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INFLUENCE OF CATALYST QUANTITY AND REACTION TIME ON *IN-SITU* PRODUCTION OF BIODIESEL FROM RAW CASTOR BEAN SEED USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Trans-esterification is the most commonly used methods of biodiesel production. *In-situ* trans-esterification process uses oil (triglycerides) in seeds directly without the need for initial extraction. The *in-situ* production of biodiesel from raw castor bean seed with a batch processor was studied at initial catalyst quantity of 0.1, 0.5 and 1.5%, reaction time of 30, 60 and 90 min with a reaction temperature of 60^{0}C and alcohol seed ratio of 1:1 using the response surface methodology. Initial catalyst concentration and reaction time were subjected to central composite experimental design of the response surface methodology. Initial catalyst quantity and reaction time were found to have significant ($P<0.05$) effects on the yield of castor biodiesel produced, with increased catalyst quantity giving a negative effect on the yield after an initial amount of between 1.0 and 1.2%. The reaction time had a positive effect on the yield until after 90 minutes after which biodiesel yield reduced with increased time. The CA-Time interactions influence was small and negative, due to the superior effect of initial catalyst quantity by the formation of by-products (soaps) leading to difficult ester separation from glycerol. A second-order model was obtained to predict the yield as a function of all factors. The model predicted well the observed data with a R^2 value of 0.983. The biodiesel produced had properties comparable to the standards of ASTM while the specific gravity was higher than the specified standards.

Keyword: *In-situ* trans-esterification, Response surface methodology, Biodiesel, Castor seed, Triglyceride, Catalyst amount, Reaction time

INTRODUCTION

Trans-esterification is the most commonly used methods of biodiesel production, which is the reaction of an alcohol with oil (triglyceride) in the presence of a catalyst to produce biodiesel (ester) and glycerol as a by-product. The *in-situ* trans-esterification process uses the triglycerides in the seed directly without the need for initial extraction. This process would have removed the expression, purification and deguming processes of the raw oil thereby reducing the production cost and providing a favourable comparison with available fossil diesel (Haas and Scot, 2007). Several researchers (Zeng *et al.,* 2009; Harrington and D'Arcy-Evans, 1985; Obibuzor *et al.,* 2002; Haas *et al.,* 2004, 2007; Silver-Marinkovic and Tomosevic, 1988; Khalil and Leite, 2006; Ozgul and Turkay, 2003) have all reported the production of biodiesel using *in-situ* method. They also produced biodiesel whose properties were comparable with biodiesel obtained with the conventional process. The ratio of alcohol to seed, initial amount of catalyst, reaction temperature, time and moisture content of seed have been reported to influence the *in-situ* process and that the *in-situ* process was mainly determined by parameters affecting the extraction rate (Ozgul and Turkay, 2003; Haas *et al.,* 2004).

Castor oil bean seed is one of the non-edible vegetable seed oil and viable feedstock for biodiesel production in Nigeria. Castor oil bean seed contains between 35 and 55% oil and do not compete with food grade oil because of the seed's toxicity (Weiss, 1983). The seed contains ricin and ricine, which are poisonous to both human and animal; however, the oil extracted from the seed is not poisonous. The density, viscosity and reactivity of castor oil are higher than triglycerides found in other vegetable oils (Heidbreder *et al.,* 1999). The oil is highly soluble in alcohol, a property that makes it most favourable for biodiesel production (Asadaukas *et al.,* 1999). Ogunniyi (2006) and Ogunleye *et al.* (2008) also reported that castor oil dissolves easily in alcohol, ether, glacia, acetic acid, chloroform and benzene.

Nigeria, the 11th largest oil producer in the world with about 36 billion oil reserves (USEIA, 2007) has little documented work on the production of biodiesel, while production of biodiesel from castor bean seed using *in-situ* trans-esterification is scarce in the literature. The objective was therefore to study the influence of initial catalyst quantity and reaction time on *in-situ* trans-esterification of castor oil bean seed using response surface methodology and develop a model to describe the influence of these factors on the yield of biodiesel.

MATERIALS AND METHODS

Castor bean seeds used for the study were procured from the experimental field of the Forestry and Wildlife Department of the University of Agriculture, Abeokuta and Odeda Farm Settlement of Ogun State, Nigeria. Initial moisture contents of the seed samples were determined and were further reduced by sun drying. The dried seeds were hand threshed, passed through a Tyler sieve set to remove impurities, chaffs and other foreign matter. The seed were ground in a blending machine to reduce its size and consequently increase its surface area. The ground seeds were divided into specified weights, sealed in double polythene bags and stored in the refrigerator prior to use.

The ethanol used had a boiling point of 78^{o}C; therefore, a reaction temperature of 60^{o}C was used as widely available in literature (Van Gerpen *et al.,* 2007). Reaction temperature for trans-esterification must be below the boiling point of the alcohol used (Van Gerpen *et al.,* 2004). The catalyst quantity usually used were between 0.1 and 1.5% weight of oil (Van Gerpen *et al.,* 2007) while reaction times between 30min and 2h has been reported (Chitra *et al.,* 2005). Therefore a reaction time of between 30 and 120 min was selected. The sodium hydroxide was of analytical grade manufactured by Aldrich Chemical Co. Ltd, England. The *in-situ* reactor was a 1.25 litre wet and dry mill multi speed Osterizer blender (Pulsematic, model Cycle blend 10, Pulsematic UK) with an incorporated 500W electric heating element (240V, Semyem Electronics, Japan). The blender has a clear glass with stainless steel cutting blades. The temperature was monitored and controlled with a temperature controller (Kazuki, model KZ 200DT, Kazuki China) of 2°C accuracy connected together with a T-type thermocouple and mercury-in glass thermometer.

Production of Castor Biodiesel (Castor Ethyl-Ester)

The method as described by Dairo (2010) was used in the laboratory production of biodiesel. 400g of ground castor bean seed was charged from the top into the reactor with the amount of alcohol (400g) at ambient conditions (29 – 32°C). Both seed and alcohol were mixed for 20 minutes to obtain a homogeneous suspension. The catalyst at quantities of 0.5, 1.0 and 1.5% by weight of seed was then added to the homogeneous mixture while still stirring. The temperature of the homogenous suspension in the reactor was raised and kept constant at 60°C with a calibrated thermostat attached to the heating system. At the end of the reaction time (30, 75 and 120min), the reaction was stopped by adding ethanoic acid (1:1) to neutralize the catalyst (Ma *et al.,* 1998). The hot mixture was decanted and filtered into the solid and liquid phases.

The solid phase was removed from the filter and dried to remove excess alcohol. The decanted liquid was allowed to settle into the heavy phase (glycerol) and the light phase (ethyl-ester) in a sealed glass jar. The ethyl-ester was transferred into a plastic bottle for washing to remove contaminants such as ethanol, glycerol or catalysts. Washing was done for four times or when water below the ethyl-ester became clear. The washed biodiesel was weighed and weight recorded to determine the yield according to Eq. (1). The above procedure was performed in triplicates for all levels of experimental variables according to experimental design.

The Yield was taken as the percentage ratio of the weight of washed castor ethyl ester to that of the expressible weight of oil in castor bean seed, Eq. (1).

$$Y = \frac{W_{ester}}{W_{oil}} * 100 \tag{1}$$

Where Y is the yield (%), Wester is the weight of washed ester (g) and W_{oil} (g) is the weight of expressible oil in seed.

Experimental Design

The Central Composite Design (CCD) of the Response Surface Methodology (RSM) was used for the study. The CCD is an experimental design useful for building a second order model for responses without the need to use a complete three-level factorial experiment. It allows seeing interactions among experimental variables within the range studied, leading to better knowledge of the process and therefore reducing research time and costs (Box and Hunter, 1978). The CCD face-centered design matrix obtained from Design Expert 7.16 software (Stat-Ease, 2007) is as shown in Table 1. Catalyst quantity and reaction time were taken as the independent variable while the yield was the dependent variable or response.

Statistical Analysis

Multiple regression procedures following a second order polynomial equation, Eq. (2) was used on the data obtained from the *in-situ* experimental runs using Design Expert 7.16 software (Stat-Ease, 2007).

$$y = \beta_0 + \sum_{i=1}^{2} \beta_i x_i + \sum_{i=1}^{2} \beta_{ii} x_i^2 + \sum \sum_{i<J=1}^{2} \beta_{ij} x_i x_j \quad (2)$$

where *y* is the response, x_i and x_j are the uncoded independent variables and b_0, b_i, b_{ii} and b_{ij} are intercepts, linear, quadratic and interaction coefficients respectively.

The Analysis of variance (ANOVA), Coefficient of determination (R^2) and the lack-of-fit statistics were used to determine whether the constructed model was adequate to describe the observed data. The lack-of-fit test was performed by comparing the variability of the current model residuals to the variability between observations at replicates settings of the process factors.

RESULTS AND DISCUSSIONS

The experimental results obtained are presented in Table 1 at various levels of initial catalyst quantity and reaction time. Each data represented a mean of three replicates. The result was analyzed and fitted to a second order polynomial equation, Eq. (2) and the coefficients were determined by multiple regression analysis procedure of Design Expert 7.16 software. The regression included all experimental variables and interactions regardless of their significant levels. The ANOVA revealed a highly significant model (p-value < 0.05) and a coefficient of determination (R^2) of 0.983 as shown in Table 2. The lack-of-fit as determined by the ANOVA (p-value > 0.05) was not significant, indicating that the response model represented the actual relationships of experimental factors well within the ranges of experimental study.

Table 1: Design Matrix for the Face-Centered Central Composite Design, with Experimental Yield, Predicted Yield and Residuals

	Catalyst Quantity (%)		Reaction Time (min)				
	Coded	Actual	Coded	Actual	Experimental Yield* (%)	Predicted Yield (%)	Residual
1	-1	0.5	-1	30	67.4	67.98	-0.58
2	-1	0.5	-1	30	68.2	67.98	0.22
3	-1	0.5	1	120	90.1	88.78	1.32
4	-1	0.5	1	120	88.2	88.78	-0.58
5	-1	0.5	0	75	86.1	86.50	-0.40
6	0	1	-1	30	81.4	79.56	1.84
7	0	1	1	120	92.4	92.76	-0.36
8	0	1	0	75	94.2	94.28	-0.08
9	0	1	0	75	93.1	94.28	-1.18
10	0	1	0	75	93.6	94.28	-0.68
11	0	1	0	75	96.0	94.28	1.72
12	0	1	0	75	92.2	94.28	-2.08
13	0	1	0	75	95.1	94.28	0.82
14	1	1.5	-1	30	80.2	81.40	-1.20
15	1	1.5	-1	30	81.1	81.40	-0.30
16	1	1.5	1	120	87.2	87.00	0.20
17	1	1.5	1	120	86.4	87.00	-0.60
18	1	1.5	0	75	94.2	92.32	1.88

* Mean of three readings

Table 2: ANOVA for Response Surface Quadratic Model

Source	Sum of squares	Df	Mean Square	F-Value	p-value (Prob >F)	Remark
Model	1228.04	5	245.61	137.59	< 0.0001	significant
X1-Catalyst Amount	84.68	1	84.68	47.44	< 0.0001	
X2-Time	435.60	1	435.60	244.02	< 0.0001	
X1-X2	115.52	1	115.52	64.71	< 0.0001	
X12	73.60	1	73.60	41.23	< 0.0001	
X22	204.53	1	204.53	114.57	< 0.0001	
Residual	21.42	12	1.79			
Lack of Fit	9.12	3	3.04	2.22	0.1548	not significant
Pure Error	12.30	9	1.37			
Coefficient of Determination (R2)						0.983

The model obtained from the analysis is presented in Eq. (3). The residual distribution, defined as the difference between calculated and observed values for the response studied (ethyl ester yield) does not follow any trend with respect to the predicted variables, which indicated that the model adequately represented the biodiesel yield over experimental range studied.

$$y = 22.74 + 57.47x_1 + 0.917x_2 - 0.169x_1x_2 - 19.49x_1^2 - 4.01x_2^2 \tag{3}$$

where x_1 is the catalyst amount (%) and x_2 is the reaction time (min).

Analysis of Response (Yield of castor ethyl-ester)

The ester yield generally increased with increased initial catalyst quantity but progressively decreased from about 1.2% to the highest level at reaction time of 30 minutes and from about 1.0% at 120 minutes as depicted in the interactive plot of Fig. 1. This finding on the effect of initial catalyst quantity may be explained by the formation of by-products, possibly due to triglycerides saponification processes, side reactions, production of sodium salt, which are favoured at high catalyst quantity, this makes glycerol separation difficult, thereby decreasing the ester yield. These observations are in consonance with findings of Ramadhas *et al.* (2005); Meher *et al.* (2006); Freedman *et al.* (1984); Dorado *et al.* (2002); Zeng *et al.* (2009). Since the acid value of castor oil was high, another possible side reaction, the neutralization of free fatty acids, which produces sodium soaps, may also be considered partly responsible for the decrease in the yield. Due to their polarity, the soaps formed dissolved into the glycerol phase during the separation stage after the reaction, leading to a decrease in yield.

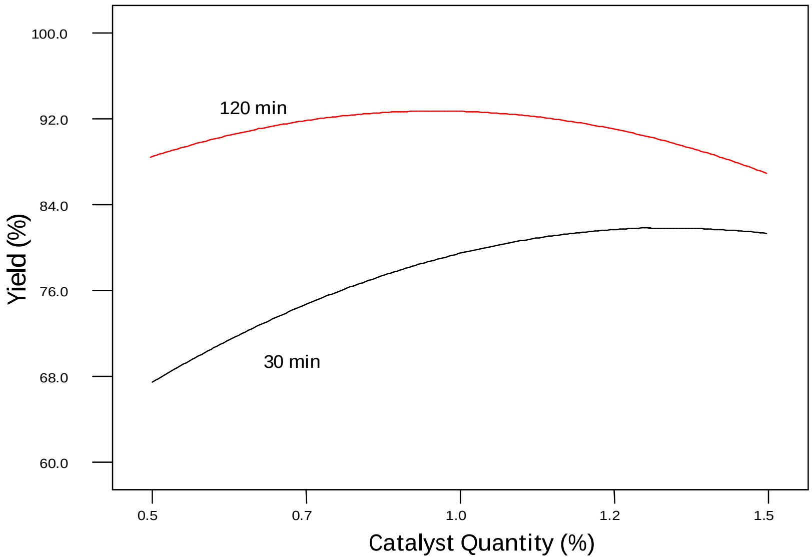

Figure 1: Interactive and Single Effect of Catalyst Quantity and Reaction Time on Yield of biodiesel

In addition, the dissolved soaps increased the ethyl ester solubility in the glycerol causing additional loss of yield. Both soaps formation and triglycerides saponification are undesirable side reaction because they partially consume the catalyst, decrease the biodiesel yield and make the separation and purification steps more difficult. This observation is also true for higher reaction times where the maximum yield was higher and was reached at a lower amount of catalyst amount. Fig.1 also depicts the interaction between catalyst quantity and reaction time on yield of biodiesel. As the reaction time increased from 30 to 120 minutes, the yield was enhanced with increased catalyst quantity from 0.5 to 1.0%, after which there was an observed reduction in yield with increased catalyst amount. The decrease in yield at increased catalyst quantity and increased time indicated a negative influence of catalyst quantity-time interaction as also observed from the response model where the coefficient was negative. Freedman *et al.* (1984); Ma *et al.* (1999); Meher *et al.* (2006); Srivastava and Prasad (2000) among others have found that reaction starts very fast and almost 80% of conversion takes place in the first 5 minutes and after 1 hour almost 93-98% conversion of triglycerides into ester takes place. In this study the yield was found to increase with reaction time from 30 minutes to between 75 and 90 minutes of reaction, after which the yield decreased with increased reaction time as shown in the contour plot of Fig. 2.

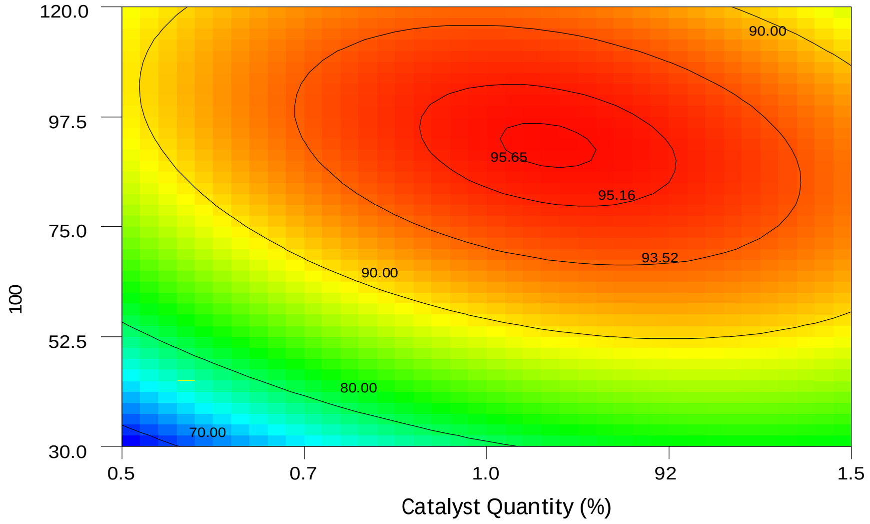

Figure 2: Contour Plot showing the Effect of Reaction Time and Initial Catalyst Quantity on Yield of Biodiesel

At early times of reaction, the yield was low due to the slow rate of conversion to ethyl-ester; however, the yield increased as reaction time increased and mixing occurs in consonance with the observations of Ma *et al.* (1999). The decrease in yield after about 90 minutes might be attributed to the interaction of reaction time with catalyst quantity. Krishnakumar *et al.* (2008) also reported a similar observation with rice bran oil after one hour of reaction. The response surface plot of ester yield versus initial catalyst quantity and reaction time obtained from the response model is shown in Fig 3. A thorough observation showed that the maximum ester yield was likely achieved at between 1.0 and 1.2% catalyst quantity and range of between 75 and 90min of reaction time.

Physical properties of biodiesel yield

The castor ethyl-ester (biodiesel) produced was characterized by determining the viscosity, specific gravity, heating capacity, acid value, flash point and pour point as shown in Table 3 in comparison to ASTM standards, EU standard, petroleum diesel, and ethyl ester from soybeans, jathropha and palm oil. All the property measured met the ASTM standards (ASTM, 2004) but the specific gravity. The specific gravity value (0.932 kg/cm^3) was 5.7% greater than ASTM standard, 8.0% greater than standard value of fossil diesel (0.850 kg/cm^3) and about 6% higher than the average estimate (0.982 kg/cm^3) for ethyl-esters. This observation is in conformity with observations of Feroro (2007) who obtained specific gravity of 0.9268 for castor ethyl-ester and that of Cvengros *et al.* (2006) on castor biodiesel using the conventional method. The specific gravity of fuel determines the amount of heat that is present in a unit of fuel, consequently the higher the specific gravity the higher the heating value. However, Sharma *et al.* (2008) reported that lower value of the specific gravity of the final product of transesterification is an indication of completion of reaction and removal of heavy glycerin.

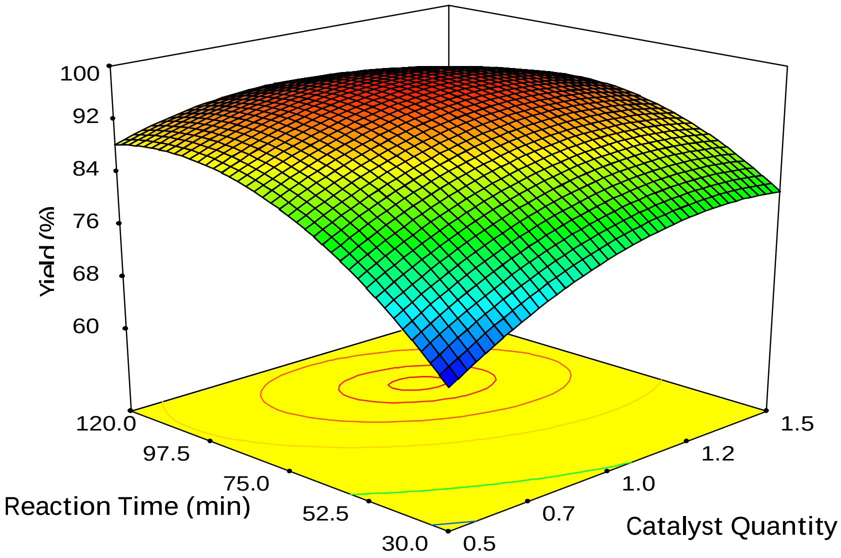

Figure 3: Effect of Initial Catalyst Quantity (0.5—1.5%) and Reaction Time (30— 120 min.) on Yield of Biodiesel

Table 3: Characterization Properties of Experimental Castor Ethyl-Ester in Comparison with, ASTM Standard, EU Standard, Esters from some Oil Seeds, and Fossil Diesel

Property	Unit	Castor Ethyl-ester[a]	ASTM Standard D6751-02[e]	EU Standard EN 14214[e]	Soy Ethyl-Ester[b]	Palm Oil Ethyl-ester[c]	Jathropha Bio diesel[d]	Fossil Diesel Fuel[e]
Specific Gravity @ 15OC	g/cm3	0.932	0.880	0.86 – 0.90	0.881	0.883	0.880	0.850
Viscosity @ 40OC	mm2/s	5.747	1.9 -6.0	3.5 – 5.0		4.839	4.84	2.0 – 3.0
Flash Point	OC	177.3	>130	>120	160	167	192	74
Pour Point	OC	-21.1	-	-	-4.0	2	6	-18
Heating Value	MJ/kg	40.46	-	-	40.0	40.56	37.2	42.9
Acid Value	mg KOH/g	0.341	<0.8	<0.50	-	-	0.24	-

a, Present study; b, Cvengros *et al.* (2006);); c, Alamu *et al.* (2007); d , Raheman and Phadatare (2004); e, Canakci and Van Gerpen (2003)

CONCLUSION

The response surface methodology, a statistical design of experiments was applied to produce ethyl-ester (biodiesel) from raw castor oil bean seed using *in-situ* trans-esterification method. The study showed that the initial catalyst quantity was the more important factor affecting the yield within the experimental range considered. Catalyst quantity had a positive influence on the yield, while the influence of the catalyst quantity-reaction time interactions were small and negative, due to formation of by-products (soaps) leading to difficult ester separation from glycerol. Reaction time also had a positive influence on the yield obtained. According to this study, biodiesel obtained from raw castor oil bean seed met some standards of ASTM D6751-02 while the specific gravity was higher than the established standards. The obtained model together with other statistical methods could be used to determine the optimum operating process factor conditions for the industrial process using a minimal number of experiments with the attendant economical benefit.

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