International Journal of Chemical Studies

P-ISSN: 2349–8528
E-ISSN: 2321–4902
www.chemijournal.com
IJCS 2020; 8(4): 3652-3655

Received: 19-04-2020
Accepted: 05-06-2020

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The production of biodiesel from sewage sludge via soxhlet extraction using petroleum ether and transesterification reaction

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DOI: https://doi.org/10.22271/chemi.2020.v8.i4at.10215

Abstract

The Lipid/oil was extracted from reduced dried primary sewage sludge particle via soxhlet extraction method with petroleum ether as solvent. The extracted oil gave 19.15% percentage yield with density of 0.992g/ml, pH value of 10.85, specific gravity of 0.992, viscosity of 41.05mm2/s, kinematic viscosity of 41.38mm2/s. The lipid/oil was brownish black in colour with a pungent smell. The chemical analyses revealed saponification value of 115.95mgKOH/g, acid value of 4.10mgKOH/g and free fatty acid value of 2.05%.

The physicochemical analyses of the biodiesel produced revealed percentage yield of 70% biodiesel, density of 0.880Kg/ml, pH value of 7.99, specific gravity of 0.88, viscosity of 4.9mm2/s, kinematic viscosity of 5.6mm2/s, acid value of 0.47mg KOH/g, flash point of 160°C, cetane number of 50.7min and copper strip corrosion of No. 1a, sulfated ash of 0.019% mass, cloud point of 10°C and pour point of -4°C, indicative of a promising source of biodiesel.

Keywords: lipids, primary sewage sludge, biodiesel, transesterification.

Introduction

Since the last century, the consumption of energy has increased tremendously due to a corresponding increase in human activities and the significant growth of population. This increase in energy demand has been supplied by the use of fossil resources, resulting in its depletion, increase in its price and a resultant environmental impacts such as global warming, acidification, deforestation, ozone depletion, eutrophication and photochemical smog. As fossil fuels are limited sources of energy, this increasing demand for energy has necessitated the search for alternative sources of energy that would be economically efficient, socially equitable, and environmentally friendly. Two of the main contributors of this increase of energy demand have been the transportation and the basic industry sectors, being the largest energy consumers.

The transport sector is a major consumer of petroleum fuels such as diesel, gasoline, liquefied petroleum gas (LPG) and compressed natural gas (CNG) [1]. Demand for transport fuels has risen significantly during the past few decades and continues to increase; and expectations are that this trend will stay unchanged for the coming decades.

The use of biofuels appears to be a credible substitute for the fossil fuels. This is because, resources for its production are renewable (as fresh supplies can be re grown), they are becoming more cost effective in comparison with fossil fuels, they appear to be more environmentally friendly and also accessible to distribute and use, given that applicable infrastructures and technologies exists and are readily available.

Conventional fuel, however, are predicted to become scarce as petroleum reserves are limited [1]. For this reason, these fuels are set to become increasingly costly in the coming decades.

Renewable fuels, made from biomass have enormous potential and can meet many times the present world energy demand. Biomass can be used for energy in several ways; one of these is the conversion into liquid or gaseous fuels such as ethanol and bio-diesel for use in mobile source combustion [2]. Global demand for liquid biofuels more than tripled between 2000 and 2007. Future targets and investment plans suggest strong growth will continue in near future. Biofuels appear to be more environment friendly in comparison to fossil fuels, considering the emission of greenhouse gases when consumed. Examples of those gases are carbon dioxide (CO_2), methane (CH_4) and nitrous oxide (N_2O). Those gases pose serious risks as they tend to warm the earth's surface. The energy content of biofuels differs from conventional fuels.

Biodiesel production is pretty recent; and therefore provides a good technological platform for researchers as an alternative fuel for diesel engines, due to the increase in the petroleum price, its renewability and the environmental advantages [3]. Biodiesel can be produced from renewable sources such as vegetable oil, animal fat and used cooking oil. Currently, the cost of biodiesel is high as compared to conventional diesel oil because most of the biodiesel is produced from pure vegetable oil. However, the cost of biodiesel can be reduced by using low cost feedstock such as municipal sewage sludge [3].

More recently, the use of municipal sewage sludge as a potential source of lipids for the production of biodiesel production has opened up [3]. Municipal sewage sludge has the potential to be exploited as a good and high energy feedstock for biodiesel production in the future. These waste water treatment plant produces primary and secondary sludge [4].

This research work, investigates the possibility of producing biodiesel from sewage sludge by first extracting lipids from samples in Lagos State treatment plant, via soxhlet extraction with petroleum ether as solvent; and converting the lipids to biodiesel through transesterification reaction.

Materials and methods

Study Site

Solid primary sewage sludge collected from Lagos state waste water treatment plant, Lagos state in Nigeria, a subsidiary of Lagos state waste water management office which was carved out of the erstwhile sewage department of the office of drainage services in the ministry of the environment, Lagos state, to oversee waste water management in the state. The office was incorporated into the Lagos state water corporation. The creation of the office arose from the need for an institutional reform in wastewater and related matters in Lagos state.

Samples collection and preparation

Solid primary sewage sludge was collected from Lagos state waste water treatment plant. The primary sewage sludge was pretreated using mechanical method and this involves switching off the connection between the sump (A hollow or pit into which liquid drains, such as a cesspool, cesspit or sink) and the aerator for about 12 hours therefore allowing the primary sewage sludge to settle beneath the sump before collection.

The primary sewage sludge was then collected and dried for 7 days with a little amount of sun and air (i.e. sun and air drying). Then particle reduction was done using local pestle and mortar. The reduced particles were weighed using the electronic weighing balance.

Sample Extraction

The reduced primary sewage sludge oil was extracted by soxhlet extraction and using petroleum ether as the organic solvent. The extracted oil was obtained after the organic solvent was removed under reduced temperature and pressure and refluxing at 70^{o}C so as to remove any excess organic solvent used for the oil extracted. The extracted oil was stored in refrigerator/ freezer at 2^{o}C for subsequent physicochemical analyses [5].

Physicochemical Analyses of the Extracted Oil

Various 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.

Production of the biodiesel

The production process was carried out in two steps: 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 95.7ml 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 in order to reduce the percentage of free fatty acid in the oil [6, 7].

The esterified oil was then transesterified by reacting the esterified oil with 16.1ml 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 and 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 [8].

Physicochemical analysis of the produced biodiesel

Various physicochemical parameters were carried out on the biodiesel obtained and they include percentage yield, pH value, density, specific gravity, acid value/free fatty acid, viscosity, kinematic viscosity, flash point, sulfated ash, cetane number, copper strip corrosion, cloud point and pour point.

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 vis-a-vis the ASTM standard in table 2, respectively.

Table 1: Summary of the physicochemical analysis on the extracted oil

Physicochemical Parameters	***Results***
Colour of oil	Brownish black
Smell	Pungent odour
Percentage yield (%)	19.15
Ph	10.85
Density (g/ml)	0.992
Specific gravity	0.992
Viscosity (mm^2/s)	41.05
Kinematic viscosity(mm^2/s)	41.38
Acid value (mg KOH/g)	4.10
FFA (%)	2.05
Saponification value (mg KOH/g)	115.95

The oil has a brownish black colour and a pungent smell. The percentage yield from calculations was found to be 19.15%. This value is lower when compared to *Lageneria siceraria* seed oil that has a yield of 39.22% [9]. Although the yield is low, but given that sewage sludge can readily be obtained in sufficient quantities, its use as a potential source of lipid for biodiesel production is still viable. The pH value for the extracted oil was 10.85.

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 0.992 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 [10]. The extracted oil had a specific gravity of 0.992 [7].

The acid value obtained from the extracted oil is 4.10 mgKOH/g. This value is smaller than 10.3 mgKOH/g reported for sheanut butter; and higher when compared to 2.455mgKOH/g, 1.265mgKOH/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, implying that the extracted oil have better storage quality when compared to that of sheanut butter oil [11].

The free fatty acid value was found to be 2.05% which is lower than that of *Hyptus spicigera* seed oil with a value of 3.50% [12].

The saponification value of the extracted lipid was found to be 115.95mgKOH/g, being lower than the value of 183.1mgKOH/g for sheabutter oil and 126.728mgKOH/g, 125.081mgKOH/g and 199.95mgKOH/g for castor seed oil, jatropha oil and cotton seed oil respectively, which have potential for soap production [11]. This suggests that the extracted oil will not be a good one for soap making due to its low saponification value [11].

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 (%)	70	-	-
Density at 15ºC (Kg/ml)	0.880	ASTM D1298	0.860 – 0.890Kg/ml
Specific gravity	0.88	ASTM D1298/4052	0.89max
Viscosity (mm^2/s) at 40ºC	4.9	ASTM D445	3.5 – 5.0 mm^2/s
Ph	7.99	-	-
Kinematic viscosity (mm^2/s) at 40ºC	5.6	ASTM D445	1.9 – 6.0 mm^2/s
Acid value (mg KOH/g)	0.47	ASTM D664	0.50 max
Flash point (ºC)	160	ASTM D93	130min
Cetane number (min)	50.7	ASTM D613	47min
Copper strip corrosion	No. 1a	ASTM D130	No. 3 max
Sulfated ash (% mass)	0.019	ASTM D874	0.020max
Cloud point (ºC)	10	ASTM D2500	Report to customer
Pour point (ºC)	-4	ASTM D97	-

The percentage yield (%) of the biodiesel produced was found to be 70% from calculations. The density of diesel fuel is an important property that affects the fuel injection system. Density of biodiesel at 15ºC is the weight of a unit volume of the biodiesel. The fuel injection equipment meters the fuel volumetrically, and high density translates into a high consumption of the fuel. The biodiesel produced has a density of 0.880Kg/ml and a specific gravity of 0.88 which falls within the ASTM standard range at 15ºC [13].

The viscosity of a liquid is a measure of its resistance to flow; this is a very important property of a diesel fuel because it affects the engine fuel injection system predominantly 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 4.9mm^2/s which is slightly viscous but still close to that of the petroleum diesel. This is an advantage of biodiesel over its source oils. The kinematic viscosity of the biodiesel (5.6mm^2/s) is consistent with the conventional biodiesel standard. The 40ºC reference point is a parameter required for biodiesel and petroleum diesel standards. At this temperature, the biodiesel has ASTM standard kinematic viscosity with range between 1.9 – 6.0mm^2/s [13]. 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 0.47mg KOH/g. This shows that the acid value of the biodiesel produced is in agreement with the conventional biodiesel standard which falls in range within 0.50max [8]. The flash point was found to be 160ºC. 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 50.7min. The value obtained fall within the limit specified for biodiesel fuels by biodiesel standard with range within 47min. This means that it will burn smoothly and with less noise in a diesel engine than petroleum diesel.

Other physicochemical parameters like cloud point, pour point and copper strip corrosion test also falls within the ASTM standard for biodiesel.

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Cloud point (ºC)	10	ASTM D2500	Report to customer
Pour point (ºC)	-4	ASTM D97	-

Conclusion

In conclusion, 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.

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