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DETERMINATION OF PHYSICO-CHEMICAL PROPERTIES OF CHRYSOPHYLLUM ALBIDUM SEED OIL TOWARDS BIODIESEL PRODUCTION

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ABSTRACT

A systematic physicochemical investigation was carried out on Chrysophyllum albidum seed oil for its suitability and applicability in biodiesel production. The oil was extracted using hot continuous extraction process with n-hexane as solvent and analyzed for sulphur content, ash content, heat of combustion, acid value, specific gravity, free glycerol and total glycerol. Others are saponification and free fatty acid values. The results showed the oil content was 22%. The density, kinematic viscosity @40^0C, pour point, flash point, cloud point, cetane number and Iodine value are $0.89g/cm^3$, $2.97mm^2/sec$, 2.14^0C, 167^0C, 3.40^0C, 40.1 and $67.60gI_2/100g$ respectively. All the values obtained were within either ASTM or EN standards.

KEYWORDS: Chrysophyllum albidum seed oil, biodiesel, physico-chemical properties, proximate analysis.

INTRODUCTION

Petroleum, natural gas and coal had being some of non-renewable resources of meeting the world energy supply (Babatunde *et al*., 2016). Large increase in number of electric generating sets of different sizes and specification in recent ages has resulted in great request for petroleum products (Jaichander and Annamalai, 2011) studies revealed that increase in consumption of petroleum products had led to depletion in the resources, increase in cost and pump prices coupled with deterioration of ambience which in turns cause global warming (Hossain *et al.*, 2010, Tint and Mya, 2009**;** Pankaj *et al*., 2006 and Halek *et al.*, 2009).

LITERATURE REVIEW

All over the world there had being an agitation for alternatives, out of which biodiesel is one. Biodiesel can be produced from livestock of agricultural derived sources. Several numbers of researchers had worked on the production of biodiesel using different vegetable oils or bio-oils such as soybean oil (Hossain *et al.*, 2010), neem seeds oil (Anya *et al.*, 2012), jatropha carcus (Anthony *et al.*, 2011; Tint and Mya, 2009; Hanny and Shizuko, 2007) chicken fat (Ertan *et al.,* 2011) and cooking oil (Rieberio *et al*., 2010).

Biodiesel is off advantage over petrol diesel when considering some factors like lubrication, because, better results are obtain in the lubricity test performed and fewer wear particles particle are produced than petrol diesel.

The presence of oxygen in ester molecules in the spring nucleus makes it to have fewer fume opacity, improvement in fuel performance and lower unburnt carbon-monoxide and hydrogen carbon emission. It is a long chain of carbon that makes the aromatic content therein diluted thus reducing the particles. Apart from all these, there is neutral balance of carbon dioxide emission due to the fact that it is biological sources. (Ertan *et al.*, 2001and Demirbas (2009).

In order to forestall engine operational problems over time, proper physicochemical analysis of any proposed feed stock for biodiesel production should be done, among which are being consider in this paper.

Chrysophyllum albidum is one of the native plant found in the coast of West Africa, it is one of the potential plant which its seed can be used to produce biodiesel which considering it abundance, low cost, no edibility and untapped value thereof.

Jayeoba *et al.,* 2007 studied the chemical composition and physical properties of Chrysophyllum albidum considering different part like the peel, pulp and juice. Adepoju *et al*., explored the nutrient compositions along with micronutrient potential of pulps of three widely grown types of Chrysophyllum albidum seed. Further studies were carried out by Olaoluwa *et al.,* 2012 on biochemical assessments of minerals and anti-nutritional constituent present in fermented chrysophilum albidium seed meal. Extraction and characterization of Chrysophyllum albidium and Luffacyl indrica seed oils were examined by Audu *et al.*, 2013.

Chrysophyllum albidum seed oil warrants a systematic investigation because limited studies were carried out on it in terms of biodiesel production. Hence adequate physicochemical characterization consideration is very important so as to ensure its suitability and applicability in the production of biodiesel. Some of these properties are; kinematic viscosity, density, specific gravity, dew point, cloud point, flash point, cetane number. Others are: ash and sulphur contents, iodine, saponification and acid values, free fatty acid, total and free glycerol.

MATERIALS AND METHODS

Chrysophyllum albidum

Chrysophyllum albidum seed apple bulbs were collected under the parent plant. Some were bought at Odo-Oba market in Oyo state, Nigeria. The fruit bulbs were carefully sliced open (to remove the seeds), the seeds were de husked (removal of the brown feel coat), oven dried at 60^{o}C for 72hrs and grinded to powder using a local grinder (Olaoluwa *et al*., 2012). Oil was extracted by solvent extraction process using n-hexane (40-60^{o}C) as solvent (AOAC (1975). Methanol 99.9% purity and n-hexane which were analytical grade obtained from Merck (Germany). Subsequently, the oil was purified prior to physicochemical analysis by degumming and de-acidification process.

Degumming

This process was done to remove phosphatides that makes the oil turbid during storage and promote the accumulation of water. Here, water was added to the oil at 60-90^{o}C to dissolve the phosphatides in the oil, the mixture was allowed to settle. Centrifuging was done to separate oil from the water phase. (Audu, *et al*., 2013).

Experimental Design

The effects of reaction temperature and reaction time, agitation speed, different ratios of catalyst concentration and mole ratio of alcohol were studied in the production of biodiesel in this research work according to Mathiyazhagan and Ganapathi, (2011). This was accomplished by applying Box-Behnken factorial experimental design of Response Surface Methodology (RSM). The lower as well as upper levels of the factors used in the design are as shown on table 1.

Table 1: Experimental range of independent variables and their limits.

Independent variables	Lower Limit	Upper Limit
Temperature (^oC)	40	70
Reaction Time (h)	1	4
Agitation Rate (rpm)	400	700
Catalyst con.	1	5
Methanol: oil	7:1	4:1

Batch process of biodiesel production

Transesterification process was adopted here for biodiesel production according to Babatunde *et al.*, (2016), using a three – necked round bottom flask glass reactor system as the batch reactor on a magnetic stirrer. The condenser was fixed at the center of the reactor; the agitator was placed inside the flask and the thermometer at the left hand side. For a particular run, 100 g of methanol and 10 g of calcined catalyst were first mixed and refluxed at 50 ^oC for 1 hr. as the catalyst activation step. Then, 250 g of refined Chrysophyllum albidum seed oil was added to transesterify with stated experimental duration stipulated by design matrix as shown on table 3.2. The reactor was heated to 65 ^oC temperatures, and then magnetic stirrer was switched on at 1000 rpm on after the 65 ^oC temperatures was attained and the reaction was allowed to take place for 3hr to obtain a clear phase separation.

The mixture achieved was then filtered to remove catalyst particles, the clear solution obtained was then separated using separating funnel and the yield of biodiesel produced was calculated.

RESULTS AND DISCUSSION

Different parameters were discussed under this section using an established international standards.

The Saponification value

Saponification value measures the index of the average size of the fatty acid present, in oil sample, which depends on the molecular weight and percentage concentration of fatty acid compounds in the oil (biodiesel). The value is found to be inversely proportional to the molecular weight of oil. (Audu *et al.,* 2013). The saponification value of Chrysophyllum albidum oil obtained here is 196.85 KOH/g, this indicates high proportion of fatty acids of low molecular weight, it shows that the oil has affinity for soap and shampoo making. (Salisu *et al.*, 2012). It is comparable to 195.00mg KOH/g oil reported by Audu *et al*, 2013, 208.207 mg KOH/g oil (Tint and Mya, 2009) and 189mg KOH/g (Mohammed and Hamza, 2008) of raw Chrysophyllum albidum, Ceiva Pentandra Linn, raw Jatropha oil and Sesamum indicum L. oil respectively.

Sulphur

In diesel engine**,** the presence of sulphur results in sulphur oxide and sulphate, that is, when sulphur chemicals are released into the atmosphere they form sulphur dioxide and when combined with water it forms sulpuric acid. This sulpuric acid is then carried by wind to another region, falling onto the inhabitants, land and vegetation when it rains, or as misty or foggy (Acid rain) (Pankaj, *et al.*, 2006). Biodiesel is sulphur content free (as it is derived from vegetable oil), its use sustainably reduces the emission of SO_2 and aromatics in the atmosphere. From the analysis obtained in this research work, 7.5 ppm of sulphur was found present in the oil, which is found to be accepted according to ASTM D5453 of biodiesel production of 15 ppm maximum content in biodiesel. This is in agreement with the work of Xiaohu *et al.*, of 2009 when the investigated the preparation and characterization of biodiesel produced from recycled canola oil, the obtained a sulphur content of **15.9** which was found to be within the standard limit.

Free Fatty Acid

Most vegetable oils used in biodiesel production contain much content of free fatty acids than can enhance saponification reaction during transesterification process. (Palligarmai *et al.,* 2008). Apart from this, it provide electrolyte and acceleration of zinc corrosion, forms organic acid salts and compounds. It was discovered that lowest-cost lipid feedstocks containing high amount of free fatty acid which has to be removed by esterification with methanol by acid catalysis process (Halek *et al.*, 2009). The free fatty acid of raw Chrysophylum albidum oil was 34%, which is high and must be drastically reduce or be neutralized for biodiesel production compare to 22.6% of Tint and Mya, 2009 after neutralization.

Although acid-catalyzed transesterification is insensitive to FFA in the feedstock, nevertheless, it requires longer reaction time and higher temperature and more so, there is evidence that the value of FFA in this feedstock will also reduce after neutralization. (Xiaohu *et al.*, 2009) and higher temperature is efficient when FFA in the oil exceed 1%. (Freedman *et al.*, 1984) and Pankaj *et al*., (2007) established alkali-catalyzed transesterification which could be completed if and only if FFA amount in the oil content is not greater than 1%.

Density @ 15^{o}C

Density @ 15^{o}C is one of the oil properties that affect the injection system of an engine and it can be measure either in g/cm^{3}or kg /m^{3}. Related to viscosity, it increases notably with respect to diesel oil. At low temperature, it generates excessive local heat in rotating distribution pumps and increase stress on the components, thereby cause premature failure of pump and also cause poor spray atomization in the nozzle.

The standards required are stated by European norm ISO 12185 for biodiesel in between 0.860 and 0,900g/cm^{3}. 0.89g/cm^{3} was obtained as the value from the analysis of ASASO, this is found to be within the limit of standard given and is found to be in line with the report of Kenneth *et al*., (2010) when looking at market penetration of biodiesel, the established decrease in densities of oil to biodiesel of different feedstocks, that is: corn oil, soya beans oil, tallow and yellow grease. This shows that a value of 0.89g/cm^{3} obtained for ASASO can still be reduced after undergoing transesterification process of producing biodiesel.

Kinetic Viscosity @ 40^{0}C

This is resistance to flow of liquids, it decreases with increase in temperature, and hence temperature at which it is being measured practically must be specified. High viscosity of vegetable oil has found to lead to unfavourable pumping and inefficient mixing of fuel with air which in turn contribute to incomplete combustion. Due to the above mentioned problems, vegetable oil must be modified to bring its combustion related properties closer to those of diesel oil. Therefore, such fuel modification is mainly aimed at reducing the viscosity and increasing the volatility (Anthony *et al.*, 2011).

From the result observed in the analysis of chrysophlum albidum oil, 2.97mm square per seconds was obtained which is found to be within the range stipulated by ASTM D6751 of 1.9-6.0 millimetre square per seconds at 40℃. The result is in accordance with reports of Tint and Mya 2009; Hossain *et al.,* 2010 and Xiaohu *et al.,* 2009 who obtained values of 4.15,1 2.7 and 4.51 mm square/seconds at 40℃ respectively for kinematic viscosity. Better performance cannot be underrated after the transesterification must have been done. Piyanuch and Sasiwimol, 2009 reported kinematic viscosity at 4.307, 4.549 and 3.057 mm/sec at 40℃ for three different biodiesel blends. For one of the main purposes of transesterification reaction is to lessen the viscosity in order to accomplish properties that are more appropriate for its function as fuel.

Ash content

This measures the amount of metals obtained in the fuel. Oyeleke, 2012 defined the ash biological mineral as inorganic residue that remains after the organic matter has being burnt. Although, it was not defined as total inorganic matter present in the original food, because there might have been chemical interactions between the constituents that makes up the food material.

Ash content is for accessing the quality of edible minerals, but it is significant because, it gives an idea about the amount of mineral element present in the sample (Olaoluwa *et al.,* 2012).

Observation of high ash content in ASASO, that is, 0.025% was found to be higher in value against ENISO 6245 maximum value of 0.02%. The high value may be due to increase in the organic mineral elements in the sample which is in correlation with the definition of Oyeleke, *et al.,* 2012, which is stated above. This indicates that there is possibility of breaking down of some organic molecules within the sample during production processes. This is in agreement with previous report by Obiazoba, 1998, when reporting the fermented foods considering nutritional quality of plant foods**.**

Acid Value

This is a measure of acidic substance in the oil. It is used as a guide in the quality control of monitoring oil degradation during storage. Acid value is one of the properties needed to establish the suitability of oil for biodiesel production. It specifies the amount of free fatty acid existing in biodiesel. Any acid value found to be lower than 0.5mgKOH/g is an ideal amount in fuel for vehicle. It was found that high acid value has a strong solvency influence on rubber seals and hoses in the engine, thereby causing untimely failure and in turn leaves deposits that can clog filter and lead to fuel pressure drop, that is; as level of acid in the engine increases, deposits are generated in the injection systems causing fuel pumping problem. They also attack the joint and metal parts of the injection system. Formation of acid value can occur when traces of water cause hydrolysis of the esters into alcohol and acid (Bonaid *et al.*, 2009). As anticipated the acid number increases with increase in peroxides, because ester first oxidize to form peroxides that include complex reaction which splits into more reactive aldehydes and oxidizes to form acid. (Hossain *et al.*, 2010).

0.15mg KOH/g is reported here of ASASO, and this is found within the limit of 0.80 mgKOH/g specified by ASTM D6751 and in line with the value reported by Hossain *et al.*, 2010, of 0.14 and 0.18 mg KOH/g reported by Xiaohu *et al.*, 2009.

Gross heat of combustion (ΔH^0C)

Gross heat of combustion is the energy released in form of heat when a compound undergoes a complete combustion with oxygen under standard conditions (en.wikipedia.org). For diesel it is 45.0KJ/g. The heat of combustion of raw Chrysophyllum albidum seed oil is 251.19KJ/g.

Table 1: Physico-chemical Properties of raw Chrysophyllum albidum seed oil.

Parameter	Standard	Units	EN14214 Biodiesel	ASTM D6751 Biodiesel	ASASO Oil
Sulphur	EN ISO 14596	ppm	10 max		7.8
Density	ENISO 12185	g/cm^3	0.86 – 0.90		0.89 g/cm^3
Kinematic viscosity, 40^oC	EN ISO3104 D 445	Cst	3.5 – 5.0	1.9 – 6.0 mm^2/sec	2.97
Flash Point	ASTM D-93	^0C		100-107	167
Pour Point	ASTM D-97	^0C		-15 to 10	2.14
Cloud Point	ASTM D-2500	^0C		-3 to 12	2.17
Ash Content	EN ISO 6245	%	0.02 max		0.03
Heat of Combustion	ASTM D-40	KJ/g			251.19
Acid Number		mgKOH/g		0.80 max	0.15
Specific gravity @ 15 ^0C	ASTM D-1298	g/ml		0.87 – 0.9	0,85
Cetane Number	ASTM D-613			48 – 60	40.1
Free glycerol	ASTM D-6751	%		0.2 % max	0.015
Total glycerol	ASTM D-6751	%		0.22 % max	0.02

Iodine value

Iodine values indicates the unsaturation tendency of fats and oils; it measures the presence of C=C bonds which makes the oil prone to oxidation. European committee for standardization (EN 14214) stressed that iodine value should be less than 120g I_2/100g sample for suitability of any seed oil as feed-stock for biodiesel production. Thus with an iodine value of 67.60g I_2/100g sample Chrysophyllum albidum seed oil can be a possible candidate for biodiesel feedstock. The value obtained here is in accordance with 100.1g I_2/100g of Tint and Myo, 2009 (Jatropha oil), 125.6g I_2/100g, Salisu *et al.*, 2012 (Ceirapentandra Linn) and 103 and 116 I_2/100g Mohammed and Hamza 2008, for both white and red Sesamum indicum L. seed oils.

Pour point

Pour point is the temperature at which oil cease to flow; this occurs most especially when there is change of state i.e. when there is change from liquid to solid state. The value obtained for raw Chrysophyllum albidum seed oil after the analysis is 2.14% and this is found to be within the limit set by ASTM D 97 of range -15 to 10^{0}C (Tint and Mya, 2009) and -15-16^{0}C (Edger *et. al.*, 2005) trans-esterification process and the oil is subject to another analysis, which will still found favorable when considering above the value obtained.

Flash point

Flash point is the lowest temperature under closely specified conditions at which combustible material will give off sufficient vapor to form a flammable mixture with air in standardized vessel. American Society for Testing and Material (ASTM D6751) give a range of 100-120 ^{0}C for biodiesel while European Norm (EN 22719) gives a minimum of 120 ^{0}C for biodiesel specification. Considering the value obtained for raw Chrysophyllum albidum seed oil for its suitability for biodiesel production, a value of 167^{0}C obtained was found within the range specified by the two specified standards.

Cloud point

According to Wikipedia, cloud point is the temperature at which dissolved solids are no longer completely soluble or precipitating as a second phase, giving the fluid a cloudy appearances whereas ASTM procedure for cloud point determination are not applicable to dark crude oil and also do not account for potential sub-cooling of the wax with reference to (Kruka *et. al.,* 1995). From the analysis done to determine the cloud point of raw Chrysophyllum albidum seed oil; a value of 3.40 ^{0}C was obtained. This is found to be within the range of -3-12^{0}C specified by ASTM D2500 of D6751 for biodiesel production (Edgar *et. al.*, 2005).

Specific gravity at 15^{0}C

This measures the ratio of density of a substance to the density (mass of the same unit volume) of a reference substance. It varies with temperature and pressure, specific gravity of Chrysophyllum albidum seed oil is 0.85g/mL, this is in agreement with specification of ASTM D1298 for biodiesel according to Tint and Mya, 2009. Mohammed and Hamza, 2008 who obtained 0.915 and 0.923g/mL for both white and red Sesamum indicum L. seed oil; Salisu *et. al.*, 2012, 0.91g/cm^3 for Ceiva Pentandra Linn oil and 0.886 and 0.896g/mL of Audu *et. al.*, 2013 for both chrysophyllum albidum and Lufta cylindrical seed oil and several values obtained for jatropha curcas by Parthiba *et. al.*, 2011.

Cetane number (CN)

Cetane is colourless hydrocarbon that ignites easily under compression, it was given a base reading of 100 that is used as standard measure of performance of compression ignition fuel, such as diesel and biodiesel (http://alternativefuels.about.com). Cetane number is therefore referred to as combustion quality of diesel fuel. It represents the time delay between the start of the injection process and the point where the fuel ignites. (http://www.chemistry.about.com) whereas Jaichandar and Annamalai,2011 reported various values of cetane number of different vegetable oils, gave a range of 35-50 and affirmed that the values are similar or close to of diesel fuel. But Edgar *et al.*, 2005 and Tint and Mya, 2009 ranges of 48-60 and 48-65 respectively according to ASTM D613. Generally, esters derived from fatty acids have a greater cetane number than diesel of 40-55.

Chrysophyllum albidum seed oil having cetane number of 40.1 is found to be in the range sesame safflower, peanut, cottonseed and Crambe oils with values of 40.2, 41.3, 41.8 and 44.6 respectively.

The free glycerol and total glycerol

The effect of free glycerol can be realized at corrosion of non –iron metal point, soaking of cellulosic filters, sediments formation in mobile plants and tendency of forming varnishes which in turn will cause blockage of filters and injector cooking. Free, total even combined glycerol are found to lower the trans-esterification reaction and it can be decreased after trans-esterification reaction if proper adequate washing is done.

0.015% and 0.02% are the value obtained for both free and total glycerol respectively in raw ASASO. This is in line with the value 0.58% and 8.27% obtained by Tint and Myo, 2009 for both free and total glycerol which was found to be on higher side and were found to be within the limit specified by ASTM D6751 of 0.2 and 0.22% max for both free and combined glycerol respectively.

CONCLUTION

Although, nearly all properties measured in this research work were established to be fairly feasible for biodiesel production, but low content oil of obtained from the seed makes it inadequate for industrial production. Optimization can be carried out to achieve maximum conversion of oil (references to good and qualitative physicochemical analysis) to ester when sufficient time have been allowed to ensure complete conversion of triglyceride. The overall analytical results obtained revealed that, it is effective to produce quality biodiesel from Chrysophyllum albidum seed oil considering its cost effectiveness being less than expensive feedstock and forestalling environmental pollution causing by the seeds.

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