waste cooking oil contains six basic fatty acids, which impact positively the properties and stability of the biodiesel. Nonanoic acid methyl ester, Azeleic acid dimethyl ester, Myristic acid methyl ester, palmitic acid methyl ester, 11octadecenoic acid methyl ester, and methyl stearate. Most of the stated fatty acid methyl esters are saturated except for 11octadecenoic acid methyl ester which is unsaturated according to Table 2. The waste cooking oil consists of more than 75 % saturated fatty acid, with about 6.68 % unsaturated fatty acid. The presence of a higher amount of saturated fatty acid methyl ester makes the biodiesel less susceptible to fuel oxidation and better stability. These findings contradict the report of Helmi et al, (2020) on the transesterification of waste cooking oil using Phosphomolybdic acid/graphene oxide.

3.4 Fuel properties of the biodiesel obtained from waste cooking oil (WCO)

The results of fuel characterization of the biodiesel obtained from waste cooking oil seeds are given in Table 3. The biodiesel produced was characterized using ASTM specification methods. The table contains some of the characterized properties of biodiesel in comparison with different ASTM specifications and other literature.

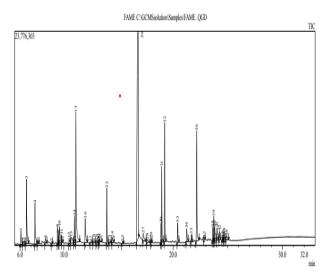


Figure 7 Chromatogram of Fatty acid methyl esters (FAME) of WCO.

Table 2 W	aste cooking	oil methyl	esters of	composition.

Peak	Fatty acid	Retention	Formul	Area
No.	methyl ester	time (min)	а	(%)
3	Nonanoic acid,	6.595	$(C_{10}H_{20}$	3.66
	me		O ₂)	
8	Nonanoic acid,	9.424	$(C_{10}H_{18}$	1.65
	9-oxo-, me		O ₃)	
10	Octanedioic	9.606	$(C_{10}H_{18}$	1.93
	acid, dime		O4)	
12	Octanoic acid,	10.488	$(C_{11}H_{22})$	0.59
	6,6-dimethoxy-,		O4)	
	me			
13	10-Undecenoic	10.681	$(C_{12}H_{22}$	1.31
	acid, me		O ₂)	
14	Dodecanoic	10.977	$(C_{13}H_{26}$	1.35
	acid, me		O ₂)	
15	Nonanedioic	11.108	$(C_{13}H_{20}$	8.95
	acid, dime		O4)	
18	Decanedioic	12.760	$(C_{12}H_{22})$	0.32
	acid, dime		O4)	
19	Octadecanoic	13.026	$(C_{19}H_{38}$	0.21
	acid, 9, 10-		O4)	
	dihydroxy-, me			
20	17-	13.198	$(C_{18}H_{32}$	0.20
	Octadecynoic		O ₂)	
	acid, me			
21	12-Tridecynoic	13.282	$(C_{15}H_{30}$	0.41
	acid, me		O ₂)	
22	Methyl	13.943	$(C_{15}H_{30}$	2.74
	tetradecanoate		O ₂)	
23	Undecanedioic	14.200	$(C_{13}H_{22})$	0.18
	acid, dime		O ₅)	

25	Pentadecenoic,	15.377	$(C_{16}H_{30}$	0.13
	me		O ₂)	
26	Hexadecanoic	16.797	$(C_{17}H_{34}$	35.6
	acid, me		O ₂)	9
29	Heptadecanoic	18.004	$(C_{18}H_{36}$	0.21
	acid, me		O ₂)	
30	13-	18.897	$(C_{19}H_{36}$	0.89
	Octadecenoic		O ₂)	
	acid, me			
31	11-	18.964	$(C_{19}H_{36}$	3.86
	Octadecenoic		O ₂)	
	acid, me			
32	Methyl stearate	19.325	$(C_{19}H_{38}$	5.84
			O ₂)	
34	Octadecanoic	21.245	$(C_{19}H_{34}$	1.15
	acid, 9-oxo-, me		O ₃)	
35	Methyl 18-	21.667	$(C_{17}H_{34}$	0.39
	ethynonadecano		O ₂)	
	ate			
38	2-	23.634	$(C_{19}H_{36}$	0.87
	Furanpentanoic		O ₃)	
	acid,			
	tetrahydro-5-			
	nonyl-, me			
39	2-	23.726	$(C_{19}H_{36}$	1.65
	Furanpentanoic		O ₃)	
	acid,			
	tetrahydro-5-			
	nonyl-, me			
44	2-	24.617	$(C_{19}H_{36}$	0.36
	Furanpentanoic		O ₃)	
	acid,			
	tetrahydro-5-			
	nonyl-, me			
46	2-	24.903	$(C_{19}H_{36}$	0.64
	Furanpentanoic		O ₃)	
	acid,			
	tetrahydro-5-			
	nonyl-, me			
Note -	me – Methyl ester	dima di math	wl actors	

Note - me - Methyl ester, dime - di methyl esters

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 Table 3 Fuel properties of the biodiesel obtained from waste cooking oil.

Parameters	Values	ASTM
% yield	76.44	
Kinematic viscosity at 40 °C	4.98	1.9-6.0 ASTM D 445-06
Sulfur content	0.02	
Flashpoint (°C)	135	>130 ASTM D 93-08
Pour point (°C)	-7	(-15) – 10 ASTM D 97-11
Cloud point (°C)	9	3-12
Specific gravity (g/cm ³)	0.85	0.845 ASTM D 1298-99

Biodiesel Yield

Table 3 depicts a Biodiesel yield of 76.44 %. This yield is slightly higher than the yield reported for the enzyme (Bashir et al, 20222), and alkaline (at initial catalyst loading of 0.13 g NaOH) catalyze transesterification (Mandari & Devarai, 2021). This reveals the catalysts have a good potential for the transesterification of waste cooking oil.

Kinematic Viscosity at 40°C

Kinematic viscosity is a measure of the resistance of fluid flow under the influence of gravity (Gerpen *et al.*, 2004). The result of this work (4.98 mm/s^2) is in agreement or falls within the ASTM range of $1.9 - 6.0 \text{ mm/s}^2$. The viscosity of a fuel is related to the lubricity. Low-viscosity fuels are unlikely to provide satisfactory lubrication in fuel injection pumps; these often lead to seepage and increase wear (Raja *et al.*, 2011). High viscosity in fuel is responsible for the atomization of fuel, incomplete combustion, and increased exhaust emissions, choking the injections thereby forming larger droplets on injectors, ring carbonization, and accumulations of the fuel in the engine. Having a value of viscosity neither lower nor higher in the ASTM range, it indicates that the produced biodiesel is safe for use in diesel engines (Wang et al., 2006).

Flash point

The flash point is the minimum temperature at which a fuel must be heated for it to ignite an air-vapor mixture. The U.S. Department of Transportation specified 90 °C as the flash point for non-hazardous fuel (Kenneth and Bruce, 2010). The result of this work (135 °C) shows appreciable consistency with ASTM specification of (>130°C) and it shows a lower value when compared to 158 °C reported by Ayoola et al., (2012) for waste cooking oil. The high value obtained in this other study signifies that the biodiesel produced is safe for handling and can reduce the negative effect of excess methanol on diesel engine parts such as fuel pumps, seals, and elastomers (Wang et al., 2006).

Pour point

The result of this work $(-13^{\circ}C)$ is in agreement or falls within the ASTM range of -15 to -10°C. The pour point is a criterion used for the low-temperature performance of a fuel (Alamu *et al.*, 2007) and the low temperature obtained indicates the biodiesel can still flow at a very low temperature.

Cloud point

Cloud point affects the use of biodiesel in cold weather. The cloud point which is the most common measure for the tendency of a fuel to crystallize was found to be 9 °C which falls between the ASTM of 3-12°C. This could be attributed to the presence of the high amount of saturated fatty acid as revealed by the GC-MS profile of the biodiesel.

Sulphur content

Higher sulfur contents in fuels affect human health because their emissions are associated with higher mutagenic potentials when exposed to the environment. Low-sulfur fuels are an important enabler for the introduction of advanced emissions control systems. Also, fuels rich in sulfur cause engine wear and reduce the efficiency and lifespan of catalytic systems. Biodiesel fuels have traditionally been found to be virtually sulfur-free. From Table 3.3 the Sulphur content of the synthesis biodiesel is 0.02% which is almost Sulphur free and negligible compared the fossil fuel.

Specific gravity

From Table 3.3 the density of the synthesized biodiesel is 0.85 mg/g which is below that of the waste cooking oil and within the ASTM D acceptable value of 0.845 mg/g (ASTM D 1298-99). This gives biodiesel better atomization and good combustion properties in diesel engines in comparison to pure oils that usually are $0.90 - 0.99 \text{ g/cm}^{-1}$.

4.0 Conclusions

In this study, successful transesterification of waste cooking oil was achieved using polyoxovanadate (POV). A biodiesel yield of 76.44 % was obtained with a catalyst loading of 0.8 g, employing a 1:9 oil-to-methanol ratio, at a temperature of 60 C for 90 minutes. The resulting biodiesel predominantly comprises saturated fatty acid methyl esters. Furthermore, the produced reveals favorable fuel properties that align with the standards set by ASTM for diesel engines. Moreover, its low sulfur content signifies its environmentally friendly nature compared to petroleum diesel.

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Declaration of conflict of interest

The authors declare no conflict of interest.

References

 A. O. C. S. (2003). Cd 3-25., Official Method Saponification
 Value, Sampling and Analysis of Commercial Fats and Oils.
 Copyright The American Oils Chemist's Society. Urbana Illinois. The USA.

Abdulkareem A. S., Uthman H., Afolabi A. S., Awenebe O. L. (2011). Extraction and optimization of oil from *moringa oleifera* seed as an alternative feedstock for the production of biodiesel, In Dr. Nayeripour, M. Author, *Sustainable Growth and Applications in Renewable Energy Sources*, 1 Ed., InTech, 243-268

Alamu O.J., Waheed, M. A. & Jekayinfa, S. O (2007). Alkalicatalysed Laboratory Production and Testing of Biodiesel Fuel from Nigerian Palm Kernel Oil. Fuel, 08. 011

AOCS (1980). Cd 3-25., Official Method Saponification Value, Sampling and Analysis of Commercial Fats and Oils. Copyright The American Oils Chemist's Society. *Urbana Illinois. The USA*.

ASTM D445-15 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity), DOI:10.1520/D0445-15, accessed 10.08.2015.

ASTM, (2002). ASTM International, Standard Test Method for Oxidation Onset Temperature of Hydrocarbons by Differential Scanning Calorimetry (E-2009-02). Annual Book of Standards, Section 12. 10, ASTM International, West Conshohocken, PA (2002) pp. 734–738.

Ayoola, P. B., Adeyeye, A. & Onawumi, O. O. (2012). Chemical evaluation of groundnut (Arachi hypogaea) seed. American Journal of Food and Nutrition. http://doi.org/2012.2.3.55.57

Barbosa, G. N., Graeff, C. F., & Oliveira, H. P. (2005). Thermal annealing effects on vanadium pentoxide xerogel films. *Eclética Química*, *30*, 7-15.

Bashir, M. A., Wu, S., Zhu, J., Krosuri, A., Khan, M. U., & Aka, R. J. N. (2022). Recent development of advanced processing technologies for biodiesel production: A critical review. *Fuel Processing Technology*, 227, 107120.

Batista, C., Teixeira, V., & Carneiro, J. (2008). Structural and morphological characterization of magnetron sputtered nanocrystalline vanadium oxide films for thermochromic smart surfaces. In *Journal of Nano Research* (Vol. 2, pp. 21-30). Trans Tech Publications Ltd.

Boakye, P., (2013). Biodiesel Production from Selected Nonedible Feedstocks Using Unsupported Potassium Carbonate, M.Sc Thesis, Kwame Nkrumah University of Science and Technology Ghana, 2013. Retrieved from, net/123456789/5515

Brooker, R. P., Bell, C. J., Bonville, L. J., Kunz, H.R., & Fenton, J. M. (2015). Determining vanadium concentrations using the UV-Vis response method. *Journal of The Electrochemical Society*, *162*(4), A608.

Canakci, M. & Sanli, H. (2008). Biodiesel Production from various feedstock and their effect on the fuel properties. Journal of Industrial Microbiology & Biotechnology, 35, 431-441. http://doi.org/10.1007/s10295-008-0337-6

Dahlquist, E, Gardy, J. (2010). Biodiesel production from waste cooking oil catalyzed by TiO 2–MgO mixed oxides Related papers. <u>http://doi.org/10.1016/j.biortech.2010.07.066</u>

Dijkstra, A. J. (2015). Vegetable Oils: Composition and Analysis. http://doi.org/ 10.1016/B978-0-12-384947-2.00708-X

Gerpen J. H., & He B. B. (2014). Biodiesel and Renewable diesel production methods. University of Idaho, USA http://doi.org.10.1533/9780857097385.2.441

Gumerova, N. I., & Rompel, A. (2020). Polyoxometalates in solution: speciation under spotlight. *Chemical Society Reviews*, 49(21), 7568-7601.

Haas, Michael & McAloon, Andrew & Yee, Winnie & Foglia, Thomas. (2006). A process model to estimate biodiesel production costs. Bioresource technology. 97. 671-8. 10.1016/j.biortech.2005.03.039.

Helmi, M., Tahvildari, K., Hemmati, A., & Safekordi, A. (2021). Phosphomolybdic acid/graphene oxide as novel green catalyst using for biodiesel production from waste cooking oil via electrolysis method: Optimization using with response surface methodology (RSM). *Fuel*, 287, 119528.

Kenneth, R. S., & Bruce, A. M. (2010). Market penetration of biodiesel. *International Journal of Energy And*

Environment, 1(1), 53-68.

Leung, D. Y. C., & Guo, Y. (2006). Transesterification of neat and used frying oil: optimization for biodiesel production. *Fuel processing technology*, *87*(10), 883-890.

Li M, Zheng Y, Chen Y, Zhu X. Biodiesel production from waste cooking oil using a heterogeneous catalyst from pyrolyzed rice husk. Bioresour Technol. 2014 Feb;154:345-8. doi: 10.1016/j.biortech.2013.12.070. Epub 2013 Dec 22. PMID: 24405650.

Mandari, V., & Devarai, S. K. (2021). Biodiesel production using homogeneous, heterogeneous, and enzyme catalysts via transesterification and esterification reactions: A critical review. *BioEnergy Research*, 1-27.

Meng, X., Chen, G., Wang, Y. (2008). Biodiesel production from waste cooking oil via alkali catalyst and its engine test. http://doi.org/ 10.1016/j.fuproc.2008.02.006

Patil, P. D., Gude, V. G., Reddy, H. K., Muppaneni, T., & Deng, S. (2012). *Biodiesel Production from Waste Cooking Oil Using Sulfuric Acid and Microwave Irradiation Processes*. 2012(January), 107–113.

Sadaf, S., Iqbal, J., Ullah, I., Nawaz, H., Nouren, S., (2018). Biodiesel production from waste cooking oil: An efficient technique to convert waste into biodiesel. https://doi.org/10.1016/j.scs.2018.05.037

Wang, S., & Yang, G. (2015). Recent Advances in Polyoxometalate-Catalyzed Reactions. https://doi.org/10.1021/cr500390v

Wang, X.H., Aliyari, R., Li, W.X., Li, H.W., Kim, K., Carthew, R., Atkinson, P., Ding, S.W. (2006). Supporting online material for RNA interference directs innate immunity against viruses in adult Drosophila. Science 312(5772)

Xie, W., Gao, C., & Wang, H. (2020). Biodiesel Production from Low-Quality Oils Using Heterogeneous Cesium Salts of Vanadium-Substituted Polyoxometalate Acid Catalyst. *Catalysts*, 10(9), 1060. MDPI AG. Retrieved from http://dx.doi.org/10.3390/catal1009Zhang, Y. (2016). VO2 (B) conversion to VO2 (A) and VO2 (M) and their oxidation resistance and optical switching properties. *Materials Science-Poland*, 34(1), 169-176.

Zhao, L., Miao, L., Liu, C., Li, C., Asaka, T., Kang, Y., ... & Su, H. (2014). Solution-processed VO2-SiO2 composite films with simultaneously enhanced luminous transmittance, solar modulation ability, and anti-oxidation property. *Scientific reports*, *4*(1), 1-11.