ALKALI-CATALYZED BEEF SUET TRANSESTERIFICATION KINETIC REACTION WITH METHYL ALCOHOL

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ABSTRACT

Beef suet obtained from an abattoir was characterized and transesterified with caustic soda and methanol. The feedstock and product were characterized with the aid of a GCMS and revealed the fatty acids as hexadecanoic acid (47.453%) and 9, 12-Octadecadienoic acid (32.455%). The highest conversion of about 94.5% was obtained at 0.6 wt% of catalyst, 60°C and 60 minutes. The minimum amount of activation energy $E_a$from the reacting molecules was estimated as $4051.49\, J/mol$with a rate constant at optimum yield$k = 0.00086 min^{-1}$ as with a frequency k_0 of $2.97 min^{-1}$ at333K. Transesterification of beef suet is very fast and favours low catalyst loading.

Keywords: Activation-energy; Beef suet; Biodiesel; Kinetics; Transesterification
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INTRODUCTION

Due to the overdependence on fossil fuel as the major source of energy, there has been a decline in petroleum reserves since it is non-renewable. With the increase in population and energy-consuming processes and equipment, global energy demand is steadily increasing from year to year and as such, there is need for a shift from petroleum to renewable sources of energy which will meet the required energy need [1].The use of fossil fuels has also generated some negative effects on the environment such as SO_2 and CO emissions which needs to be eradicated. Biodiesel has shown to be free from environmental pollution thus an excellent alternative diesel fuel as it is a renewable and biodegradable fuel which can be produced from any oil-bearing seed or animal fat [2]. It contains a long-chain fatty acid methyl ester made by chemically reacting alcohol with lipids in the presence of a suitable catalyst [3].

The biodiesel industry has some significant difficulties. In particular, feedstock selection can have a profound impact on the production process but also on food prices when food crops such as palm oil are diverted to energy [3]. The need for biodiesel production from non-edible oils is being studied to limit the competition between food and fuel. Biodiesel can be produced in different ways but the most common method used is the transesterification procedure [4]. The animal fat undergoes a transesterification reaction with methanol in the presence of a catalyst to yield fatty acid alkyl esters, otherwise known as biodiesel. The transesterification reaction can be carried out under various reaction conditions such as molar ratios, temperatures, reaction time and catalyst concentration. It can also be carried out in the presence of different catalysts which could be homogeneous or heterogeneous, acid or alkali, organic or inorganic and so on [4]. It is important to find the optimum reaction conditions and the best catalyst for the transesterification of particular feedstock.

The most popular animal sources used as a feedstock are beef tallow, chicken fat, lamb fat, lard, yellow grease, hemp oil and the greasy by-product from omega-3 fatty acid production [5]. The major components of fats and oils are triglycerides which are made of different types of fatty acids with different chemical and physical properties [6]. The properties of the fatty acids play a major role in influencing the properties of the biodiesel obtained [6]. Tallow consists of fat rendered from lamb and cattle but mainly from cattle. The fatty acids composition of tallow mainly comprises of oleic acid $(26 - 40\%)$, stearic acid $6 - 40\%$, palmitic acid $(17 - 37\%)$, myristic acid $(1 - 8\%)$, linoleic acid $(0 - 5\%)$ and other branched-chain acids [7]. The fatty acids are mostly saturated and monounsaturated fatty acids and only a minimum amount of essential fatty acids can be found in the tallow. Tallow has a high cholesterol level and lacks in natural antioxidants [5, 7]

There are two basic groups of catalysts, homogeneous or heterogeneous catalyst. This group is classified as alkali and acid-based catalysts. Alkaline catalysts have been reported to be more effective in transesterification reaction [8, 9]. Hossain *et al.* [10] noted that the triglycerides should have free fatty acid concentration of less than 1.0%, and all materials should be substantially anhydrous. More caustic soda (NaOH) will be required to neutralize the free fatty acids (FFA) if the concentration was greater than 1.0% [11]. Water molecules present also caused soap formation, which consumed the catalyst and reduced catalyst efficiency.

Beef suet was used as a raw material of transesterification to make biodiesel in this study, because the high price of beef tallow as it edible by many people and thus influenced the market interest. Beef suet is the fat within the intestine and it is often wasted in abattoir. This study was aimed at investigating of the effects of industrial-grade caustic soda as catalyst because it is the most economical catalyst used by biodiesel production industries and its efficacy in the transesterification of beef suet has been presented in this study.

The use of beef suet as a feed stock for biodiesel production had been performed by many authors. Many authors had produced biodiesel from animal fat through one-step base-catalyzed transesterification, whereas others used a two-step catalyzed transesterification such as acid and base catalyzed transesterification [6, 7, 12]. Limited literatures were reported about the production of biodiesel from beef suet through a one-step alkali catalyzed transesterification using industrial-grade caustic soda. Therefore, in this study, fat which was extracted from Adamawa Banyo Breed was converted into biodiesel using base-catalyzed transesterification.

MATERIALS AND METHODS

Materials

Beef suet was collected from an abattoir in Benin City, Edo state from freshly butchered Adamawa Banyo Breed cattle in a 25 litre capacity bucket. Methanol used in the transesterification reaction was pure and analytical grade produced by JHD, Guangdong, China. Sodium hydroxide was purchased as caustic soda in a local industrial chemical shop.

Materials preparation

The suet was taken to the laboratory for separation of pure fat from other unwanted materials such as beef parts and bone pieces. This separation was done by gently heating the fat, beef, and bones in a pot until it melted thereby filtering out pure fat. Commercial grade caustic soda and methanol used in this study were used as purchased without any modification.

Experimental set-up

A 2-neck flask of 1000 ml capacity was placed on a constant temperature magnetic stirrer and fitted with a stir bar to achieve a homogeneous mixture at a constant mixing speed of about 380 rpm. The flask was connected to a Braham condenser, such that any methanol vapour given off by the heated reaction mixture will be condensed and return to the flask. The reaction temperature was made constant via a thermocouple inserted into the flask.

Production of biodiesel

Physical and chemical properties of the feedstock were conducted using standard methods described by ASTM. Biodiesel was produced by transesterification reaction [13]. 100 grams of the fatwas weighed into the flask and heated to reaction temperature. Caustic soda (NaOH) was added to the methanol and stirred until completely dissolved. This was poured into the heated fat in the reactor [14]. The reaction was left to proceed for 60 minutes with constant stirring 380 rpm. The procedure was repeated multiple times with different amounts of catalyst between 0.2 to 2.0 wt% and temperatures between 30 to 80℃, reaction time between 10 to 90 minutes and methanol to oil mole ratio between 3:1 to 9:1 [15]. The product which was a mixture of biodiesel and glycerin obtained from the transesterification reaction was poured into a separating funnel and allowed to separate into layers after 24 hours. The glycerol which settles at the bottom was separated first. The biodiesel yield was estimated after purification according to Equation 1.

$$Biodiesel\ Yield(\%) = \frac{Weight_{Biodiesel}}{Weight_{Oil}} \times 100 \text{ (Equation 1)}$$

Where the weight of triglyceride was taken as the initial mass of oil used in the reaction. The weight of biodiesel produced from each experimental run was calculated after washing the biodiesel formed from the transesterification reaction.

Crude biodiesel purification

Water washing was adopted by mixing with warm water at about 50℃ which was twice as the volume of the biodiesel and swirled gently before it was poured into a separating funnel. After some time, the water which had dissolved the impurities in the biodiesel settled to the bottom of the separating funnel and was drained off [16]. This procedure was repeated multiple times until the wash water had a clear appearance, signifying that there were no more impurities in the biodiesel.

Characterization of biodiesel produced from beef suet

Biodiesel was characterized using standard methods. Density, specific gravity, kinematic viscosity, Carbon residue, Oxidation stability, Flashpoint, Calorific value and pour point were determined using the corresponding ASTM standard procedure. Fatty acid compositions were determined using a gas chromatograph equipped with a mass spectrometry detector (GC-MS) Single Quadruple GC/MS Instruments 5977B GC/MSD.

The biodiesel sample was subjected to GC/MS analysis to characterize the various fatty acid compositions. The gas chromatographic Model: 7890A (GC) analysis was performed on an Agilent Technologies interfaced with Mass Selective Detector Model: 5977B (MSD). The electron ionization was at a 70V with anion source temperature at 250ºC. Highly pure helium gas of about 99.9% purity was used as carrier gas, while HP-5 (30mm X 0.25mm X 0.320µm) was used as the stationary phase. The oven temperature was at 140ºC held for 5 minute at 8ºC/minutes and ramped to 180ºC at the rate of 4ºC/minutes and was increased to 210ºC at the rate of 20ºC/min and finally increased to 250ºC holding for 7 minutes and about

1µ/L of sample was auto injected for ionization and mass spectrometric detection.

RESULTS AND DISCUSSION

Physiochemical properties of beef suet used in the production of biodiesel

Table 1 shows some of the physical-chemical properties of beef suet used in this study. The acid value was 0.6473 $mgKOH/g$ while the FFA content was estimated to be 0.3237%. This value of FFA is satisfactorily low because per cent FFA for transesterification should not be greater than 1. After all, higher FFA per cent concentration has been reported to cause the oil to hydrolyze during transesterification reaction [12, 17, 18]. The saponification value implies that the fat contains short-chain fatty acids [19].

Table1: Properties of animal fat (beef suet)

Property	Value
Moisture content	0.2137%
Acid Value	0.6473 mgKOH/g
FFA Content	0.3237%
Saponification Value	217.046 mgKOH/g
Density @ 40°C	0.8913 g/cm^3
Specific Gravity @ 40°C	0.8983
Iodine value	56.04 $I_2g/100g$
Peroxide value	9.45 m_{eq}/g

Results of gas chromatographic analysis (Table 2) showed the fatty acids contained as Hexadecanoic acid (47.453%), 9, 12-Octadecadienoic acid (32.455%), methyl stearate (13.439%), and 10, 13-Octadecadienoic acid (6.654%). Though with little variations in concentration which could be attributed to breed and the environmental effects [20]. Table 2 shows the fatty acids present in the animal fat as analyzed by the GC/MS.

Table 2: Fatty acid composition of beef suet

Fatty Acid	Common Name	Percentage (%)
Hexadecanoic acid	Palmitic Acid	47.453
(9Z)-Octadec-9-enoic acid	Oleic Acid	28.462
Octadecanoic acid	Stearic Acid	13.439
10,13-Octadecadienoic acid	Linolenic acid	6.654
9,12-Octadecadienoic acid	Linoleic acid	3.455
Tetradecanoate	Myristic Acid	0.501

Effects of catalyst concentration on biodiesel yield

From Figure 1, the maximum yield of 94.5% was obtained at a catalyst of 0.6 wt%. Higher catalyst amounts were observed not to have effectively catalyzed the reaction thereby resulting in the formation of soap by reacting with excess caustic soda [9]. Lower catalyst concentration generally was observed to be more effective in the transesterification of the animal fat due to the high reactivity of the catalyst [18].

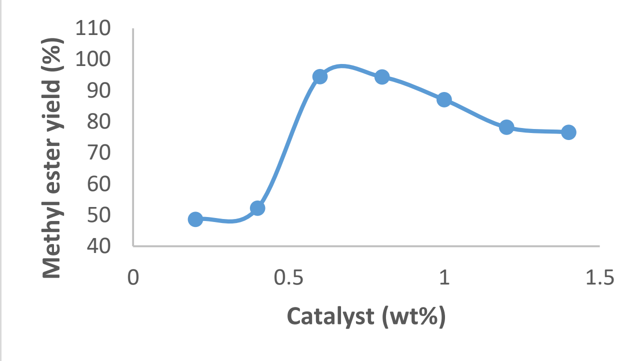

Figure 1: Effects of catalyst amount on Methyl ester yield

Effect of reaction temperature on biodiesel yield

The results shown in Figure 2 revealed that conversion increased correspondingly with an increase in temperature to a peak obtained at 60°C with a yield of 94.81% with catalyst amount 1.0 wt%, methanol-to-oil mole ratio 6:1, and reaction time of 60 minutes. When the reaction temperature increased to 60°C, there was also an increase in the reaction rate due to higher energy input and reduced mass transfer resistance [21]. Increasing the temperature beyond 60°C resulted in a decrease in the production because any reaction occurring closed to or above methanol boiling point resulted in its intense continuous vaporization [22]. Hence it remained in the gas phase in the reflux condenser, causing a reduction in the amount of methanol present in the reaction media [21].

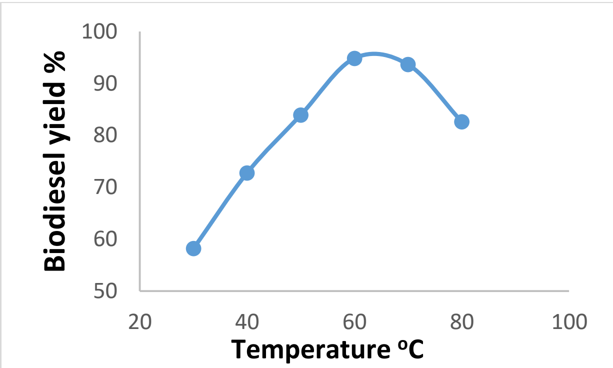

Figure 2: Effect of reaction temperature on biodiesel yield

Reaction time on biodiesel yield

The time of reaction of methanol and triglyceride has proved to be a major parameter in the transesterification reaction animal fat using NaOH catalyst [14]. Figure 3 shows an increase in the yield with time from 10 to 100 minutes with a catalyst amount of 0.6 wt% concerning fat and 6:1 mole ration of methanol to oil. The maximum yield of 96.39% was obtained in 60 minutes. In the initial stages of the transesterification reaction, production of biodiesel was rapid, and the rate diminished into a steady-state at 60 minutes and beyond.

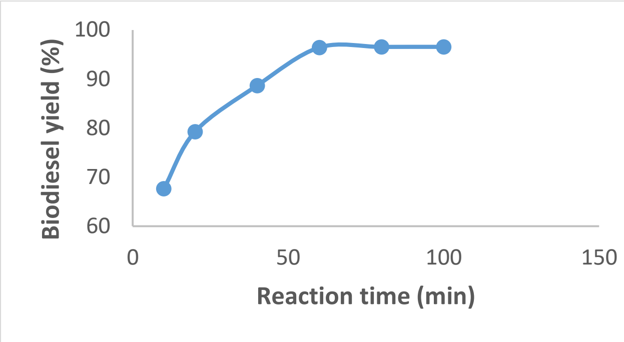

Figure 3: Effect of reaction time on biodiesel at 60ºC reaction temperature

Properties of biodiesel using ASTM standard

The properties of biodiesel after purification by water washing adequately conform to the properties stated by the American Society for Testing and Materials Standard (ASTM) for (B100) [23]. The summarized results as shown in Table 3 shows that all of the measured values were within the standard limit.

Table 3: Physical and chemical properties of animal fat biodiesel

Property (nit)	Value	ASTM Standard
FA (%)	0.247	0.25 %, max
Density @ 30°C (g/cm^3)	0.8214	0.820 – 0.845g/cm^3
Specific gravity @ 30°c	0.8077	NA
Cloud point (°C)	-4.7	NA
Kinematic viscosity @ 30°C (mm^2/s)	3.919	1.9 - 6.0 mm^2 /s
Flash point (°C)	118	93°C min
Oxidation stability (hours)	3.23	3 hrs min
Calorific value (MJ/kg)	40.41	NA

*NA=Not Available

The density of biodiesel was observed to decrease as the catalyst amount was increased from 0.2 wt% to 1.0 wt% and beyond. The lowest density was obtained with 1.0 wt% of catalyst (0.8414 g/ml) as well as the specific gravity of 0.8480. The viscosity. The kinematic viscosity was 3.919 mm^2/s as shown in Table 3. According to Raja *et al.* [25], biodiesel kinematic viscosity at 40°C is from 1.9 to 6 mm^2/s. Figure 4 presented the chromatogram of the GC-MS analysis showing the specific compounds. As shown in Table 3, the major fatty acid methyl ester present in the biodiesel sample was cis-13-Octadecenoic acid methyl ester (38.342%) and Hexadecanoic acid methyl ester (23.443%).

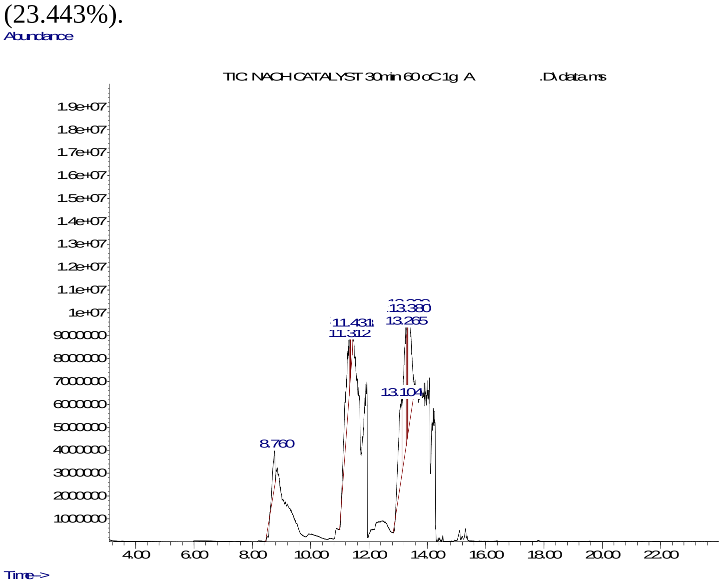

Figure 4: Chromatogram of biodiesel produced from beef suet

Table 4: Fatty acid methyl ester composition of biodiesel produced beef suet

Fatty Acid	Common Name	Percentage Present (%)
Methyl Tetradecanoate	Myristic acid	4.300
Hexadecanoic acid, methyl ester	Palmitic acid	23.443
9-Octadecenoic acid, methyl ester	Oleic acid	16.830
cis-13-Octadecenoic acid, methyl ester	Oleic acid	38.341
9-Octadecenoic acid (Z)-, methyl ester	Oleic acid	17.085

The reaction kinetics

From rate constants at the various reaction temperature, an increase in reaction temperature also increases the conversion of triglyceride to fatty acid methyl ester (FAME). The overall rate equation (k) can be found from first-order reversible reaction (Equation 2) [26, 27].

$kt = -In(1 - Y_a)$ Equation 2

Let Y_a be taken as the yield of triglyceride. The values of rate constant k collected between 303K and 333K were tested in 1st order reaction kinetics equation (Equation 2).

Table 5: Values for estimation of activation energy

Reaction Temp. (K)	1/T (K^{-1})	Y_A	Rate Constant k (min^{-1})	*-ln k*
303	0.00330	0.5816	0.00697	4.9657
308	0.00325	0.6409	0.00599	5.1185
313	0.00319	0.7294	0.00451	5.4015
318	0.00314	0.7657	0.00391	5.5455
323	0.00310	0.8387	0.00269	5.9188
328	0.00305	0.8526	0.00246	6.0089
333	0.00300	0.9487	0.00086	7.0644

The results of the experiment conducted at variable temperatures are presented in Table 5 as estimated using the Arrhenius equation (Equation 3). The experimental data fitted well for 1st order kinetics and were linearized using Arrhenius equation (Equation 4) [28]:

$k = k_0 * \exp\{-\frac{E_a}{RT}\}$ Equation 3

Finding the natural log of both sides of equation 3 produces equation 4

$Ink = \{-\frac{E_a}{RT}\} + Ink_0$ Equation 4

Where E_a is the activation energy, R is the gas constant (8.314 J/mol. K),T is an absolute temperature, and k_0 is the frequency factor.

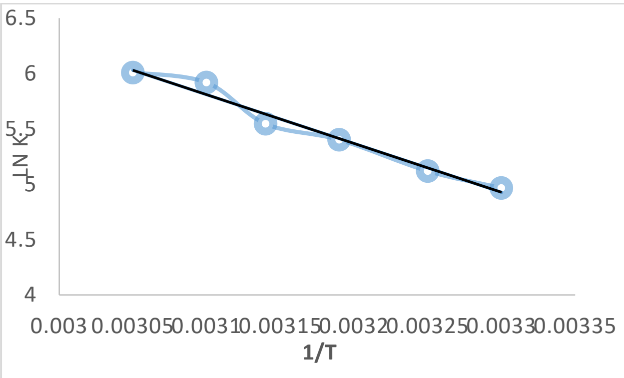

Figure 5: plot of ln*k* versus 1/T

From the plot of ln*k* versus 1/T (Figure **5**) $-\frac{E_a}{R} = Slope\ of\ the\ graph = -4412.4$. Therefore, Activation energy E_a = 4051.49 J/mol, and, k_0 = 2.97 (Table 6)

Table 6: Summary of constants for kinetics study

Constant	Value
E_a	4051.49 J/mol
k (at 60°C)	0.00086 ***min^{-1}***
k_0	2.97 ***min^{-1}***
R	8.314 J/mol.K

From the Arrhenius plot, the Arrhenius constant and activation energy were estimated. Figure 5 reveal that activation energy E_a increases with increasing temperature. This suggest there is strong surface interactions as corresponding frequency factor k_o increase. This is the frequency of collisions and the orientation of a favorable collision probability [29].

Table 6 summarizes the results for all kinetic constants. It is observed that the increase in temperature had a corresponding increase in the activation energy by 4051.49 J/mol. The activation energy is the energy required for the transesterification reaction to proceed. Higher value of E_a is an indication that alkali catalyst provided a high catalytic activity in reducing the energy barrier of the reaction process.

CONCLUSIONS

Beef suet is an excellent feedstock for biodiesel production due to its extremely low free fatty acids concentration. From this study, it has been seen that Beef suet favours low alkaline catalyst concentration for effective transesterification reaction. Biodiesel from beef suet has a very low specific gravity and viscosity, hence a quality fuel for the diesel engine. Fatty acid methyl esters present in beef suet biodiesel are cis-13-Octadecenoio acid methyl ester, Hexadecanoic acid methyl ester, 9-Octadecenoic acid methyl ester, 9-Octadecenoic acid methyl ester and Methyl tetradecanoate. The lack of polyunsaturated hydrocarbon in the biodiesel produced is a good reason for the high oxidative stability of the biodiesel. The overall physicochemical properties meet the requirements set by the American Standard (ASTM) for biodiesel.

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