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Acid-Catalyzed Transesterification Reaction of Beef Tallow For Biodiesel Production By Factor Variation

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ABSTRACT : *Biodiesel is a diesel grade fuel made by transesterification reaction of vegetable oils and animal fats with alcohol. Three variable factors that affect the yield of biodiesel namely, reaction time, reaction temperature and catalyst concentration were studied in this work. The biodiesel was produced via a batch-process acid-catalyzed transesterification reaction of beef tallow with methanol. Optimal conditions for the reaction were established in a three factor two-level (2^3) central composite design with the biodiesel pre-treatment yield as the response surface. The results show that the mean yield of biodiesel was 92.04% with a standard deviation of 5.16. An optimal biodiesel yield of 96.30% occurred at 0.5% HCl catalyst concentration and at constant conditions of 1.5h reaction time, 60°C reaction temperature and 6:1 methanol: tallow volume ratio. Gas chromatographic analysis of the beef tallow identified palmitic, stearic and oleic acids in it while the fatty acid methyl esters in the biodiesel product were oleate and linoleate. Catalysis was the most significant factor in the transesterification process.*

KEYWORDS: *Biodiesel; Transesterification; Tallow; Factor Variation.*

I. INTRODUCTION

Biodiesel is a mixture of mono-alkyl esters of triglyceride that can be used in compression ignition engines and as home heating oil. Current research efforts are being directed towards the use of biomass for bio-fuel production principally because the fuels are renewable, bio-degradable, non-toxic and eco-friendly[1-3]. Biodiesel has gained prominence as a suitable alternative to fossil fuel-based diesel due to environmental benefits and its direct use in compression ignition engines with little or no engine modifications.Biodiesel can be made by the process known as transesterification[4, 5]. Transesterification of vegetable oils and animal fats can be carried out by using alkali-, acid- or bio-catalysis but the alkaline-catalyzed process has short reaction time, low cost of production, gives high yield and is often commercially employed[1,6,7]. Problems associated with alkaline-catalyzed transesterification are the competing saponification reaction forming soap that occurs when the product is washed with water to remove the alkaline catalyst[4, 7, 8] and the presence of free fatty acids (FFAs) and water in the triglycerides[7-10]. The FFAs cause saponification while water leads to hydrolysis reaction and eventually reduce the yield of biodiesel[6, 8, 9].

Several feedstock oils and fats are available for the production of biodiesel. They include soybean oil, rapeseed oil, palm oil, palm kernel oil, *jatropha-curcas* oil, sunflower oil, peanut oil, rubber seed oil, waste cooking oil, canola oil and tallow[3, 4, 11-17]. Variable reaction conditions have been identified to affect the transesterification process and consequently the yield of biodiesel. These conditions include reaction temperature, catalyst type and catalyst concentration, alcohol type, alcohol: oil molar or volume ratio, reaction time, the reactor process and downstream operations, etc. [3-7, 18].In this study biodiesel was produced using acid-catalyzed transesterification reaction of beef tallow with methanol. Hydrochloric acid was used as catalyst. The objective of the study was to investigate the relationship between reaction time, reaction temperature and catalyst concentration at constant alcohol: oil volume ratio and to also determine their optimal conditions in the transesterification process and the associated maximum biodiesel yield. The Box-Wilson central composite design (CCD) and response surface methodology (RSM)[19-20] were used for the optimization analysis.

II. MATERIALS AND METHODS

Materials: Beef tallow (BT) raw material was obtained from Meat Market in Abakpa Enugu Township. The fat samples were heated to 120^oC to obtain oil-like liquid usually at the start of the production. The fatty acid composition of the oil was determined using the Hewleth Packard 6890 Gas Chromatograph. Analar grade hydrochloric acid, methyl orange indicator and methanol were supplied by May and Baker Ltd, Bagenham, England.

Experimental Design: A 2^3 (three-factor at two levels) factorial experimental design was used to determine the optimum conditions of the three variables studied. The variables were both at high and low levels. One variable, the methanol: oil volume ratio was kept constant at 6:1. Generally, a higher alcohol: oil molar or volume ratio favours high biodiesel yield[2-7, 12-18]. The response is biodiesel yield in the pre-treatment step. Table 1 shows the high, low and centre values of the variables studied.

Experimental Procedure: A preliminary titration was carried out by using the oily tallow sample, methanol and methyl orange indicator solution to find out how much of HCl catalyst of given concentration was needed for complete transesterification reaction. Transesterification reaction proper was conducted without adding indicator solution thus: 3 cm^3 of the oily tallow sample was taken in a 250 cm^3 conical flask equipped with a reflux condenser. A pre-determined volume of HCl catalyst was dissolved in 18 cm^3 of methanol to obtain HCl-methanol mixture, which was then gently added to the tallow in the reactor. The mixture was heated to selected temperature and for a specified time to produce biodiesel and glycerol. At the end of reaction the products were cooled to room temperature and transferred to a separating funnel in which biodiesel was the upper layer and glycerol the lower layer. The two layers were eventually separated by gravity or sedimentation. The volume of biodiesel produced in the pre-treatment step was recorded. Thereafter, the biodiesel was purified by washing with hot distilled water for four times. It was eventually dried for 24 hours in desiccator over anhydrous sodium carbonate. The biodiesel product was analyzed for its fatty acid methyl esters (FAMEs) composition using the Hewleth Packard 6890 Gas Chromatograph. Helium gas was used as the carrier gas. The biodiesel product yield was calculated using Eq-1

$$\text{Yield of biodiesel} = \frac{\text{Volume of product}}{\text{Volume of Beef Tallow sample}} \times 100\% \qquad \text{...Eq-1}$$

Additional experiments were carried out to study the effect of catalyst concentration on biodiesel yield at ethanol: oil volume ratio of 6:1, reaction time of 1.5h and reaction temperature of 60^oC.

Table 1: High, low and centre values of the variables studied

Variable	High value	Centre value	Low value
Residence time (h)	2.5	1.5	1
Reaction temperature (^oC)	70	60	50
Catalyst concentration (%)	2.5	1.5	0.5

III. RESULTS AND DISCUSSION

Free Fatty Acids Composition of Beef tallow

The free fatty acids (FFAs) in the beef tallow sample used for this study are presented in Table 2. The FFAs consists of palmitic, stearic and oleic acids.

The reaction conditions and experimental results of the transesterification process are shown in Table 3. The biodiesel yield was taken as the response variable of the experimental design. Residence time (X_1), reaction temperature (X_2) and HCl catalyst concentration (X_3) were the independent variables selected to be optimized for the transesterification of beef tallow. The results show that the mean yield of biodiesel was 92.04% with a std dev. of 5.16%. There were also two fatty acid methyl esters (FAMEs)- oleate and linoleate identified in the biodiesel product (Table 4).

Table 2: The free fatty acid composition of beef tallow

Fatty acid composition	Percentage (%)
Palmitic $C_{16:0}$	19.41749
Stearic $C_{18:0}$	35.22732
Oleic $C_{18:2}$	45.35519

Table 3: Reaction conditions and experimental results

Run	Manipulated variables						Response
	X_1		X_2		X_3		Y_i
	Residence time(h)	Level[a]	Reaction temperature (oC)	Level[a]	Catalyst concentration (%)	Level[a]	Yield (%)
1.	1	-1	50	-1	0.5	-1	95.00
2.	1	-1	50	-1	2.5	+1	96.00
3.	1	-1	70	+1	0.5	-1	90.00
4.	1	-1	70	+1	2.5	+1	90.00
5.	2.5	+1	50	-1	0.5	-1	96.30
6.	2.5	+1	50	-1	2.5	+1	91.70
7.	2.5	+1	70	+1	0.5	-1	96.00
8.	2.5	+1	70	+1	2.5	+1	80.00
9(C).	1.5	0	60	0	1.5	0	93.33
Mean biodiesel yield							92.04
Std dev.							5.16

a -1: low value, 0: centre value (C), +1: high value.

Table 4: Fatty acid methyl esters (FAMEs) in the biodiesel product

Fatty acid methyl ester	Percentage (%)
Oleic $C_{18:1}$	52.18262
Linoleic $C_{18:2}$	47.81738

Table 5: Effect of catalyst concentration on yield

Catalyst concentration (%)	Biodiesel yield (%)
0	44.50
0.5	96.30
1.0	86.70
1.5	93.33
2.0	93.33
2.5	90.00

Effect of Catalyst Concentration on Yield

Hydrochloric acid was used as catalyst for the transesterification in this work. The effect of the catalyst on the pretreatment yield of biodiesel is presented in Table 5. The results show that an optimal biodiesel yield of 96.30% occurred at 0.5% HCl catalyst concentration (at constant conditions of 1.5h reaction time, 60^{o}C reaction temperature and 6:1 methanol: tallow volume ratio). The data also indicated that the yield was 44.50% when no catalyst was used. Catalysis was the most significant factor in the transesterification process at 60^{o}C reaction, 1.5h reaction time and 6:1 volume ratio of methanol to oil.

IV. CONCLUSION

The study shows that the mean pretreatment yield of biodiesel produced was 92.04% with a standard deviation of 5.16. The Box-Wilson CCD and RSM were effective in determining the relationship among the variables studied. Catalysis was the most significant factor in the transesterification reaction of beef tallow to biodiesel.

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