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The Production of Biodiesel from Sewage Sludge Via Soxlet Extraction Using Hexane and Ethyl Acetate in Ratio 2:1 As the Organic Solvent and Transesterification Reaction

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Authors' contributions

This work was carried out in collaboration between both authors. Author WI designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author BO managed the analyses of the study and literature searches. Both authors read and approved the final manuscript.

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ABSTRACT

The Lipid/oil was extracted from reduced dried primary sewage sludge particle using soxhlet extraction method with Hexane and Ethyl Acetate in the ratio 2:1 as the extracting solvent. The extracted oil gave 15.03% percentage yield with density of 0.845 g/ml, pH value of 8.97, specific gravity of 0.845, viscosity of 41.20 mm2/sec, kinematic viscosity of 42.18 mm2/sec. The lipid/oil was brownish black in colour with a pungent smell. The chemical analyses revealed saponification value of 168.00 mgKOH/g, acid value of 5.60 mgKOH/g and free fatty acid value of 2.80%.

The physicochemical analyses of the biodiesel produced gave a c percentage yield of 40% biodiesel, density of 0.845 Kg/ml, pH value of 8.50, specific gravity of 0.845, kinematic viscosity of 4.80 mm2/s, acid value of 0.37 mg KOH/g, and flash point of 150°C, cloud point of 5°C and pour point of -2°C. The results of the physicochemical parameters from the research shows that the feedstock (primary sewage sludge) would be a good source for the production of biodiesel.

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1. INTRODUCTION

There has been a tremendous growth recently over the demand for petroleum product in the transportation sector, which has led to increase in crude oil price. The depletion in fossil fuels around the globe, calls for a need to search for alternative source to replace these geological deposits of organic materials. In addition, there has been a massive problem in reducing pollutant emissions; and millions of money has been spent to reduce the pollution arising from global warming, acidification, deforestation, ozone depletion, eutrophication and photochemical smog. This has necessitated the need for research on the possibilities of having alternative renewable energy source as a substitute to fossil fuel.

Biodiesel also known as fatty acid methyl esters (FAME) is a mixture of monoalkyl esters of long chain fatty acids derived from lipids which can be obtained from oil, fat, waste oil and many more via transesterification reaction [1]. It is catalyzed by either acid or base catalyst, and also produces glycerol as its side product. The use of biofuels seems to be a better substitute for the fossil fuels because, its production resources are renewable, less expensive in comparison with fossil fuels, more environmentally friendly and also accessible to distribute and use.

The extraordinary human knowledge has resulted in the process of converting sewage sludge to produce energy in the form of renewable biofuel such as biodiesel which is quite complicated but economically valuable. Lipid is a natural mixture of triglycerides, diglyceride, monoglyceride, cholesterols, free fatty acids and phospholipids [2]. A portion of the collected primary sludge recycled back to the aeration basins to maintain a sufficient concentration of microorganism [3] to ensure quality biodiesel.

This research work, investigates the possibility of producing biodiesel from sewage sludge by first extracting lipids from samples obtained from a sewage treatment plant located in Udu local government area of Delta State, Nigeria, via soxhlet extraction with the mixture of hexane and ethyl acetate (2:1) as solvent; and converting the lipids to biodiesel through transesterification reaction.

2. MATERIALS AND METHODS

2.1 Study Site

Solid primary sewage sludge collected from a sewage treatment plant located in Udu local government area of Delta State, Nigeria, a subsidiary of Delta state waste water management office, which was carved out of the one-time sewage department of the office of drainage services in the ministry of the environment, Delta state, to oversee waste water management in the state. The office was incorporated into the Delta state water corporation. The creation of the office arose from the need for an institutional reform in wastewater and related matters in Delta state.

2.2 Samples Collection and Preparation

Solid primary sewage sludge was collected from the waste water treatment plant. The primary sewage sludge was pretreated using mechanical method and this involves switching off the connection between the sump and the aerator for about 12 hours, therefore allowing the primary sewage sludge to settle beneath the sump before collection. The sample was collected and sun dried for seven days to remove moisture, the dried primary sludge was reduced by using a pestle and a mortar, where it was pounded until it was finally reduced to granule. The reduced particles were weighed using the electronic weighing balance.

2.3 Sample Extraction

The reduced primary sewage sludge oil was extracted by soxhlet extraction techniques, using a mixture of hexane and ethylacetate (2:1) as the organic solvent. The extracted oil was obtained after the organic solvent was removed under reduced temperature and pressure and refluxing at 70°C so as to remove any excess organic solvent used for the oil extracted. The extracted oil was stored in refrigerator/ freezer at 2°C for subsequent physicochemical analyses [4].

2.4 Physicochemical Analyses of the Extracted Oil

Physicochemical parameters were carried out on the extracted oil obtained and they include Percentage yield, pH value, Density, Specific

gravity, Saponification value, Acid value/Free fatty acid, Viscosity and Kinematic Viscosity.

2.5 Production of the Biodiesel

The production processes were carried out in two steps and that include the Acid Catalysed Transesterification Process also known as Esterification Process and the Base catalysed transesterification process. Esterification with the extracted oil was carried out with 95.7 ml of methanol and 2.1ml of concentrated sulphuric acid (catalyst). This was heated at a temperature of 60°C for about 1 hour. This process was done to reduce the percentage of free fatty acid in the oil [5,6].

The esterified oil was then transesterified by reacting the esterified oil with 16.1 ml of methanol and 1.6ml of sodium hydroxide NaOH (catalyst); and was heated at a temperature of 60°C for 1 hour with constant stirring of the mixture using a hot plate magnetic stirrer. After the reaction process, the mixture was transferred into a separatory funnel, allowed to settle; and cooled while the separation of the two phase layer took place to separate into the methyl ester phase (Biodiesel) and glycerin phase (Glycerol) as the upper and lower phase respectively [7].

2.6 Physicochemical Analysis of the Produced Biodiesel

The biodiesel was further analyzed using ASTM procedure for the physicochemical analysis and physicochemical parameters carried out includes percentage yield, pH value, density, specific gravity, acid value/free fatty acid, viscosity, kinematic viscosity, flash point, cetane number, cloud point and pour point.

3. RESULTS AND DISCUSSION

The experimental result of the physicochemical analyses conducted on the extracted oil is presented in Table 1, while those on the biodiesel produced in accordance with the ASTM standard in Table 2, respectively.

The oil has a brownish black colour and a pungent smell. The percentage yield from calculations was found to be 15.03% and this was found to be lower than the yield obtained for lageneria siceraria seed oil with a value of 39.22% [8]. Although, sewage sludge can readily be obtained in sufficient quantities and its use as a potential source of lipid for biodiesel production is still viable. The pH value for the extracted oil was 8.97.

Table 1. Summary of the physico-chemical analysis result on the extracted oil

Physicochemical parameters	Results
Colour of oil	Brownish black
Smell	Pungent odour
Percentage yield (%)	15.03%
pH	8.97
Density (g/ml)	0.845
Specific gravity	0.845
Viscosity (mm^2/s)	41.20
Kinematic viscosity(mm^2/s)	42.18
Acid value (mg KOH/g)	5.60
FFA (%)	2.80
Saponification value (mg KOH/g)	168.00

Density is the ratio of the mass or weight (g) of a substance to the volume of the substance (ml). The density of the extracted lipid was found to be 0.845 g/ml, being lower than water (1.0 g/ml). Specific gravity is the ratio of the density of a substance to the density of water at 4°C [9]. The extracted oil had a specific gravity of 0.845 [6].

The acid value obtained from the extracted oil is 5.60 mgKOH/g and this is smaller than 10.3 mgKOH/g reported for Sheanut butter and higher when compared to 2.455 mgKOH/g, 1.265 mgKOH/g and 0.82 mgKOH/g for castor seed oil, jatropha oil and cotton seed oil respectively. The higher the acid value of an extracted oil, the lower its storage quality and vice-versa, this simply shows that the extracted oil have better storage quality when compared to that of Sheanut butter oil [10].

The free fatty acid value was found to be 2.80% which is lower than the Hyptus spicigera seed oil with a value of 3.50% (Ladan et al., 2010). The high FFA (>2%) favours soap formation.

The saponification value of the extracted lipid was found to be 168.00 mgKOH/g which is lower than the value of 183.1 mgKOH/g for sheabutter oil and 199.95 mgKOH/g of cotton seed oil, but higher than 126.728 mgKOH/g, 125.081 mgKOH/g for castor seed oil and jatropha oil respectively, indicating a potential for soap production [10]. This suggests that the extracted oil will also be a good source for soap making due to its high saponification value [10].

Table 2. Summary of the physicochemical analysis results on the biodiesel produced

Physicochemical parameters	Experimental results on the biodiesel	Test method	ASTM standard results limits D-6751-02
Percentage yield (%)	40	-	-
Density at 15°C (Kg/ml)	0.845	ASTM D1298	0.830 – 0.890 Kg/ml
Specific gravity	0.88	ASTMD1298/4052	0.89 max
Viscosity (mm^2/s) at 40°C	4.70	ASTM D445	3.5 – 5.0 mm^2/s
pH	8.50	-	-
Kinematic viscosity (mm^2/s) at 40°C	4.80	ASTM D445	1.9 – 6.0 mm^2/s
Acid value (mg KOH/g)	0.37	ASTM D664	0.50 max
Flash point (°C)	150	ASTM D93	130 min
Cetane number (min)	56.8	ASTM D613	47 min
Cloud point (°C)	5	ASTM D2500	Report to customer
Pour point (°C)	-2	ASTM D97	-

The percentage yield (%) of the biodiesel produced was found to be 40% from calculations. The density of diesel fuel is an important property that affects the fuel injection system and high density translates into a high consumption of the fuel. The biodiesel produced has a density of 0.845 Kg/ml and a specific gravity of 0.85 which falls within the ASTM standard range at 15°C [11].

The viscosity of a liquid is an important property of diesel fuel, it measures the resistance to flow and affects the engine fuel injection system at low temperatures. A highly viscous fuel will result in poor atomization hence a loss of power of the engine and production of smoke. The biodiesel produced has a viscosity of 4.7 mm^2/s which is slightly viscous but still close to that of the petroleum diesel. This is an advantage for the use of the biodiesel. The kinematic viscosity of the biodiesel (4.8 mm^2/s) is consistent with the conventional biodiesel standard. The 40^oC reference point is a parameter required for biodiesel and petroleum diesel standards. At this temperature, the ASTM standard of diesel oil kinematic viscosity with range between 1.9 – 6.0 mm^2/s [11].

This implies that the primary sewage sludge, from which the biodiesel was produced, satisfies the fluidity requirement.

Acid value is a direct measure of the level of free fatty acids that may be present in biodiesel. The acid value of the biodiesel produced was found to be 0.37 mg KOH/g. This shows that the acid value of the biodiesel produced is in agreement with the conventional biodiesel standard, which falls in range of 0.50 max [7].

The flash point was found to be 150°C and the falls within the flammability limit of diesel fuel. This value is reasonably good for the purpose of handling/storage.

The cetane number of a fuel is a measure of the ignition quality of the fuel, the higher the cetane number, the better the ignition quality. The cetane number of the biodiesel produced was found to be 56.8 min. The value obtained falls within the limit specified for biodiesel fuels by biodiesel standard with range within 47 min. This means that it will burn smoothly and with less noise in a diesel engine than petroleum diesel.

Other physicochemical parameters like cloud point and pour point also falls within the ASTM standard for biodiesel.

4. CONCLUSION

The soxhlet extraction process using n-hexane and diethyl ether was able to extract lipids from primary sewage sludge. The lipid extracted was successfully used through transesterification reaction to produce biodiesel which can serve as a viable alternative to petroleum diesel. The physicochemical properties of the biodiesel produced are consistent with ASTM standards for biodiesel. Although, a percentage yield of 15.03 % was obtained for lipid from the sewage sludge, while 40% recovery was recorded for biodiesel via transesterification reaction, the availability of human excreta in significant quantity makes this research finding a reasonable alternative to petroleum diesel. This research is still open for further investigation, especially exploring the use of other solvents mixtures for the enhancement of lipid yield; as well as the best operating conditions for

maximum biodiesel production through the transesterification process.

The physicochemical parameters obtained from this study for the extracted oil from the primary sewage sludge; and the biodiesel produced from the extracted oil via transesterification reaction, being within the ASTM range, shows a promising source of lipids for the commercial production of biodiesel in the future as an alternative energy source to fossil fuel.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Demirbas A. Biodiesel from waste cooking oil via base catalytic and supercritical methanol transesterification. Energy Conversion and Management. 2009;50(4): 923-927.
2. Alhassan FH, Uemura Y. Isopropanolysis of cottonseed oil to biodiesel via potassium hydroxide catalyst. Elsevier: Procedia Engineering. 2016;148:473–478.
3. Bharathi P, Karthiga R, Shamli M, Vivetha K, Subathra M. Extraction of lipids from municipal sewage sludge for production of biodiesel via transesterfication process. International Journal of Pharma and Bio Sciences. 2014;5(4):B132-B141.
4. Warra AA, Wawata IG, Hassan LG, Gunu SY, Aujara KM. Extraction and physicochemical analysis of some selected Northern Nigerian industrial oils. Scholars Research Library: Archives of applied science research. 2011;3(4):536-541.
5. Karaosmanoglu F, Cigizoglu KB, Tuter M, Ertekin S. Investigation of the refining step of biodiesel production. Energy and Fuels. 1996;10:890-895.
6. Lang X, Dalai AK, Bakhshi NN, Reaney MJ, Hertz PB. Preparation and characterisation of biodiesels from various bio oils. Bioresour. Technol. 2011;80:53-62.
7. Gerpen JV, Shanks B, Pruszko R, Clements D, Knothe G. Biodiesel production technology. National Renewable Energy Laboratory. Colorado. 2004;105.
8. Hassan LG, Sani NA. Comparative studies on the physico-chemical properties of bottle gourd lageneria siceraria seeds oils extracted by two methods. Nigeria Journal of Basic and Applied Sciences. 2007; 15(1)(2):50-51.
9. Bello EI, Agge M. Biodiesel production from ground nut oil. Journal of Emerging Trends in Engineering and Applied Sciences. 2012;3(2):276-280.
10. Warra AA, Gunu SY, Jega A, Aisha JA. Soap production from sheanut butter. International Journal of Applied Sciences. 2011;5(4):410-412.
11. Idibie CA, Awatefe KJ, Ogboru RO. Biodiesel production from dika seed (*Irvingia gabonensis*) oil via soxhlet extraction and transesterification reaction. Journal of Chemical Society of Nigeria. 2020;45(1):143-148.

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