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Effect of Process Variables on the Production of Biodiesel by the Non-Catalytic Supercritical Trans-Esterification of Sheanut Oil

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Abstract

The production and characterization of biodiesel from sheanut oil was carried out using the supercritical trans-esterification process and the effects of process variables such as temperature, feed blending ratio, and time were investigated. The physico-chemical properties of the biodiesel produced were compared to the conventional light atmospheric gas oil (LAGO) which is commercially used as fuel in diesel engines. Result showed that supercritical trans-esterification method could be used for the production of biodiesel from sheanut oil. Optimum yield of 89.5% was observed at the feed blending ratio of 20:1, a temperature of 240°C, and a reaction time of 240 minutes

Keywords: Biodiesel, Supercritical, Transesterification, Sheanut, Temperature, Reaction time, Blending Ratio

Introduction

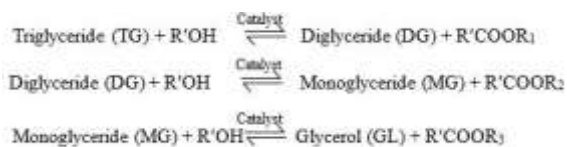
The main objectives of the present ecological policy includes reduction of fossil energy consumption of the economy and development of branches related to renewable energy sources which include energy provided from biomass, wind, geo-thermal, water and solar system. Renewable energy sources enable both improvement of environmental protection and are an important element of sustainable development. According to the experts programs of the world committee of energy council, it is predicted that in 2070, the contribution of renewable energy to the total world energy balance will be about 60%. This will result mainly from depletion of the territorial energy sources and general availability of unconventional energy. The resultant effect will be the improvement in environmental protection due to reduction of the emissions of carbon, sulphur and nitrogen oxides and waste minimization (Wladyslaw et al., 2007). The production and use of biodiesel have increased significantly in many countries around the world using numerous feedstock sources. Unfortunately, it is in nascent status in many African countries. Over the past decade, consumption of transport fuels in Sub-Saharan Africa has increased at a rate of about 7% per year in line with increased economic activity (Mulugetta, 2008). This has had a great economic impact on about thirty-five crude oil-importing countries in Africa. With large landmass

for farming and abundance of edible and non-edible oils, some of which grow in the wild, Sub-Saharan Africa is a region with a fairly high potential for biodiesel production. One of the promising sources of biodiesel production is the shea oil from shea tree (*Vitellariaparadoxa*), a deciduous tree which grows naturally in the wild, especially in the savannah belt of Africa. Shea tree produces the shea nut which is processed into shea butter. Shea butter is classified as oleaginous product. Shea nut contains 37-55% of fats; it is composed mainly of two fatty acids, stearic and oleic, which together account for 85-90% of the total fatty acids (Maranzet al., 2004). Soft shea butter has high oleic content (Badifu, 2000). The physico-chemical properties of shea butter are comparable with the properties of groundnut oil which has been used in biodiesel production (Ogbonnaya and Adgidzi, 2008). Also, it has been observed that the properties of biodiesel are largely dependent on the physicochemical properties of the feedstock. According to some researchers (Ramadhas et al., (2005), Sahoo et al., (2007)), the methyl esters of saturated fatty acids have a higher cloud point, cetane number and better biodiesel stability. Shea butter consists of 41.1% of saturated fatty acids, comprising palmitic and stearic acids. To ensure the quality of biodiesel as an alternative fuel, it has been proposed to limit the unsaturated fatty acid in biodiesel species,

especially the content of higher unsaturated fatty acid such as linolenic acid which, on heating, polymerises and leads to gum formation (Lang et al., 2001). Unlike many biodiesel fuels, the biodiesel from shea butter may less likely undergo oxidation since shea butter contains natural antioxidants such as tocopherols and some phenolic compounds (Maranz, 2003). Hence, biodiesel from shea butter will make a good alternative fuel. Several approaches have been used in the production of biodiesel from vegetable oils and animal fats.

Transesterification of fat and oil

Transesterification of vegetable oil is a reaction by which a triglyceride reacts with an alcohol in the presence of a strong acid or base producing a mixture of fatty acid alkyl esters and glycerol. The suitable alcohols used are methanol, ethanol, propanol and butanol but methanol is the most commonly used because of its low cost and its good physico-chemical properties. The stoichiometric ratio for transesterification reaction requires three moles of alcohol and one mole of triglycerides to yield three moles of fatty acid esters and one mole of glycerol. The overall process is a sequence of three consecutive and reversible reactions in which the triglycerides are converted to diglycerides, which in turn are converted to monoglycerides and then to glycerol. Each step of the processes produces a molecule of an alcohol ester of fatty acid. Several aspects including the types of catalyst (alkaline or acid), alcohol-vegetable oil molar ratio, temperature, purity of the reactants mainly water content and free fatty acid content have an influence on the course of transesterification.



Three consecutive and reversible reactions for ester production. R_1 , R_2 , R_3 and R' represent alkyl group since naturally occurring fats and oils are mixtures of different triglycerides, they may contain a number of different fatty acids.

The Supercritical Processes

Performing the esterification in supercritical conditions has been studied initially as a method to solve the problem of miscibility of oil and methanol that hinders the kinetics in normal conditions. Since the critical coordinates of methanol are $T_c = 239^\circ \text{C}$

and $P_c = 80 \text{ bar}$, raising the temperature and pressures at sufficiently high values is necessary. The reaction involves no catalyst. The advantage of avoiding a catalyst is obvious.

The addition of co-solvent in combination with supercritical conditions seems to be an efficient means to reduce significantly the operating temperature. Due to the absence of the catalyst the process flow sheet employing the supercritical technology should be much simpler, but in exchange the manufacture of hardware is much more demanding. Effective energy integration is also necessary. An alternative catalyst-free method for transesterification uses supercritical methanol at high temperatures and pressures in a continuous process. In the supercritical state, the oil and methanol are in a single phase, the reaction occurs spontaneously and rapidly. Free fatty acid is converted to methyl ester instead of soap, so as wide variety of feed stocks can be used. Also the catalyst removal step is **eliminated** (Vera, C.R., S.A.D' Ippolito; C.L., Pieck, J.M. Parera (2005-08-14). [PLEASE THIS IS NOT LISTED IN THE REFERENCE SECTION] A system for continuous transesterification of vegetable oil using supercritical methanol was developed using a tube reactor. Increasing the proportion of methanol, reaction pressure and reaction temperature can enhance the production yield effectively. However, side reactions of unsaturated fatty acid methyl esters (FAME) occur when the reaction temperature is over 300°C , which lead to much loss of material. There is also a critical value of residence time at high reaction temperature, and the production yield will decrease if the residence time surpasses this value. **The optimal reaction condition under constant reaction temperature process is 40:1 of the molar ratio of alcohol to oil, 25min of residence time, 35MPa and 310°C . However, the maximum production yield can only be 77% in the optimal reaction condition of constant reaction temperature process because of the loss caused by the side reactions of unsaturated FAME at high reaction temperature.** [IS THERE ANY REFERENCE THAT CAN BE CITED FOR THIS ASCERTION?] To solve this problem, **we proposed** [WHO ARE THOSE WHO PROPOSED THIS?] a new technology; gradual heating that can effectively reduce the loss caused by the side reactions of unsaturated FAME at high reaction temperature. With the new reaction technology, the methyl esters yield can be more than 96% (Huayang He, Tao Wang and Shenlin Zhu (2006).

According to Kusdiana and Saka (2001), Biodiesel fuel from rapeseed oil was prepared in supercritical methanol to study the effect of reaction temperature,

reaction pressure and molar ratio of methanol to triglycerides in the methyl esters formation. The results revealing that the supercritical treatment of

350°C, 30MPa and 240sec with molar ratio of 42 in methanol is the best condition for transesterification of rapeseed oil to biodiesel fuel

Table 2.11. Comparisons between the common method and supercritical methanol method for biodiesel fuel production from rapeseed oil

	Common method	Super critical methanol
Reaction time	1-6h	240sec
Reaction condition	0.1MPa,30-65°C	35MPa,350°C
Catalyst	Acid or alkali	None
Free Fatty acids	Saponified products	Methyl esters
Yield	97% (normal)	98.5%(higher)
Removal for	Methanol, Catalyst	Methanol
Purification	And saponified products	
Process	Complicated	Simple

(Source: Adapted from Kusdiana and Saka (2001))

Materials and method

Preparation of sheanut

Sheanut (*Butyrospermumparkii*) seeds were bought from central market Ikpata, Kwara State. The seeds were dehulled to remove the outer seed coat. The dehulled seeds were then spread and dried in the sun for three consecutive days to reduce the moisture content present in the seed. Also, raw Shea butter used in ethyl ester production was bought from New Market, Enugu State.

The dried seeds were size reduced with mechanical grinder. The particle size was in the range of 1-2mm. This was done in order to create large surface area for contact with the solvent for maximum extraction.

Extraction of sheanut oil

a) Mechanical Press

200g of ground Shea nut seeds were placed inside a porous sack. The sample was placed inside the mechanical press where it was pressed to squeeze out oil. The oil was collected using a beaker. The mass of oil was obtained as the difference between the mass of beaker with oil and the mass of empty beaker. Standard analysis was also conducted with expressed oil.

b) Solvent Extraction

Extraction of the oil was carried out using soxhlet extractor. 100ml of n-hexane was poured into a distillation flask and placed on a heating mantle. 20g of the sample was placed in a porous thimble and inserted in the soxhlet apparatus which was coupled to a reflux condenser to return the condensate to the round bottom flask as the heating proceeded. The heating mantle was set at a temperature of 70°C. The whole set up was observed for 2 hours and later dismantled and the full miscella (oil + solvent) was

distilled to obtain the oil. The mass of the cooled oil was obtained as difference between the weight of the evaporating dish with oil and weight of empty evaporating dish. Standard analyses were then carried out on the oil extracted to determine the chemical properties.

Determination of specific gravity

An empty 10ml specific gravity bottle as weighed. The bottle was filled with water and weighed. The bottle was emptied, dried and the oil was poured into it. The new weight was emptied, dried and the oil was poured into it. The weight was taken and recorded.

Calculation:

$$\text{Specific Gravity} = \frac{\text{Weight of oil}}{\text{Weight of equal volume of water}} \dots\dots\dots 1.0$$

Determination of acid value

2g of oil sample was weighed and transferred into 250ml conical flask. 50ml of neutral solvent was added and 3 drops of phenolphthalein indicator was added. This was immediately titrated using standard (0.1N) sodium hydroxide (NaOH). The end point was the appearance of a permanent pink colour.

$$\text{Calculation: Acid Value} = \frac{56.1NV}{W} \dots\dots\dots 2.0$$

Where;

N is the normality of NaOH

V is the volume of NaOH used in ml

W is the weight of the oil sample used in g

Mass of oil sample

Determination of moisture content

The Petri dish was dried and weighed as W_1 , 5g of the oil was put into the Petri dish and the new weight W_2 was recorded. The dish with the oil was transferred into an electric oven at 105°C and allowed to dry. For 1 hour. The weight of the dish was weighed as W_3

$$\text{Calculation: \% Moisture Content} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \dots\dots\dots 3.0$$

Where;

Mass of empty dish, M_1

Mass of dish + water, M_2

Mass of dish + oil sample, M_3

Determination of ash content

The Petri dish was weighed and then 5g of oil was poured into it and also weighed. Then the dish containing oil was placed into a furnace and heated at 600°C for 2 hours. The dish was allowed to cool for 1 hour. Thereafter that the content was weighed.

Weight of dish = W_1

Weight of oil = W_2

Weight of oil + dish = W_3

Heat at 600°C

Weight of dish + Ash = W_4

$$\text{ASH CONTENT} = \frac{(W_3 - W_1) - (W_4 - W_1)}{W_2} \dots\dots\dots 4.0$$

Determination of saponification value

0.5g of oil sample was weighed and transferred into a clean dry round bottom flask. 50ml of 0.5m alcoholic NaOH was weighed out and added to the oil. To the round bottom flask, was a fixed condenser and the contents were refluxed for about 30mins. Porous bites were added to the flask to ensure uniform heating. The content of the flask were cooled and titrated with standard HCl. A blank test was run in the same way without oil sample. This was then titrated against 0.5m HCl.

$$\text{Saponification value} = \frac{56.1N(V_2 - V_1)}{W} \dots\dots\dots 5.0$$

Where;

N = Normality of HCl

V_2 = Titre value for the blank run

V_1 = Titre value for the oil

W = Weight of oil used

Determination of iodine value

1g of oil sample was weighed into a small glass tube and placed in a 300ml conical flask with ground glass stopper. 15ml of CCl_4 was added, the mixture was agitated until dissolved. 25ml of Wigg's solution was

added to the mixture, agitated and stopper. The solution as allowed to stand in the dark for 30mins at room temperature. 100ml of distilled water and 20ml of sodium iodine solution were added. The whole content of the conical flask were titrated with 0.1N (standard) sodium thiosulphate using starch solution as the indicator. Colour change from ox-blood to pink and then to colorless. The blank was run in the same way without the oil sample.

$$\text{Calculation: Iodine value} = \frac{12.69N(V_2 - V_1)}{W} \dots\dots\dots 6.0$$

Where;

Weight of oil sample used (g) = w

Normality of the Sodium thiosulphate Sodium = N

Volume of $\text{Na}_2\text{S}_2\text{O}_3$ used, titre value of oil = V_1

Volume of $\text{Na}_2\text{S}_2\text{O}_3$ used in blank = V_2

Normality of $\text{Na}_2\text{S}_2\text{O}_3$, $N = 0.1N$

Determination of cloud point

The kryostat as switched on for 60 minutes before analysis and the bath was filled with Oil. The sample was poured into test tube up to a mark inscribed in the cup and the equipment was switched on. The sample was monitored closely to determine the exact temperature at which the oil will start to solidify. The temperature was recorded as 31°C

Determination of the refractive index

The refractometre was cleaned with distilled water and a drop of oil was dropped at the surface of the refractometre and the reading was obtained as 1.467

Determination of viscosity

The oil was poured into 100ml beaker. The brook viscometer was set to a spindle speed of 100rpm. After 5 seconds, it was immersed into the oil. The temperature was noted using thermometer. The viscometer reading was recorded as 2.6 when it was noted to be steady for 2 – 3 seconds.

Methodology**Effect of temperature on ethyl ester yield**

240g of crude Shea Oil at a molar ratio of 20:1 of ethanol/oil was transferred into the beaker of 250ml. The mixture was stirred vigorously using Electronic stirrer at a speed of 500 r.p.m for 20min. The mixture was transferred into 250ml of round bottom Pyrex flask-batch reactor and heated for 4 hours. The reaction product was allowed to separate into two places by standing for 3 hours in a separatory funnel. The tap of the separatory funnel was opened to remove the dark brown layer, glycerol and the upper amber yellowish layer, ethyl ester was left in the

separatory funnel. The experiment was repeated setting the temperature of the water bath at varying temperature of 230°C, 240°C, 250°C, 260°C, 270°C, 280°C, and 300°C for each experiment. The results were tabulated as in table 4.3.0.

Effect of ethanol/oil molar ratio on ethyl ester yield

240g of crude Shea Oil at a molar ratio of 20:1 of ethanol/oil was transferred into the beaker of 250ml. The mixture was stirred vigorously using Electronic stirrer at a speed of 500 r.p.m for 20min. The mixture was transferred into 250ml of round bottom Pyrex flask-batch reactor and heated for 4 hours. The reaction product was allowed to separate into two places by standing for 3 hours in a separatory funnel. The tap of the separatory funnel was opened to remove the dark brown layer, glycerol and the upper amber yellowish layer, ethyl ester was left in the separatory funnel. The experiment was repeated at varying ethanol/oil molar ratio of 3:1, 5:1,6:1, 8:1,

10:1, 15:1, 20:1, 25:1, and 30:1. The results were tabulated as in table 4.2.0.

Effect of time on ethylester yield

240g of crude Shea Oil at a molar ratio of 20:1 of ethanol/oil was transferred into the beaker of 250ml. The mixture was stirred vigorously using Electronic stirrer at a speed of 500 r.p.m for 20min. The mixture was transferred into 250ml of round bottom Pyrex flask-batch reactor and heated for 4 hours. The reaction product was allowed to separate into two places by standing for 3 hours in a separatory funnel. The tap of the separatory funnel was opened to remove the dark brown layer, glycerol and the upper amber yellowish layer, ethyl ester was left in the separatory funnel. The experiment was repeated at varying time of 60mins, 90mins, 120mins, 150mins, 180mins, 240mins, and 300mins. For each experiment. The results were tabulated as in table 4.4.0

Results

Table 4.1: Comparism of the Physico-chemical Properties of Shea nut oil from mechanical expression and solvent extraction with shea butter

PHYSICO-CHEMICAL PROPERTIES	MECHANICAL EXPRESSION METHOD	SOLVENT EXTRACTION METHOD	SHEA BUTTER
Specific gravity	0.8664	0.882	0.8776
% Moisture content	0.6	19.10	0.5
Acid Value (mgNaOH/g)	36.04	40.50	21.03
Free fatty Acid value	18.02	20.37	10.525
Iodine Value (mg Iodine/100g)	7.12	-	207.57
Saponification value (mg NaOH/g)	196.40	199.72	1.00
Ash Content	1.00	1.00	7.11
Cloud point (°C)	29	27	31
Refractive index	1.466	1.458	1.467
Viscosity (cP)	2.8	2.6	2.6

Table 4.2:Percentage Yield at varied molar ratio of Ethanol/Oil

Molar ratio of ethanol/oil	Percentage Yield
3:1	50
5:1	57.6
6:1	66.67
8:1	71.75
10:1	76.8
15:1	80.7
20:1	89.8
25:1	80.0
30:1	73.9

Reaction conditions: Temperature = 240°C, Reaction Time = 240mins, Stirrer speed = 500r.p.m.

Table 4.3: Percentage Yield at varied Temperature

Temperature (°C)	% Yield
220	79.70
230	80.43
240	82.86
250	53.40
260	57.30
270	59.00
280	59.70
300	64.50

Reaction conditions: Temperature = 240°C, Molar ratio = 20:1, Reaction Time = 240mins, Stirrer speed = 500r.p.m.

Table 4.4: Percentage Yield at varied Time

Time (mins)	% Yield
60	47.80
90	49.40
120	52.10
150	56.60
180	56.90
240	80.00
300	76.10

Reaction conditions: Temperature = 240°C, Molar ratio = 20:1,
Stirrer speed = 500r.p.m

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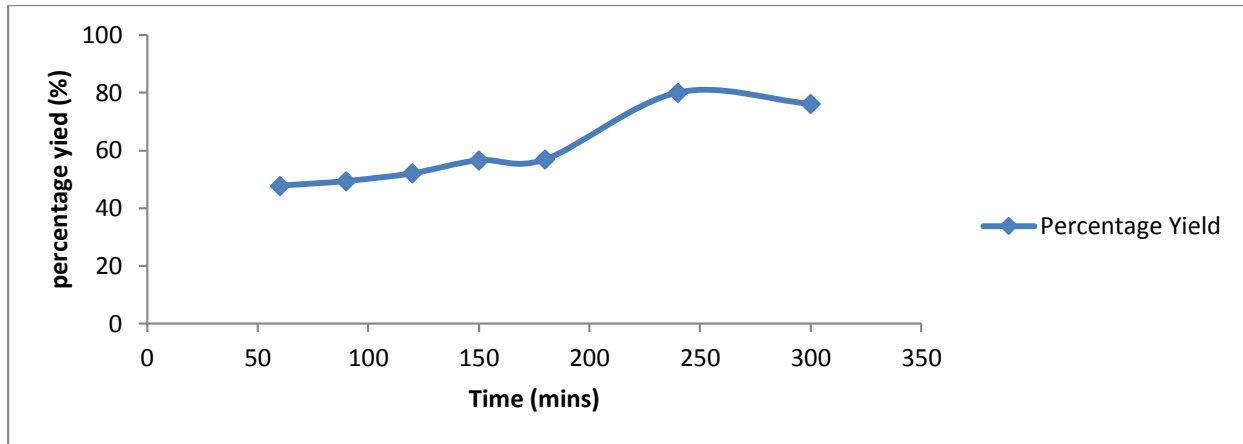


Fig. 3.0 Effect of reaction time on the yield of bioethanol

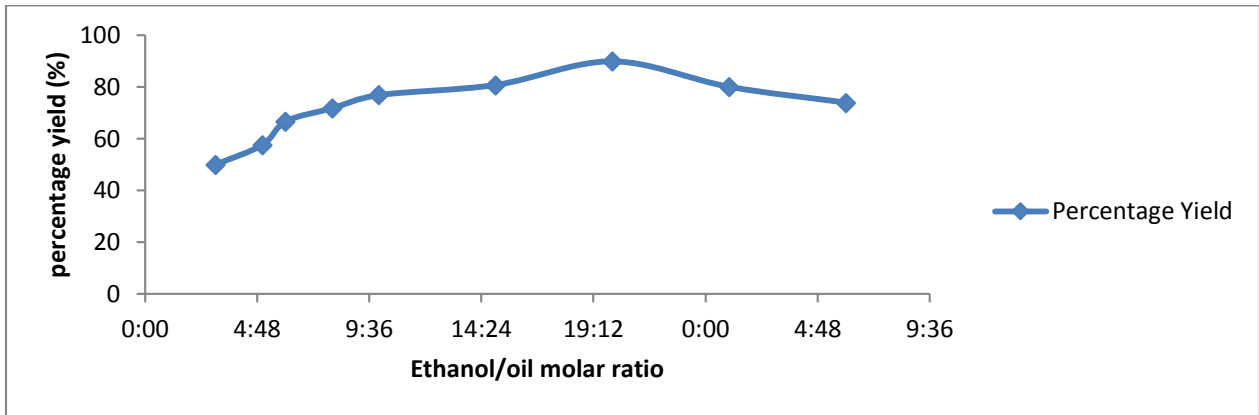


Fig. 1.0 Effect of feed blending ratio on the yield of biodiesel

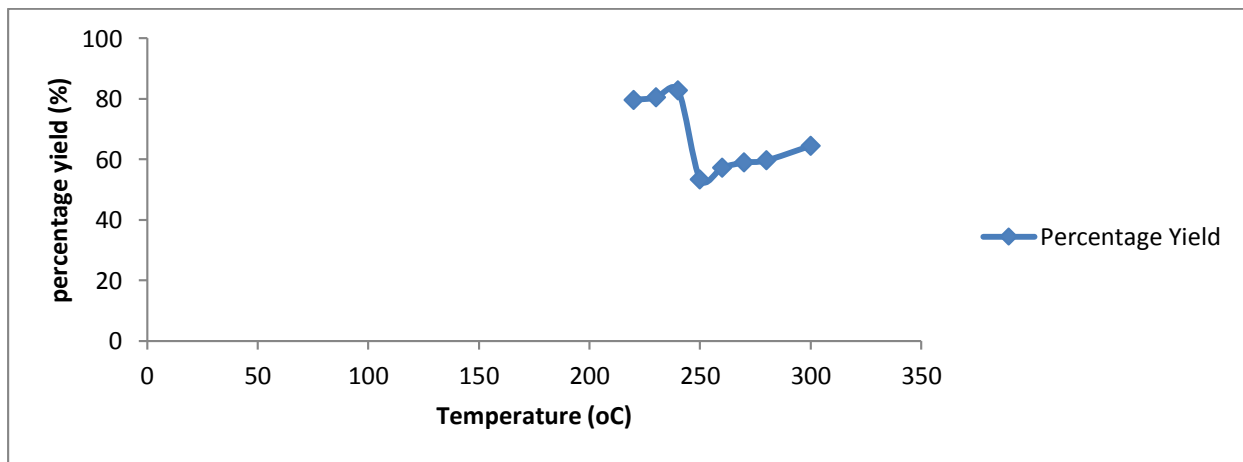


Fig. 2.0 Effect of temperature on the yield of biodiesel

Table 4.5: Physiochemical Properties of Ethyl ester in relation to Biodiesel Specification

Parameter	biodiesel from shea nut	Unit	ASTM Specifications (D6751)	EN 14214	N0.2 DIESEL
S.G. 60/60°F	0.870	-	0.80 – 0.90	1.9-6.00	820-860
Iodine Value	39.2	(I ₂ g100g ⁻¹)		120max	–
Ash Content	0.12	-			
Acid Value	0.35	mgNaOH/g		0.5max	–
Cloud point	32	°C		–	-19 to -8
Kinematic viscosity @40°C	5.92	mm ² s ⁻¹	1.9 – 6.0	3.5-5.0	2.5-3.5
Pour point	28	°C			-34 to -10
Cetane number	53.20	-	47min	51min	49-55
Flash point	173.5	°C	130min	>101	>55
Copper strip Corrosion	3a	-	3max		

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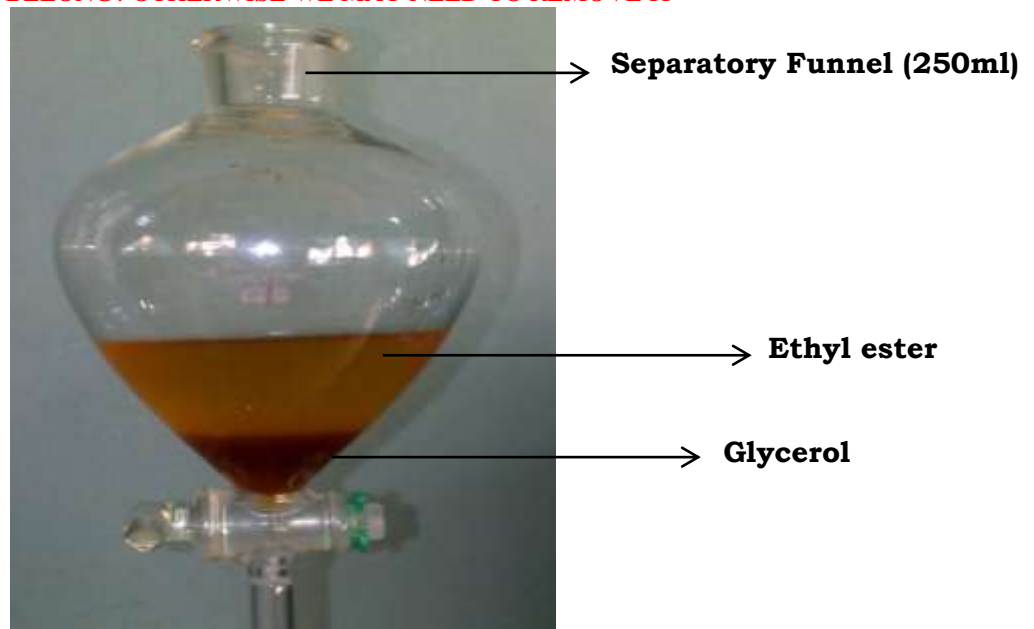


Fig. 4.0 PRODUCT MIXTURE IN A SEPARATORY FUNNEL

Discussion

The effect of reaction temperature on yield of ester was shown in figure 4.3. In carrying out this test, all other parameter remaining constant and temperature varied from 220°C and 300°C to determine the effect of heating on yield of ester. The result showed that yield increased with increase in reaction temperature. Heating at higher temperature also reduced the viscosity of the oil. This can attributed to the higher temperature which helped in faster settlement of glycerol. (Gupta et al, 2007). Increase in temperature increased the yield of ester and decreased the reaction

time.(Darnoko and Cheryan, 2000).With respect to effect if reaction time on yield of ester, it was observed that the yield increased as the reaction proceeded although the reaction required longer time to reach completion. The time was varied between 60mins to 300mins.It could be seen from the curve of yield against time of figure 3.0, that the optimum time was 240mins.Several investigators had shown that the conversion rate of biodiesel increased with increase in reaction time. (Freedman et al, 2004).

The fuel properties of biodiesels are characterized by their viscosity, density, cetane number, Cloud and

pour points, ash content, acid value, copper strip corrosion etc. However, the ethyl ester produced under optimum condition of 20:1 ethanol/oil ratio with non-catalytic supercritical method at temperature of 240°C in 240mins was analyzed in respect of some significant properties of diesel fuel as mentioned above. Most of these properties conformed to Biodiesel Standard. ASTM D-6751, specifies the quality for pure biodiesel (B100) and the international standard, EN 14214, and describes the minimum requirements for biodiesel produced from feed stocks. These standards are shown in table 4.0 for biodiesel except in the cloud point and pour point where ethyl ester showed higher values which may have resulted from the high Stearic acid content of Shea oil and also the temperature at which a liquid fatty material becomes cloudy due to the formation of crystals and solidification of saturated components

Conclusion

Alternative fuels for diesel engines has become increasingly important due to diminishing petroleum reserves and the growing environmental concerns which has made renewable fuels an exceptionally attractive alternative as a fuel for the future. Biodiesel is derived from a varied range of edible and inedible vegetable oil, animal fats, used frying oil and waste cooking oil.

Ethyl ester was produced from Supercritical transesterification of Shea oil. The parameters affecting the ethyl ester formation such as reaction temperature, feed blending ratio and time, were investigated to determine the best strategy for producing biodiesel from shea oil. It was observed that the ethyl ester yield increases with increase in feed blending ratio, with the ratio of 20:1 giving the best result. Also, heating at higher temperatures helped to reduce the viscosity of the oil. From the properties of ethyl ester produced, it can be concluded that Sheanut oil can be utilized as source of fuel for use in diesel engines if the main barrier to employing it that is, its high cloud and pour points which affect its flow is overcome. The properties of the fuel tested were within the ASTM and EN14214 standards and were found to be very close to those of light atmospheric gas oil which is used as conventional diesel

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