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Effect of Temperature on the Transesterification of Animal Fat

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Abstract

Biodiesel is a potential replacement for petroleum diesel. It is renewable, environmentally friendly and can be sourced from vegetable oil and animal fat oil. In this study, the effect of temperature on the transesterification of animal fat (beef tallow) was investigated. The transesterification reaction of animal fat oil was conducted using sodium hydroxide as the catalyst. In the production process, 1.0g of sodium hydroxide and 100g of animal fat with an 8:1 molar ratio of alcohol to oil was used to produce biodiesel at varied temperatures of 60°C - 80°C. The biodiesel produced was characterized using Gas Chromatography-Mass Spectrometry (GC-MS). From the analysis, the high yield and conversion of the triglycerides to fatty acid methyl ester (FAME) was found to be at an optimum temperature of 60°C. The high yield and conversion of FAME obtained from the analysis indicated that animal fat is a good feedstock for biodiesel production. The values of viscosity, density and flash point of the biodiesel produced at optimum temperature were compared with ASTM D6751 and EN14214 standards, and the values obtained were found to be within the required limits.

1. Introduction

The increase in industrialization and population over the years has led to an increase in the demand for energy. Petroleum-based fuel which serves as the major source of energy has been proven to be inefficient with its increasing cost and unfavourable environmental consequences [1]. Research has shown that renewable energy sources such as biodiesel is a promising alternative to petroleum fuel, and it is environmentally friendly [2]. Biodiesel has been shown to have advantages over petroleum diesel. These advantages include high cetane number, high oxygen content, zero sulphur content, lower emission of carbon monoxide, particulate matter, polycyclic aromatic hydrocarbons, and aldehydes [3]. Biodiesel can be produced from feedstock such as vegetable oil or animal fat with alcohol in the presence of a catalyst. The feedstock accounts for about 70-80% of the cost of biodiesel production [4], and as such it is important to consider feedstocks that are economically viable. The motivation for using animal fat as a feedstock for this experiment is because of its abundance in abattoirs (meat processing facilities) in Benin City, Edo State, and its inappreciable usage. In most cases, the animal fat is left to waste. Animal fat contains low free fatty acids and unlike other feedstock, such as jatropha oil or waste cooking oil where two-steps esterification is required. The animal fat can easily be converted to biodiesel with one step transesterification thereby reducing the cost of production [5]. Biodiesel produced from animal fats compared with biodiesel from vegetable origin has the advantage of a higher cetane number, the most significant indicator of diesel combustion behaviour [6].

The transesterification of the triglycerides present in the animal fat can be processed using alcohols such as methanol or ethanol in the presence of acidic (sulphuric acid) or basic (sodium hydroxide) or enzymatic catalyst. The transesterification reaction produces fatty acid with glycerol as a byproduct. The reaction is a reversible one and the rate of conversion of fatty acid is greatly affected by several factors such as type of catalyst used, reaction time, the molar ratio of alcohol, temperature etc.
In this research work, the effect of temperature on the transesterification of animal fat with methanol using sodium hydroxide as catalyst was studied.

2.0 Methodology

2.1 Materials

The animal fat (beef tallow) used was purchased from an abattoir in Benin City. The chemicals and reagents used include; methanol (99.8%), benzene (99%), absolute ethanol (99.8%), sodium hydroxide (99%), and hydrochloric acid (90%). All chemicals used in this study were of analytical grade.

2.2 Determination of Free Fatty Acid (FFA) of Animal Fat

Titration was conducted on the oil sample and the blank. For the oil sample, a mixture of 10ml of ethanol and 10ml of benzene was added to 100g of animal fat oil (AFO). The mixture was stirred and titrated against 0.1mole of KOH with four (4) drops of phenolphthalein until a pale and permanent pink colour was observed. The same procedure was carried out for the blank without the presence of the AFO. The values obtained for the blank and the sample were inputted into the formula below to obtain the acid value [7]. The molar mass of KOH used in the calculation equals 56.1g/mol.

$$AV = \frac{(V_S - V_B) \times M \times 56.1}{W} \tag{1}$$

The Free Fatty Acid (FFA) content was calculated using the expression below:

$$FFA = \frac{AV}{2} \tag{2}$$

2.3 Determination of Saponification Value of Animal Fat

The indicator method as specified by ISO 3657 was used to determine the saponification value of the animal fat. 10ml of 0.1M KOH was added to 100g of AFO in a conical flask, the mixture was constantly stirred and allowed to boil for 60 minutes. A reflux condenser was placed on the flask containing the mixture. 2.0 drops of phenolphthalein solution were added to the warm solution and then titrated with 0.5M HCL to the endpoint until the pink colour disappeared. The same procedure was done for the blank [7]. The saponification value is expressed as:

$$SV = \frac{5.61 \times (B - A) \times N}{W_{Oil}} \tag{3}$$

2.4 Production of Biodiesel from Animal Fat

The transesterification reaction was carried out using AFO, methanol, and sodium hydroxide. A 1000ml round bottomed flask served as the reactor. It was placed on a constant temperature magnetic stirrer. A temperature sensor probe was fitted in the reactor to stabilize the temperature. The AFO was preheated using a hot plate. 100g of the preheated AFO was placed in the reactor and the methoxide mixture containing 76ml methanol and 1.0g of sodium hydroxide (8:1 mole ratio) was also added. A reaction time of 60 minutes and a rotational speed of about 600-1000 rpm was employed in the reaction process. The first temperature of the reaction was stabilized to 60^0C with the help of the magnetic stirrer. At the end of the reaction time, the product was poured into a 1000ml separating funnel and was allowed to separate into two layers of biodiesel and glycerol. The product was allowed to settle down for 24 hours and the denser glycerol was withdrawn through the drain at the bottom of the funnel. The other reactions were carried at temperatures of 70^0C and 80^0C.

2.5 Separation and Purification of Biodiesel

Once the reacted mixture was allowed to settle down, the biodiesel formed was separated from the glycerol and poured into a separate beaker where it was washed with warm water to filter out impurities such as glycerol, excess methanol, catalyst, and unreacted fat. The washing process was repeated until a colourless wash water was observed. The biodiesel obtained from the washing process was heated to a temperature of 100^oC and the water was left to evaporate out of the mixture leaving a clear amber yellow coloured biodiesel [8].

2.6 Characterization of Biodiesel

The density of the biodiesel was measured using the density bottle, and the kinematic viscosity was measured with an Ostwald viscometer. The flash point was determined using Pensky Martens closed cup tester.

2.7 Characterization of Biodiesel using GC-MS

The biodiesel produced at optimum conditions was analyzed to determine the fatty acid composition using Gas Chromatography-Mass Spectrometry (GC-MS). The result obtained showed that the transesterification reaction had broken down the large fatty acids with many double bonds into smaller fatty acids with a few double bonds and single bonds.

3.0 Results and Discussion

3.1 Physical Properties of Animal Fat

The physical properties of the animal fat are shown in Table 1. The acid value determines FFA in the oil. Studies have shown that high FFA reduces the effectiveness of the catalyst and react with the base catalyst to form soap [9]. The FFA composition obtained from the analysis was within the standard value. The recommended amount of FFA for AFO should not exceed 0.5% [10]. In the case where it is above the recommended amount, the AFO should be esterified before being used for biodiesel production [11], [12].

The standard saponification value is within the value of 193 - 202mgKOH/g [13]. From Table 1, the saponification value obtained from this experiment is higher than the standard value. This implies that animal fat is a good feedstock for soap making [12].

Table 1: Physical Properties of Animal Fat

Property	**Value**
Acid value (mgKOH/g)	0.7076
FFA%	0.3538
Saponification value (mgKOH/g)	247.046

3.2 Fatty Acid Composition of Animal Fat

Figure 1 shows the GC-MS chromatogram of animal fat. The chromatogram in Figure 1 shows the various peaks of fatty acid present in the fat. The GC-MS results identified Palmitic Acid (47.453%), Linoleic acid (39.109%) and Stearic Acid (13.439%) as the dominant fatty acid present in the animal fat. As shown in Table 2, the results obtained from this analysis were in disparity with that obtained by [9] and **[14]**. This disparity could be due to the differences in breed and the diet of the cattle from which the animal fat was obtained.

Table 2: Fatty Acid Composition of Animal Fat

Fatty Acid	**GC-MS Result**	**Literature Value [14]**	**Literature Value [9]**
Palmitic Acid	47.45%	25.33%	27.30%
Linoleic Acid	39.11%	0.75%	10.40%
Stearic Acid	13.44%	34.70%	11.70%

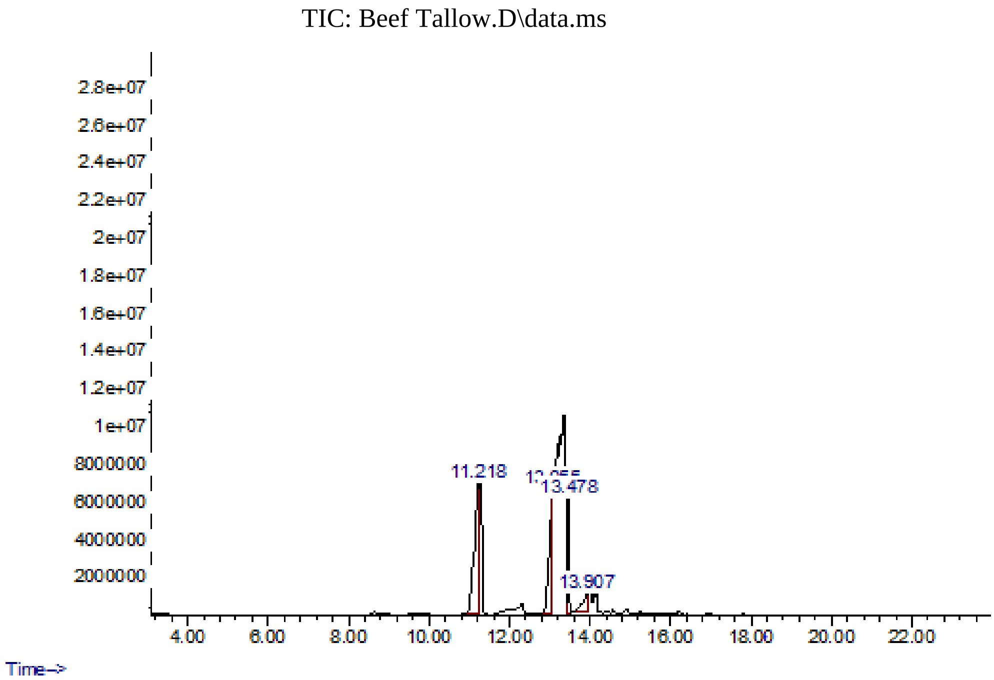

Figure 1: Chromatogram of Animal fat

3.3 Effect of Temperature on Biodiesel Yield

The temperature for the transesterification reaction was optimized by varying temperatures from 60^{o}C to 80^{o}C and keeping other parameters like catalyst concentration, molar ratio, and reaction time constant. From the analysis, the volume yield of biodiesel produced at 60^{o}C, 70^{o}C and 80^{o}C are 89.91%, 85.32% and 83.78% respectively. From Table 3, the GC-MS analysis showed that the highest fatty acid methyl ester (FAME) composition of biodiesel was achieved at 60^{o}C. At 60^{o}C, the bond between the fats experienced a weak Van der Waal's force of attraction and was attracted to the strong nucleophilic substances that were present in the reaction mixture. Simultaneously, methanol in its liquid-vapour phase combined with the fats thus favouring the forward reaction of the transesterification reaction which led to high conversion. Methanol has a boiling point that is around 64^{o}C and at temperatures above this, the methanol evaporates gradually out of the reactor, and this led to the reduction in the biodiesel yield for temperatures of 70^{o}C and 80^{o}C [2].

The result from the GC-MS analysis also showed that the transesterification reaction had broken the large FAME with many double bonds into smaller FAME with a few double bonds and into fatty acids with no double bond at all. The high conversion of FAME obtained from the transesterification reaction showed that animal fat is a good feedstock for biodiesel production [15].

Figure 2, Figure 3, and Figure 4 show the various peaks on the chromatogram obtained from the GC-MS analysis of biodiesel samples produced at temperatures of 60^{o}C, 70^{o}C and 80^{o}C respectively. The various peaks on the chromatogram represent specific compounds that have been identified from the library match software (NIST 14 mass spectral database) as shown in the appendix section.

Table 3: FAME Composition of Biodiesel at Different Temperatures.

FAME	Percentage Present (%)		
	60°C	**70°C**	**80°C**
Stearic Acid	41.322	24.226	0.931
Oleic Acid	26.94	52.117	35.364
Palmitic Acid	25.701	20.233	59.698
Myristic Acid	5.847	2.364	2.166
Total	99.81	98.94	98.159

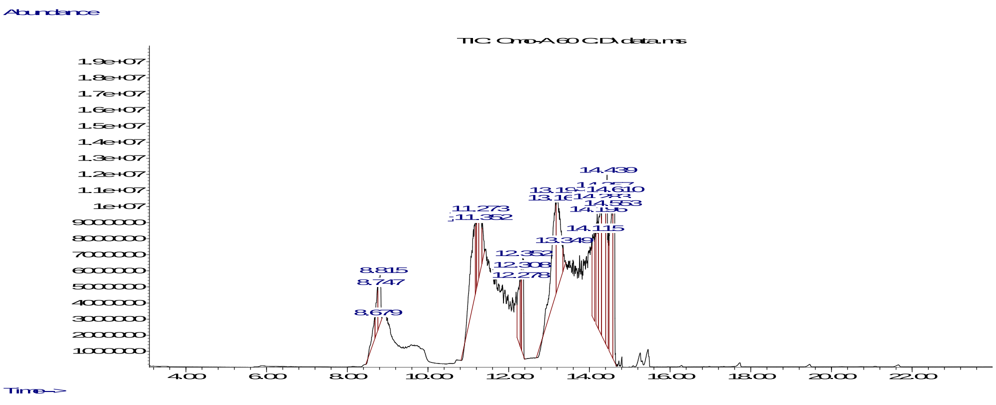

Figure 2: Chromatogram of Biodiesel produced at 60°C

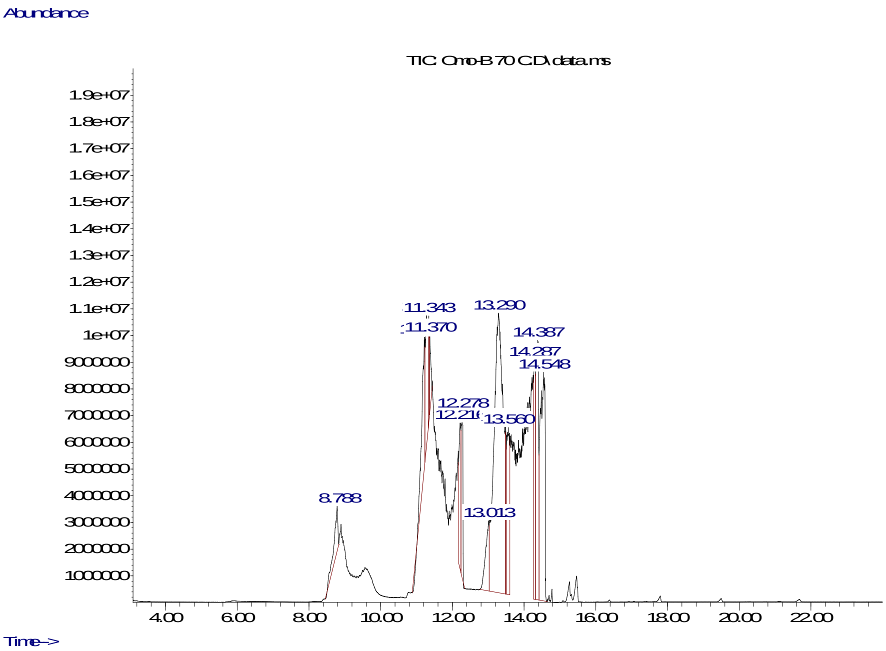

Figure 3: Chromatogram of Biodiesel produced at 70°C

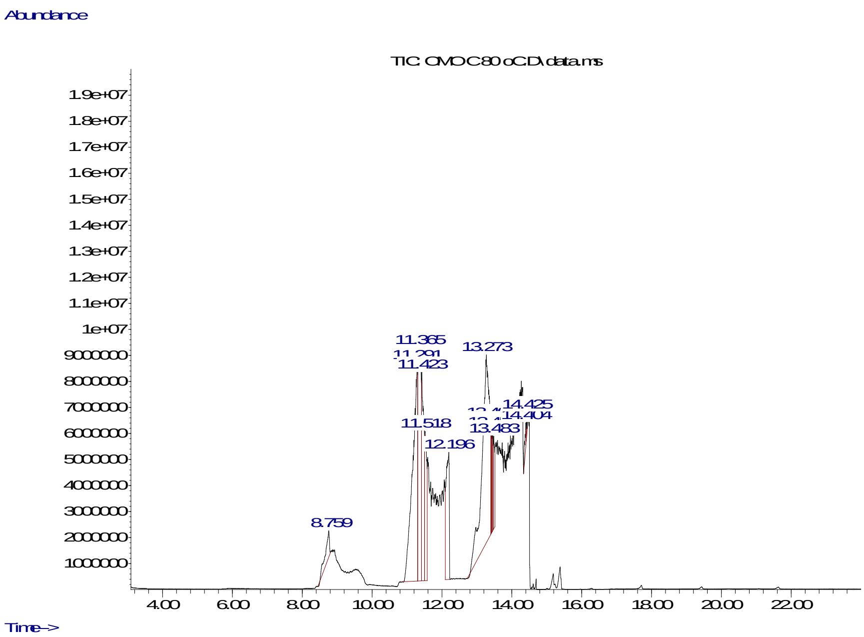

Figure 4: Chromatogram of Biodiesel produced at 80°C

3.4 Physical Properties of Biodiesel Produced at 60ºC

Table 3 shows the properties of biodiesel produced at 60ºC. Density indicates the delay between the injection and combustion of a fuel in a diesel engine (ignition quality) and the energy per unit mass (specific energy). It influences the efficiency of the fuel atomization for airless combustion systems. An increase in temperature results in a decrease in viscosity. From Table 4, the results obtained for density and viscosity are within the range specified by international standard (ASTM D6751 and EN 14214) as stated in [16], [17].

Kinematic viscosity is a measure of the resistance of oil to flow. High kinematic viscosity leads to poor atomization of the fuel spray and less accurate operation of the fuel injectors [18]. From Table 4, the viscosity of the biodiesel falls within the standard specification indicating that the biodiesel will perform well in the internal combustion engine [16].

The flash point of a fuel is the temperature at which it will ignite when exposed to flame or spark. Flash point is an indication of unreacted alcohol remaining in the biodiesel fuel. It also indicates the precaution that must be taken during the handling, transport, and storage of the fuel [14], [19]. From Table 4, the biodiesel produced at a temperature of 60^{o}C had a flash point of 140 ^{o}C. From the result obtained, the flash point of the biodiesel produced falls within the standard specification as shown in Table 4.

Table 4: Properties of AFO Based Biodiesels and Specification and International Standard.

Properties	**AFO Based Biodiesel**	**ASTM D6751**	**EN 14214**
Density (g/cm^3)	0.8423	-	0.86 – 0.9
Kinematic Viscosity (mm^2/s)	4.682	1.9 – 6.0	3.5 – 5.0
Flash Point (^{o}C)	140	130 minimum	101 minimum

4.0 Conclusion

The results of this work revealed that animal fat transesterified at temperatures of 60^{o}C, 70^{o}C and 80^{o}C gave 89.91%, 85.32% and 83.78% yield of biodiesel respectively. The optimum yield of biodiesel was obtained at 60^{o}C. The density, kinematic viscosity, and flash point of the biodiesel produced are within the specifications of international standards. Therefore, oil extracted from animal fat can be used as feedstock for biodiesel production.

Conflict of Interest

There is no conflict of interest associated with this work.

Nomenclature

A	Volume of ethanol potassium hydroxide used in titration with the oil
AFO	Animal Fat Oil
ASTM	American Society for Testing and Material International
AV	Acid Value
B	Volume of standard ethanol potassium hydroxide used in blank titration
EN	European standard
FAME	Fatty Acid Methyl Esther
FFA	Free fatty acid
GC-MS	Gas Chromatography/Mass Spectrophotometer
HCl	Hydrochloric acid
ISO	International standard organization
KOH	Potassium hydroxide
M	Concentration (moles/dm3) of KOH solution
N	Normality of standard acid
V_B	Volume (ml) of KOH solution for titration of blank

V_S Volume (ml) of KOH solution for titration of the sample
W Weight of AFO sample
Woil Weight of AFO used

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Appendix

NIST 14 Mass Spectral Database

Hexadecanoic acid, methyl ester 130813 000112-39-0 97
Hexadecanoic acid, methyl ester 130820 000112-39-0 97
Pentadecanoic acid, 14-methyl-, methyl ester 130841 005129-60-2 97
10,13-Octadecadienoic acid, methyl ester 153881 056554-62-2 99

9,12-Octadecadienoic acid (Z,Z)-, methyl ester	153889	000112-63-0	98
7,10-Octadecadienoic acid, methyl ester	153871	056554-24-6	97
Methyl stearate	157884	000112-61-8	98
Methyl stearate	157885	000112-61-8	97
Methyl stearate	157879	000112-61-8	96
Methyl 9-eicosenoate	182550	1000336-50-5	98
cis-Methyl 11-eicosenoate	182558	002390-09-2	98
cis-11-Eicosenoic acid, methyl ester	182571	1000333-63-8	96
Methyl 18-methylnonadecanoate	184595	1000352-20-6	98
Eicosanoic acid, methyl ester	184599	001120-28-1	94
Methyl 8-methyl-decanoate	65001	1000336-49-1	93
Docosanoic acid, methyl ester	209113	000929-77-1	99
Docosanoic acid, methyl ester	209118	000929-77-1	98
Methyl 20-methyl-heneicosanoate	209123	1000336-47-4	98
Heneicosanoic acid, methyl ester	197601	006064-90-0	95
Tetracosanoic acid, methyl ester	228678	002442-49-1	91
Tetracosanoic acid, methyl ester	228680	002442-49-1	90