USE OF RESPONSE SURFACE METHODOLOGY (RSM) IN OPTIMISATION OF BIODIESEL PRODUCTION FROM COW TALLOW

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ABSTRACT
In this study, beef tallow, waste from slaughterhouses was used as a feedstock for the production of biodiesel due to its high yield capacity, availability, and low cost. Sodium hydroxide and methanol were used as catalyst and solvent respectively. Characterization of oil and biodiesel samples were carried out using the Association of analytical chemist (AOAC) and American society of testing and materials (ASTM) respectively. Other characterization methods used are Fourier transform infrared spectroscopy (FTIR) and gas chromatography-mass spectrophotometry (GC-MS) techniques. FTIR was carried out to characterize (identify the constituent elements) of both the feedstocks and their methyl esters. The fatty acid profile of the raw feedstocks and the produced methyl esters were obtained using the gas chromatograph to ascertain the % concentration of the different fatty acids. The physicochemical properties of the oils and biodiesel were also determined and compared with standards. Optimization of the processes was carried out using response surface methodology (RSM) under the platform of Design Expert 7.0.0 which uses statistical regression models. The optimized yield from transesterification of cow tallow using RSM was 83.82%, obtained with optimum operating parameters of Temperature (54.3°C), Time (51.65 mins), Catalyst concentration (1.82 wt%), Methanol/oil ratio (4.08 mol/mol) and stirring speed (302 rpm). This work thus not only confirms cow tallow as a viable feedstock for the production of biodiesel but also proves that a high enough yield of cow tallow biodiesel can be obtained at feasible reaction conditions. This study concluded that the use of response surface methodology proves to be a viable optimization technique albeit with its limitations in the transesterification process.

Keywords: Biodiesel, Optimisation, Cow Tallow, Beef Tallow, RSM

INTRODUCTION
The ever increasing global energy demand and environmental problems created by fossil fuels are creating opportunities to explore alternative energy sources [1]. One such alternative is biodiesel. Biodiesels are renewable, non-toxic, and biodegradable, and therefore, they are a promising alternative to fossil diesel fuel. The study of biofuels as an alternative to fossil fuels has gained popularity over the last decade due to their reliability as a renewable fuel. Biodiesel productions from low-cost feedstocks are needed since biodiesels from food-grade oils are not economically competitive with petroleum-based diesel fuel. Biodiesels are renewable, non-toxic, and biodegradable, and therefore, are a promising alternative to fossil diesel fuel [2]. In addition to

several technical advantages of biodiesel, such as prolonged engine life due to better lubricating properties, a higher flashpoint that makes them safer during storage and transport, biodegradability, and non-toxicity, it is also environmentally friendly due to reduced exhaust emissions [3]. Biodiesels are fuels composed of mono-alkyl esters of long-chain fatty acids. The long and branched-chain triglyceride molecules are transformed into mono-alkyl esters and glycerol through esterification/transesterification reactions. Biodiesel can be prepared from animal fats by the transesterification of triglycerides (TG) with methanol using alkaline, acid or enzyme catalysts [4]. Cow/Bovine tallow however stands out because of its low cost, availability without competition with a food market, high calorific value, a high number of cetanes, and most importantly, because of its 100% conversion rate [5]. Transesterification is performed commonly using a base catalyst, such as sodium hydroxide or potassium hydroxide (NaOH or KOH), and methanol due to their low prices, effectiveness, low concentration, and heat requirements in the reaction.

Optimisation of the transesterification reaction involves varying the process parameters to obtain maximum yield and identifying the significance of each parameter that affects the yield. Parameters such as reaction temperature, duration of reaction, the concentration of acid catalyst, the molar ratio of oil to methanol and agitation speed were considered for their impacts on the process. The response surface methodology (RSM) model has been known as a powerful tool for optimization in several fields such as chemical engineering process control and chemical analysis among many other applications [6]. The response surface methodology (RSM) is used to determine the optimum conditions for the transesterification reaction. Response surface methodology (RSM) is a useful statistical technique that has been applied in research into complex variable processes. It employs multiple regression and correlation analysis as tools to access the effect of two or more independent factors on the dependent variables according to Awolu & Layokun [7] and Kalil et al. [8] have earlier described RSM as a good optimizer involving a collection of statistical techniques for designing experiments, building models, evaluating the effects of factors and searching for the optimum conditions. This paper however seeks to establish the viability of using cow tallow to produce biodiesel through esterification and thus obtain the reaction parameters needed to get optimum (highest) biodiesel yield.

MATERIALS AND METHODS

2.1 Reagents and Materials

Methanol (Sigma-Aldrich), NaOH flakes, Phenolphthalein, Magnesium trisilicate ($MgSi_3$), Sodium Sulphate, Diethyl ether.

2.2 Apparatus and Instruments

Beakers (20ml, 50ml, 100ml), Centrifuge (Hettich University II), Conical Flasks (20ml, 50ml,100ml, 1000ml), cuvettes, electronic weighing balance (B. Bran Scientific, England), heat drying oven (DHG Series Ocean Med$^+$ England), electronic temperature regulation heating mantle (98-I-B Series), HH-S thermostatic waterbath (DKS Series; Ningbo Biocotek scientific instrument company limited), measuring cylinder, pipette (1ml, 2ml, 5ml, Pyrex), test tubes (5ml, 10ml, Pyrex), gas chromatography coupled with flame ionization detector (FID) and electron capture detector (ECD), Buck scientific infra-red spectrophotometer (Model, M530), separating funnel.

2.3 Cow tallow sample collection

Fat from a cow was obtained from a slaughterhouse in Kwata, Awka, Anambra state. The fat was carefully selected to remove impurities such as bone, meat and dirt. It is then subjected to a low temperature just enough to melt it. The tallow is then collected in a clean container.

2.4 Transesterification of cow tallow

An equal volume of cow tallow and methanol (5 ml each) were mixed and stirred. 2% NaOH catalyst was added to the sample and stirred. Temperature, speed and time were also varied. The solution was poured into a separating funnel, washed with hot water and the biodiesel separated. The transesterification reaction is represented in equation (1) below

$$A + 3B = C + D \quad (1)$$

where A is the triglyceride, B is methanol, C is FAME and D is glycerol.

2.5 Separation of Biodiesel Extract

After the base transesterification process, the reaction mixture was allowed to settle for 24 hours inside a separating funnel to allow clear separation of biodiesel from glycerin by gravity. The layer on the top was the biodiesel while the bottom layer was the glycerol. Thereafter, the two layers were separated by settling using a separating funnel. The biodiesel separation was carried out by decanting as the glycerol was drained off while the biodiesel remained.

2.6 Gas Chromatography Analysis

The fatty acid composition of the biodiesel samples was analyzed by gas chromatography coupled with a mass spectrometer according to AOCS official method Ce 2-66. The gas chromatographic analysis was made using GC-MS-QP2010 Plus, Shimadzu. The GC column used was calibrated by injecting methyl ester standards. It is worth noting that good separations were achieved by diluting the samples (n-hexane collected) in a small amount of ethyl acetate. The carrier gas used was hydrogen and its flow rate was regulated at 41.27 ml/mins while the column flows at 1.82 ml/mins. The oven temperature was set at 80^{o}C before rising at 6^{o}C/min until 340^{o}C. The identification of peaks was done by comparison of their retention time and mass spectra with the mass spectra library (NIST05s LIB) [9].

2.7 Physicochemical Analysis

The physicochemical analyses of the oils were determined by the American society of testing and materials (ASTM) and the Association of analytical chemist (AOAC) standard methods. The moisture content was obtained using the air oven method using the rotary evaporator oven (BTOV 1423).

2.8 Optimisation Using Response Surface Methodology (RSM)

Response surface methodology (RSM) utilizes mathematical and statistical techniques to perform modeling and analysis of problems in which a response of interest is influenced by several variables [8]. The objective is to optimize the response from the tested variables. A standard RSM design called a central composite design (CCD) was applied to develop the experimental design of the esterification and transesterification processes. The best-fitted model based on the model fit summary and statistical analysis for linear, two-factor interactions quadratic and cubic models of the RSM is quadratic for both the esterification and transesterification reactions. The quadratic model has the least standard deviation closest difference between R-squared, adjusted R-squared and predicted R-squared, highest lack of fit p-value and least sequential p-value. Regression analysis and analysis of variance (ANOVA) were performed using design expert 7.0.0 version software. The fitted polynomial equations obtained from the regressional analysis was used to develop the response surface plots.

RESULTS AND DISCUSSION

3.1 Cow Tallow Biodiesel Composition and Physicochemical Properties

Cow tallow and other animal fats which are common raw materials for biodiesel production are composed majorly of fatty acids which influence the properties of the biodiesel. These properties are both physical and chemical properties, including the fuel properties (cetane number, viscosity, density, bulk modulus and pour point), combustion characteristics (fuel injection timing, ignition delay and ignition timing) and exhaust emissions (HC, CO, NOx and smoke). The fatty acid profile of the biodiesel produced from cow tallow is shown in Table 1.

Table 1. Tallow Biodiesel Fatty Acid Profile

Component	Name	Concentration (µg/ml)	% Concentration
C18	Stearic Acid	28.2844	31.862
C16	Palmitic Acid	15.5274	17.491
C14	Myristate	3.1727	3.574
C18:2	Linoleic Acid	4.2171	4.75
C20:2		0.7706	0.868
C20:5		2.9883	3.366
C18:1	Oleic Acid	32.5555	36.673
C12	Lauric Acid	1.255	1.413
Total		88.771	

Several researchers have established relationships between the fatty acid profile and the physicochemical properties of produced methyl ester. Knothe & Steidley [10] investigated the kinematic viscosity of numerous fatty acid compounds which are mainly contained in biodiesel. They found that the kinematic viscosity of fatty compounds is significantly influenced by the compound structure and influencing factors such as chain length, position, number and nature of double bonds, as well as nature of oxygenated moieties. Graboski and McCormick [11] expressed in their paper that the chemical composition of fat and oil esters is dependent upon the length and degree of unsaturation of the fatty alkyl chains. Also, Graboski and McCormick [11] stated that the cetane number of fatty acid increases with the chain length, decrease with a number of the double bond and carbonyl groups move toward the centre of the chain. Yamane *et al*. [12] carried out a study to investigate the effect of fatty acid methyl ester content on the peroxide value and acid value of biodiesel. Based on their investigation they reported that polyunsaturated fatty acid methyl ester, such as linolenic acid methyl (C18:3) can be oxidized easier than linoleic acid methyl (C18:2) and oleic acid methyl (18:1). Worthy of note is that most of the researches conducted on fatty acid composition of biodiesel emphasized on the fuel properties of biodiesel such as viscosity, pour point, octane number and oxidation stability. The physicochemical properties of raw cow tallow and cow tallow biodiesel is shown in Table 2.

Table 2 Physicochemical properties of cow tallow and cow tallow biodiesel

Physicochemical properties	Cow tallow	Cow tallow biodiesel
Acid value (mg KOH/g)	0.22	0.984
Specific gravity (m/v)	0.875	0.842
Ash content	0.081	0.09
Moisture content	1.32	0.61
Viscosity (mpas)	35.6	1.52
Calorific Value (KJ/Kg)	34,032	34,216
Refractive index	1.614	1.4435
Cloud point (°C)	47	-3
Pour point (°C)	-	-9
Flash point (°C)	135	81
Saponification value (mgKOH/g)	200.72	161.5
Iodine value (Wij's)	47.5	33.5
Peroxide value (Meq/Kg)	1.7	1.9
Smoke point (°C)	130	78
Titre (°C)	47	-15
Conductivity (µs/cm)	-	nil
Sulphur (%)	0.1	0.08

The ideal mixture of fatty acid composition/ratio for biodiesel has been suggested to be C16:1, C18:1 and C14:0 in the ratio 5:4:1 [13]. Such biodiesel would have the properties of very low oxidative potential [13,14]. Though this ratio is not always realized most of the time, it paints a picture of the ratio of unsaturated to saturated fatty acid profile needed for oxidative stability in biodiesel fuel. It shows that the unsaturated fatty acid should have a higher ratio to the saturated ones. This was not the case in the cow tallow biodiesel as the unsaturated fatty acid was lower than the saturated fatty acid in the ratio (45:54) as shown in Table 1. This is not ideal as oxidative stability is influenced by unsaturation [15]. The high percentage of oleic acid which is a low degree unsaturated fatty acid however compensates for the saturates in stearic and palmitic acid. In view of this, saturated fatty acids could be reduced or unsaturated fatty acids increased by controlling culture conditions.

The important parameters for a potential biodiesel are viscosity (mm^2/s), oxidative stability (h), cetane number, cold filter plugging point (°C), density (kg/m3), saponification value (mg KOH/g-oil), iodine value ($mgI_2/100$ g), and high heating value [16].

These biodiesel properties are majorly affected by compositional variations including fatty acids type, chain length, number and position of double bonds. Hoekman *et al* [17], proposed a correlation between unsaturation and biodiesel properties such as viscosity (mm^2/s), cold filter plugging point (°C), iodine value (mg $I_2/100$ g), density (kg/m^3), and high heating value. High saturation in the fatty acid profile supports the kinematic viscosity and cold flow behaviour. The effect of saturation on the cloud point (CP) can be highlighted when the cloud point of fish oil methyl ester (FOME) prepared by Kudre *et al* [18], with a lower ratio of saturated fatty acid (30.8) had a low cloud point of -5°C while cow tallow methyl ester with a higher degree of saturation (54) recorded a higher cloud point of -3 to 9°C as seen in Table 2. Unsaturation in fatty acid profile supports the density and high heating value of biodiesel [19]. The flashpoint of highly unsaturated methyl esters such as soyabean oil methyl ester (SME) as studied by Shumaker *et al.* [20], has a higher flashpoint of 171°C when compared to cow tallow methyl ester (CTME) with a lower ratio of unsaturates having a lower flash point of 81°C. The oxygen content/peroxide value of biodiesel is directly proportional to cetane number (CN) which improves smooth engine operation when increased [21]. Properties like density and heating value are also directly correlated with CN. It has been observed by several researchers that the combustion characteristic of fuel is also dependent on properties of particular biodiesel in which cetane number (CN) play an important role in engine performance [22]. The cetane number (CN) of biodiesel varies according to different feedstocks utilized in the production of biodiesel. Research conducted by Sivaramakrishnan and Ravikumar [23] noted that biodiesel possesses a lower viscosity when compared to vegetable oil due to its high flow rate. The main problem associated with biodiesel is low-temperature performance due to its high cold filter plugging point. Parameters like cold filter plugging point (CFPP), cloud point (CP), low-temperature filterability test (LTFT) which determines the cold flow behaviour of diesel fuel are also affected by the compositional changes in fatty acids [24]. Cow tallow biodiesel recorded a high acid value of 0.984 mg KOH/g as compared to ASTM D6751 standard of 0.5 mg KOH/g. This could be attributed to the high moisture content of the cow tallow (feedstock). Do Santos et al. [5] compared two bovines tallow with moisture contents of 0.2% and 0.8%, and obtained acid values of 0.6 mg KOH/g and 2 mg KOH/g respectively. This thus inferred that high humidity forms additional free fatty acids (FFA) leading to saponification reaction, which cause losses during the biodiesel production. The low peroxide/acid value (1.9) could be attributed to the low percentage of polyunsaturated fatty acids available in cow tallow biodiesel. This is because polyunsaturated fatty acid methyl ester such as linolenic acid (C18:3) which is abundant in some other biodiesel fuels with higher peroxide values oxidize faster than other less unsaturated fatty acids like oleic acid (C18:1) [12]. This thus makes biodiesel with less unsaturated fatty acids not easily oxidized thus resulting in lower peroxide values as in the case with cow tallow biodiesel.

3.2.1 Optimization of Cow Tallow Biodiesel Production using RSM

Parametric effects such as reaction time, temperature, catalyst concentration, methanol/oil ratio and stirring speed are all important factors in the transesterification of cow tallow. Their effect on cow tallow FAME yield was illustrated in Figure 1 below. Experimental runs were carried out by a combination of the five variables resulting in a total of 32 experimental runs as presented in Table 3 below. A maximum yield of 83.82 % was obtained using optimum operating parameters of temperature (54.3ºC), time (51.65 mins), catalyst concentration (1.82 wt%), methanol/oil ratio (4.08 mol/mol) and stirring speed (302 rpm). The effect of the different parameters on the actual yield and their respective RSM predictions is reported in Table 3. It was observed that run 25 had the highest actual yield of 83.9% with reaction parameters: temperature (50ºC), reaction time (50 mins), catalyst concentration (2), methanol/oil ratio (5) and stirring speed (5). The RSM prediction for this run was however 81.99% which though lower than the actual yield was high enough compared to the average yield. Also, run 6 had the highest predicted yield of 82.61% which is lower than the highest actual yield (83.9%). This however shows that RSM though an effective optimization technique has its limitations in the transesterification of cow tallow. Furthermore, the high standard deviation values (2.03) and low predicted R^2 values (0.8942) (Table 5) showed the RSM's limited ability as a prediction tool in the transesterification of cow tallow.

Table 3. Transesterification of cow tallow and RSM prediction

Run	Temp (°C)	Reaction Time (min)	Cat. Conc. (wt%)	Methanol/oil ratio (mol/mol)	Stirring speed (rpm)	Actual Yield	Rsm Prediction
1	50	50	2	5	400	80.9	78.63
2	50	50	2	7	400	79.4	80.23
3	50	50	3	5	400	81.8	81.99
4	50	50	2	5	400	80.9	80.09
5	65	45	2.5	4	500	79	78.54
6	50	45	2.5	4	300	83	82.61
7	65	55	2.5	6	500	79.4	81.99
8	50	45	2.5	6	500	80.1	81.99
9	50	45	1.5	6	300	79.3	81.99
10	50	55	2.5	6	300	79.3	77.63
11	50	50	2	5	400	80.7	80.52
12	50	50	2	5	400	80.2	81.19
13	65	55	2.5	4	300	80.9	81.14
14	45	50	2	5	400	79.8	81.18
15	65	55	1.5	4	500	80	79.03
16	50	50	2	5	400	83.4	82.21
17	50	50	2	5	600	83.3	81.31
18	50	50	2	5	400	83.1	79.42
19	50	50	1	5	400	79.4	81.13
20	50	55	1.5	4	300	83.8	81.02
21	65	45	2.5	6	300	77.9	77.73
22	50	55	1.5	6	500	78.4	76.63
23	65	45	1.5	6	500	77.5	81.99
24	50	60	2	5	400	78.4	79.62
25	50	50	2	5	200	83.9	81.99
26	50	55	2.5	4	500	79.5	77.04
27	50	45	1.5	4	500	80.1	79.46
28	50	50	2	3	400	80.2	78.13
29	65	55	1.5	6	300	76.4	79.67
30	70	50	2	5	400	77.7	79.82
31	50	45	2	5	400	83.8	80.69
32	65	45	1.5	4	300	79.3	78.03

Table 4. ANOVA for response surface quadratic cow tallow transesterification model.

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob> F	
Model	69.47	20	2.17	3.02	0.0004	significant
A-Temp (°C)	0.55	1	0.46	0.61	0.5113	
B-Rxn Time (min)	8.19	1	7.09	3.02	0.0104	
C-Catalyst con (wt%)	3.16	1	3.16	2.86	0.0112	
D-Methanol /oil ratio (mol/mol)	18.71	1	18.71	16.96	<0.0001	
E-Stirring speed (rpm)	1.17	1	1.17	1.06	<0.0001	
AB	0.031	1	0.031	0.028	0.8702	
AC	0.11	1	0.11	0.098	0.7599	
AD	2.03	1	2.03	1.84	0.0018	
AE	0.048	1	0.048	0.044	0.8384	
BC	3.03E-03	1	5.75E-01	2.24E-01	0.9592	
BD	11.26	1	10.26	10.2	0.0085	
BE	0.51	1	0.21	0.19	0.0032	
CD	1.35	1	1.45	1.32	0.2756	
CE	0.12	1	0.16	0.15	0.0002	
DE	1.7	1	1.7	1.54	0.0399	
A^2	11.92	1	11.92	10.81	0.0072	
B^2	15.67	1	13.67	12.21	0.0031	
C^2	2.84	1	2.84	2.58	0.1367	
D^2	4.18	1	4.88	4.42	0.5593	
E^2	0.15	1	0.15	0.13	0.7235	
Residual	11.14	11	1.1			
Lack of Fit	7.76	6	1.29	1.72	0.122	not significant
Pure Error	4.37	5	0.67			
Cor Total	101.61	31				

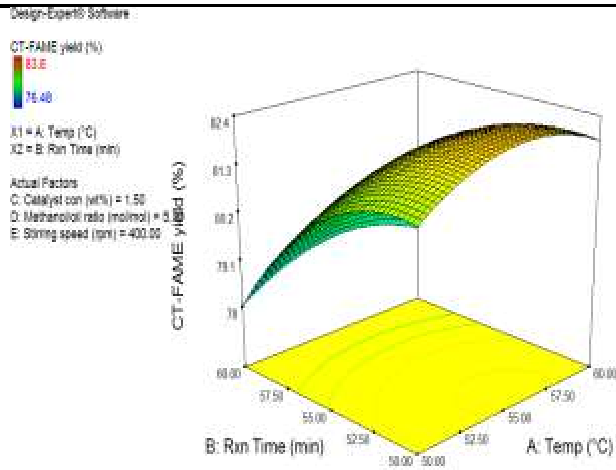
Table 5: Summary of Cow tallow transesterification regression values

Std. Dev.	2.03	R-Squared	0.9106
Mean	60.01	Adj R-Squared	0.9034
C.V. %	2.11	Pred R-Squared	0.8942
PRESS	347.26	Adeq Precision	6.012

3.2.2 Effects Of Parameter Adjustments On The Transesterification responses are Of Cow Tallow Yield

The interactive effects of adjusting the process variables within the design space were monitored using 3D surface plots. The analysis and optimization of cow tallow transesterification were completed using the Design Expert 7.0.0 and the graphical solutions presented in Figures 1a-1j. Figure 1a shows the interactive effect of temperature and reaction time on the yield of the cow tallow transesterification process. It can be seen that the shape of the surface plot increases from a reaction time of 60mins with the convex peak seen between the temperature of 54ºC to 58ºC. The maximum height of the surface plot is located at the lowest reaction time is 50 mins. This implies that the point identified for reaction time at a maximum height of the convex shape and temperatures are within the boundary conditions where the optimum yield was obtained and maximum yield is at its peak. Figure (1b) shows the effect of varying catalyst concentrations and temperature on the cow tallow trans-esterification. It can be seen that as the catalyst concentration decreases from 2.0 wt%, the shape of the surface plot being convex undulates. Hence, the peak of the convex shape is located between the catalyst concentration of 1.75 wt%to 2.00 wt% and temperature of 52ºC to 56ºC which suggests the boundary position of the optimum response and maximum yield. Figure (1c) shows the effect of varying methanol/oil ratios and temperature on cow tallow transesterification. It can be seen that the shape of the surface plot is convex which undulates at a methanol/oil ratio of 4.5 mol/mol% with the maximum height observed at 4.00 mol/mol around which the optimum response was obtained. The peak of the convex curve for temperature is between 55ºC and 57.5ºC which houses the optimum point. Hence, the optimal yield is located at the maximum height and peak of the shape. Figure (1d) shows the surface plot of the effect of interacting varying temperature and stirring speed. The shape of the surface plot is convex shaped which rises with an increase in stirring speed. The convex peak is situated at a temperature of 54ºC to 56ºC where the effect was maximally seen. Hence, this implies that the optimum yield is located at the maximum rise (when stirring speed was 300 rpm) and highest convex peak.

The surface plot of the interaction of catalyst concentration and reaction time is shown in Figure 1e. The surface plot has a convex shape that undulates almost sharply from the reaction of 52 mins and has a rise on the catalyst concentration axis. The maximum rise is seen between catalyst concentration 1.75 wt% to 2.00 wt% and reaction time of 50 mins to 52 mins. The optimum yield is contained at the maximum rise of the surface plot. Figure (1f) shows the effect of the interaction of methanol/oil ratio and reaction time. The surface has a convex shape and undulates at a methanol/ratio of 4.00 mol/mol. The convex peak is located between the reaction time of 50.3 mins and 55.2 mins and methanol/oil ratio of 4.00 mol/mol and 4.50 mol/mol. The optimum yield is located at the specified ranges of the methanol/oil ratio and a reaction time of the convex peak. Stirring speed and reaction time effect on the cow tallow FAME yield was highlighted in Figure (1g). The surface plot has its convex-shaped that undulates almost sharply at a reaction time of 55mins. The peak of the convex shape is seen at the reaction time of 52 mins. Maximum yield can be seen between reaction time of 50mins and 52mins and the stirring speed of 300 rpm and 450 rpm which houses the optimum yield. The effect of the interaction of methanol/oil ratio and catalyst concentration is shown in Figure 3.1h. The shape of the surface plot is almost a constantly rising surface shape which rises from methanol/oil ratio of 6 mol/mol and catalyst concentration of 1.00wt%. However, a drop of the surface can be seen when the catalyst concentration is 1.8wt% at a methanol/oil ratio of 4.00%. The optimum yield is located between 4.0mol/mol to 4.5 mol/mol for methanol/oil ratio and catalyst concentration of 1.50wt% and 2.00 wt%. The interaction between stirring speed and catalyst concentration is shown in Figure 1i. It is observed that the surface plot has an almost constant-rising shape on both the x-axis and y-axis. However, a bend can be seen at a catalyst concentration of 1.75 wt% which normalizes to 2.00 wt% at stirring of 300 rpm to 400 rpm. The catalyst concentration and stirring speed range specified houses the optimum yield of the CT-FAME. Figure (1j) shows the effect of the interaction between the stirring speed and Methanol/oil ratio on the FAME yield of cow tallow. The shape of the surface is a cconvex shape with the peak which accounts for the optimum yield, obtained at a Methanol/oil ratio range of 4 mol/mol to 4.50 mol/mol and a stirring speed range of 400-500 rpm.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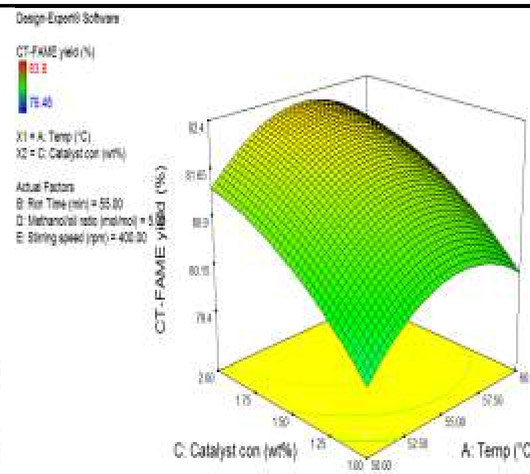
CT-FAME yield (%)
83.8
76.48
X1 = A: Temp (°C)
X2 = B: Rxn Time (min)
Actual Factors
C: Catalyst con (wt%) = 1.50
E: Stirring speed (rpm) = 400.00
CT-FAME yield (%)
B: Rxn Time (min)
A: Temp (°C)

(a)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CT-FAME yield (%)
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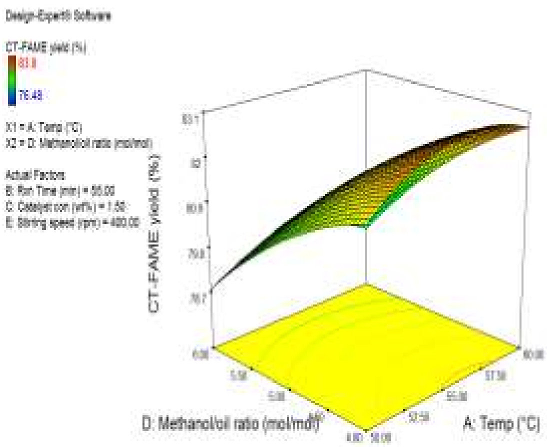
X1 = A: Temp (°C)
X2 = C: Catalyst con (wt%)
Actual Factors
B: Rxn Time (min) = 55.00
E: Stirring speed (rpm) = 400.00
CT-FAME yield (%)
C: Catalyst con (wt%)
A: Temp (°C)

(b)


Design-Expert® Software
CT-FAME yield (%)
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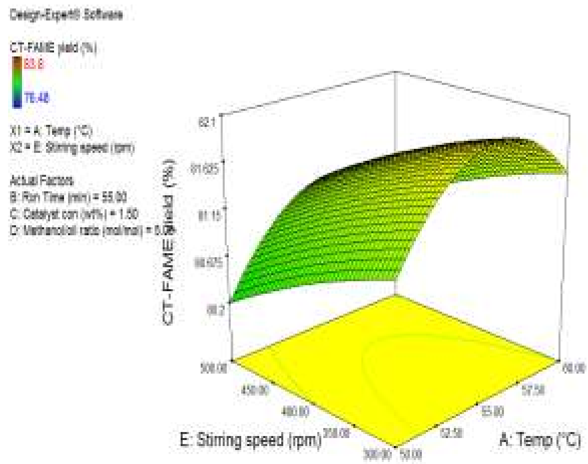
76.48
X1 = A: Temp (°C)
X2 = D: Methanol/oil ratio (mol/mol)
Actual Factors
B: Rxn Time (min) = 55.00
C: Catalyst con (wt%) = 1.50
E: Stirring speed (rpm) = 400.00
CT-FAME yield (%)
D: Methanol/oil ratio (mol/mol)
A: Temp (°C)

(c)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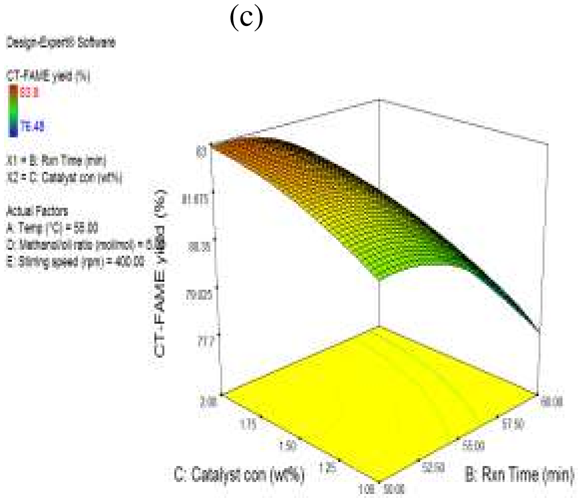
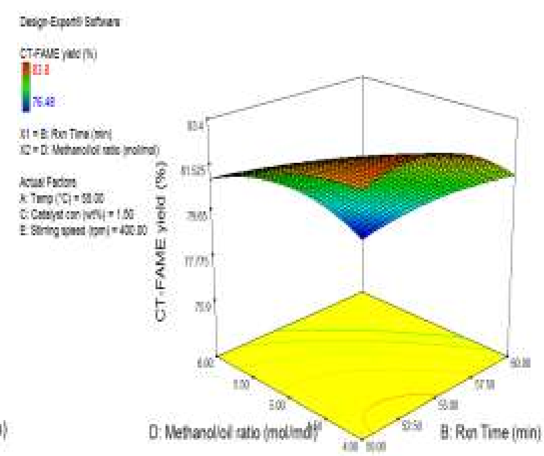
B: Rxn Time (min) = 55.00
C: Catalyst con (wt%) = 1.50
CT-FAME yield (%)
E: Stirring speed (rpm)
A: Temp (°C)

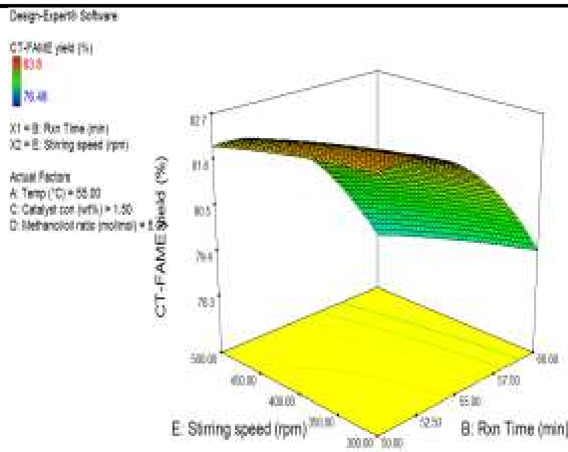
(d)


Design-Expert® Software
CT-FAME yield (%)
83.8
76.48
X1 = B: Rxn Time (min)
X2 = C: Catalyst con (wt%)
Actual Factors
A: Temp (°C) = 55.00
E: Stirring speed (rpm) = 400.00
CT-FAME yield (%)
C: Catalyst con (wt%)
B: Rxn Time (min)

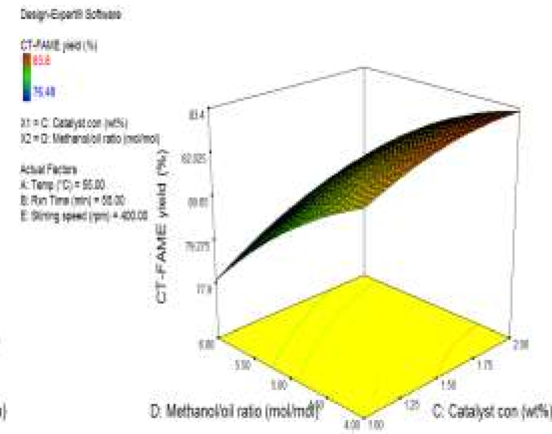
(e)


Design-Expert® Software
CT-FAME yield (%)
83.8
76.48
X1 = B: Rxn Time (min)
X2 = D: Methanol/oil ratio (mol/mol)
Actual Factors
CT-FAME yield (%)
D: Methanol/oil ratio (mol/mol)
B: Rxn Time (min)

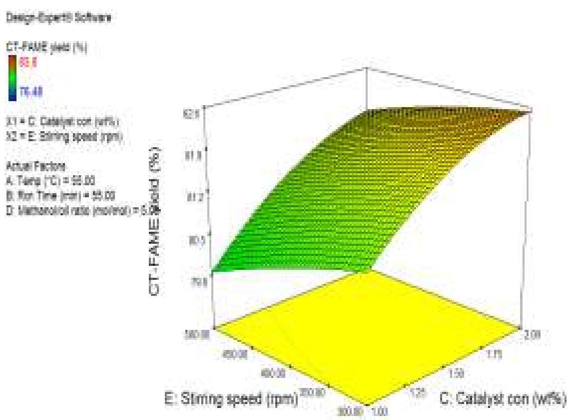
(f)



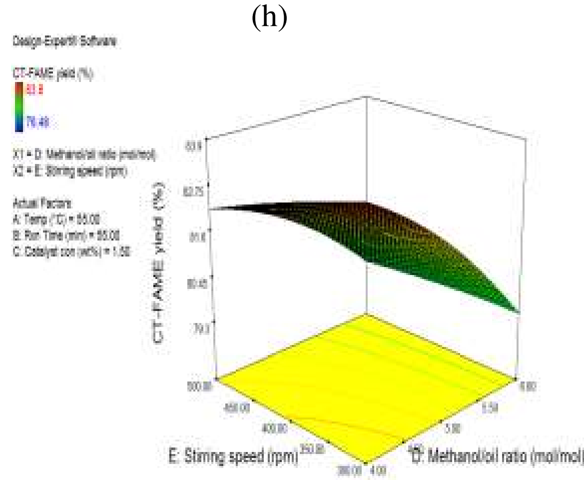
(g)



(h)



(i)



(j)

Figure 1. The 3D response surface plot of the effects of variables on CTME yield.
(a) Reaction time and temperature (b) Catalyst concentration and temperature (c) Methanol/oil ratio and temperature (d) Stirring speed ad temperature (e) Catalyst concentration and reaction time (f) Methanol/oil ratio and reaction time (g) Stirring speed and reaction time (h) Methanol/oil ratio and catalyst concentration (i) Stirring speed and catalyst concentration (j) Stirring speed and Methanol/oil ratio.

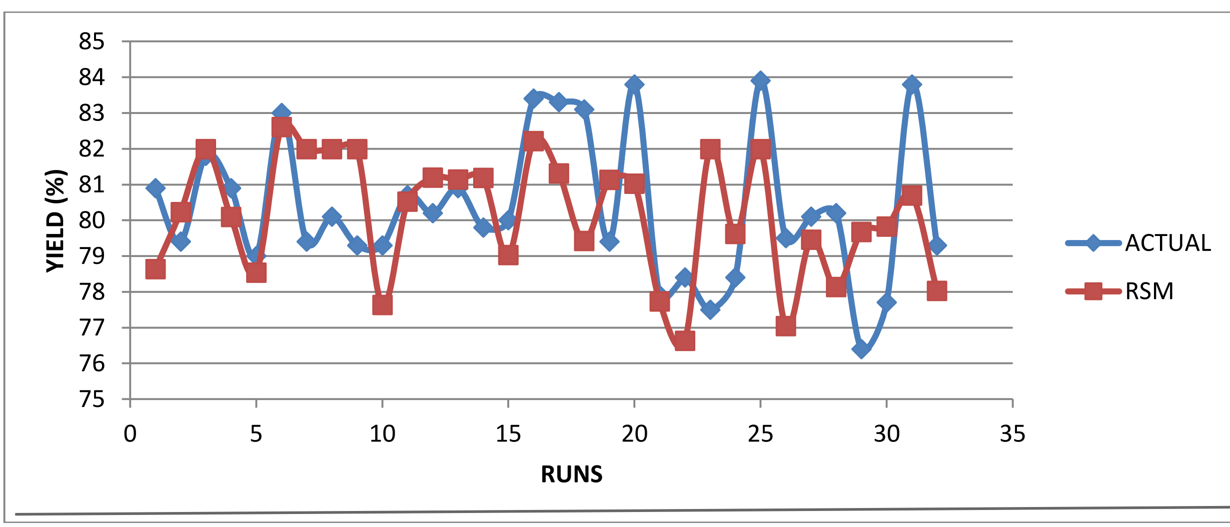


Figure 2. Comparison plot of actual yield vs RSM prediction

It can however be deduced from Figure 2 above that RSM predicted values mirror the trend created by the actual and thus can be said to be a true reflection of the actual. This shows it is a reliable optimization technique. The deviation that exists in the trend and the fact that the actual recorded higher yields than that obtained from RSM prediction thus indicates the limited ability of RSM as an optimization technique in cow tallow transesterification. Esonye et al. [25] investigated the optimization of biodiesel production from Sweet Almond (Prunus amygdalus) Seed oil (SASO) using Response Surface Methodology (RSM) and Artificial Neural Networks (ANN) models through base (NaOH) transesterification. The Central Composite Design (CCD) optimization conditions were temperature (30ºC to 70ºC), catalyst concentration (0.5%w/w to 2.5% w/w), reaction time (45 min-65 min) and oil/methanol molar ratio (1:3 mol/mol to 1:7 mol/mol). An optimized biodiesel yield of 94.36% from the RSM and 95.45% from the ANN models respectively were obtained at catalyst concentration of 1.5 w/w%, reaction time of 65 min, oil/methanol molar ratio of 1:5 mol/mol and temperature of 50ºC. The higher yield obtained using ANN compared to RSM further emphasizes its limited ability as an optimization technique in transesterification reactions. Esonye et al. [25] also analyzed the RSM model using Analysis of Variance (ANOVA). Model statistics of the ANN showed more comfortable values of Mean Squared Error (MSE) of 6.005, Mean Absolute Error (MAE) of 2.786 and Mean Absolute Deviation (MAD) of 1.89306 compared to RSM models. The RSM model also gave a coefficient of determination (R^2) of 0.9446 compared to 0.96637 obtained for the ANN model. This low R^2 value obtained using RSM is also reflected in this work with a low R^2 value of 0.9106 realized as seen in Table 5 and thus further highlighting the limited ability of RSM as an optimization technique.

CONCLUSION

It can be concluded from this work that cow tallow is a good and viable feedstock for the production of biodiesel with the varying of the reaction parameters having a significant effect on the yield. The Reaction parameters required for the optimised biodiesel yield were temperature (54.ºC), time (51.5 mins), catalyst concentration (1.82 wt%), methanol ratio (4.08 mol/mol) and stirring speed (302 rpm). The optimum yield obtained from transesterification of cow tallow using RSM was 83.82% which is almost the value of the highest actual yield at 83.9% which thus establishes RSM was a reliable optimization tool albeit with limitations in transesterification reactions. The fatty acid profile of the methyl esters also had a considerable effect on the physicochemical properties and quality of the produced biodiesel. It is recommended that the use of by-products/waste products such as in this case cow fat should be looked into as a way of sourcing cheaper feedstock for biodiesel production. Also, improved methods of cow tallow processing (collection and purification) should be harnessed to help improve biodiesel yield.

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