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EFFECT OF ROASTING CONDITIONS ON THE YIELD AND BIOMASS QUALITY OF OIL EXTRACTED FROM JATROPHA SEEDS

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Abstract

The focus of this study was to evaluate jatropha oil characteristics as a potential source of energy that would substitute kerosene and diesel as most affordable, dependable, sustainable and cleaner alternative energy for cooking and lighting. Extraction of oil from Jatropha seeds was performed with the aid of a solvent extraction process. Optimization of the process was achieved by applying Central Composite Rotatable Design technique of Response Surface Methodology. The independent variables were roasting temperature (120-160^oC) and roasting duration (30-70mins), while the responses were Oil Yield (OY), Free Fatty Acid (FFA), Saponification Value (SV), Peroxide Value (PV), Iodine Value (IV), Kinematic Viscosity (KV) and Specific Gravity (SG). Data obtained from this study were analyzed using Analysis of Variance (ANOVA) and Regression analyses. The results show that the combined effect of the roasting conditions significantly influenced all the responses at $p<0.05$ and with coefficients of determination R^2 of the models ranging between 0.80-1.00, suggesting that all the models developed had good fits. Further more, super-imposition and overlay plot of the seven responses showed that roasting at 148.18^oC for 37.34 mins would give optimum oil yield (39.0%) and good quality attributes (FFA=1.96%, SV=123.27mg/ml, PV=5.97meq/kg, IV=81.83g/100g, KV=14.99mm^2/s and SG=0.916g/ml). The oil obtained at this level might be used as biokerosine in stoves or processed into biodiesel with minute or tolerable emlusion problem.

Keywords:Jatropha seeds, Roasting Condition, Renweable Energy, Oil extraction, Optimization.

INTRODUCTION

Oil is an important component of food plant, and found along with other components such as protein, carbohydrate (cellulose, hemicelluloses, starch) in plant cell (Akinoso and Raji, 2011, Dominguez *et.al*., 1996). The oil content of various seeds varies and this serves as a basis for the classification of seeds into oilseeds and non oilseeds. Seeds with oil content greater than

17% are classified as oilseeds (Bachman, 2004).

Oils and fats are composed primarily of triglycerides. Tri-glycosides consist of a glycerin backbone with fatty acid radicals attached in place of the hydroxyls. The relative amounts of the different fatty acid radicals determine the properties of the specific triglyceride (Canakci and Van-Gerpen, 2001). Liu (1994) established that oil should not contain more than 1% FFA for alkaline–catalyzed trans-esterification reactions. This corresponds to an acid value of 2 mg KOH/g. If the FFA level exceeds this amount, the formation of soap will inhibit the separation of the ester from the glycerin and also reduce the ester conversion rate.

The depletion of world petroleum reserves and the increased environmental concerns have stimulated the search for alternative sources for petroleum-based fuel, including diesel fuels. Because of the closer properties, biodiesel fuel (fatty acid methyl ester) from vegetable oil is considered as the best candidate for diesel fuel substitute in diesel engines. *Jatropha curcas* (Linaeus), a non-edible oil-bearing seed with 30% oil (Pramanik, 2003) and drought-hardy shrub with ecological advantages, belonging to the *Euphorbiaceae* family, was found to be the most appropriate renewable alternative source of biodiesel (Tint and Mya,2009). However, using raw vegetable oils for diesel engines can cause numerous engine–related problems (Korus *et al*., 1982; Van der Walt and Hugo, 1982). The increased viscosity and low volatility of vegetable oils lead to severe engine deposits, injector coking, and piston ring sticking (Perkins and Peterson, 1991). These effects can be reduced or eliminated through trans-esterification of vegetable oil to form methyl esters, commonly known as biodiesel (Perkins and Peterson, 1991). The oil extracted from jatropha can also be used as a substitute for kerosene on aristo stoves without any further processing. This is more economical compared to kerosene from crude oil, which are used for rural electrification. Moreover, the raw oil is used by most rural folks for soap making which ease them of most economic problems (Mndeme, 2008).

Pretreatments are conventional methods of preparing oil-seeds for oil extraction. These include operations such as grinding, roasting, dehulling, flaking, cooking, or steaming (Akinoso and Raji, 2011). It is usually carried out to fractionate oil intact bodies in order to enhance the release of oil during extraction (Kumar, 2009). Most oil seeds and nut are heat-

treated by roasting to liquefy the oil in the plant cells and facilitate its release during extraction (Cammerer and Kroh, 2009).

However, some of Jatropha oils are lost in the cake as well as the degradation of oil properties which jeopardizes biofuel efficiency because the seeds are not pretreated prior to oil extraction. Hence, there is the growing demand for transportation fuel for vehicles, trucks and trains in most countries couple with the current global energy crisis and it attendant effect on rural dwellers. There is therefore the need to explore the possibility of optimizing Jatropha oil yield by pre-treating the seeds at different roasting temperature and duration combinations in order to ascertain optimum conditions (temperature and duration) for best oil yield without compromising its biomass quality.

METHODOLOGY

The experimental design adopted for this study was central composite rotatable design technique (response surface methodology) for a two-variable case as described by Montgomery (2005). Roasting temperature (X1: 120,130,140,150 and 160^{o}C) and roasting duration (X2:30,40,50,60 and 70 min) were the independent variables (Table 1), while the responses were oil yield (OY) and oil quality (free fatty acid (FFA), kinematic viscosity (KV),peroxide value (PV), iodine value (IV), saponification value (SV) and specific gravity (SG). The choice of levels was influenced by results of experimental trials and literature. Design-expert version 6.0.10 (Stat Ease Minneapolis, MN) software was used for ANOVA, mathematical modeling, regression analysis, and optimization. Optimized conditions were obtained by keeping FFA, PV, SV, and KV to be minimum, and maximizing IV,SG and OY.

Determination of moisture content

Jatropha seeds obtained from Ilorin, ASA Local Government Area of Kwara State, Nigeria were manually cleaned and the moisture content of the seeds was determined using AOAC (2007) method.

Heat treatment

Roasting of the seed was done in an oven (model CS-01b, Classic Scientific, Mumbai, India). Temperature stability was achieved by Akinoso (2006) method. The granule initial temperature was raised to equilibrium with roasting temperature. This was achieved by wrapping it in polythene bag and placing it in oven at desired roasting temperature level. This sample was later

heated by spreading thinly on a heat conductor tray in the oven at a preset temperature for required duration. Stopwatch was used to monitor the time.

Oil extraction

Each treated sample (100 g) was separately soaked in solvent (n-hexane) at RT (2928C) for 48 h (Table 1). Solvent granules ratio of 2:1 v/w was used as recommended by Ismail and Yee (2006). Oil–solvent solution was separated by evaporation in water bath heated at 70^{o}C. The OY was calculated as the ratio of the weight of the extracted oil to the weight of the raw sample.

Chemical Evaluation of Jatropha Oil.

The extracted Jatropha oil was analyzed for some important chemical properties. The acid value, iodine value, peroxide value, and saponification value of the oil were determined following the standard American Oil Chemists' Society (AOCS) methods F 9a-44, Cd 1-25, Cd 8-53,and Cd 3-25, respectively (AOCS,1997).

Determination of specific gravity (SG)

This was carried out as described by Pomeranz and Meloan (1987) method.

Determination of Kinematic Viscosity (KV)

The kinematic viscosities were determined at 40°C, using a calibrated viscometer. The procedure of ASTM D 445 was followed.

RESULTS AND DISCUSSIONS

Moisture content

The moisture content of un-treated jatropha seed was determined to be 4.8% wet basis and it is within the recommended moisture content range (2.9–6.2% wet basis) for obtaining high yield and good quality oil from oil seeds (Akinoso and Raji, 2011).Therefore, results of this experiment are not likely to be badly influenced by moisture content of the raw materials.

Effect of roasting conditions on oil yield (OY)

Oil Yield ranged from 27.0 to 39.0% (Table 1), with a mean value of 32.83±0.59%.This showed that roasting enhanced the release of available oil by 9.0%, as compared with 30.0% of unroasted jatropha seed reported by Akintayo (2004). Similar result was also reported by Kumar *et al.* (2009) for pulsed infrared roasting of groundnut under different roasting temperature and duration. They stated that roasting enhanced the release of groundnut oil by 0.83-8.09% depending on the roasting

temperature (160^0C-200^0C) and duration (5.2-12.0 min) level combinations. The oil content obtained fell within the range (30-40%) reported by Kandpal and Madan (1995) for jatropha, but contrary to 80% oil reported by Belewu *etal*.(2010). Variation in the oil content might be as aresult of varietal differences. Increase in availability and extractability of oil from roasted seed can be attributed mainly to the ablility of as a heat treatment to fractionate oil bodies and to rupture cellular structure (Kumar *et al*.,2009). The graph in Figure 1 showed a cubic relationship with peak point (maximum oil yield) at 150^0C and 40 min roasting temperature and duration respectively. Higher temperature resulted into loss of oil, as a result of partial decomposition of the sample during roasting .Lower oil yield was observed at temperature lower than 150^0C even at higher roasting duration, because of the inability of the roasting temperature to fractionate oil bodies. The linear and quadratic effects were insignificant but significant at the cubic term ($p<0.05$). Equation 1, expressed functional relationship between OY and the independent variables. Roasting temperature (^{0}C) and duration (min) were represented by X_1 and X_2 respectively. Higher value of coefficient of determination (R^2 = 0.61) indicated that the model had a good fit (Table 2).

TABLE 1: LEVEL COMBINATIONS FOR ROASTING CONDITIONS AND RESULTS OBTAINED

	Coded		Actual								
Runs	X1	X2	X1	X2	Oil Yield (%)	Free Fatty Acid(%)	Peroxide Value (mEq/kg)	Saponification Value (ml)	Iodine Value (g/100g)	Kinematic Viscosity (mm/s)	Specific Gravity (g/ml)
1	-1	-1	130	40	32.2±0.04	1.5±0.17	4.9±0.14	126.0±0.14	83.0±2.12	15.2±0.28	0.9±0.01
2	1	-1	150	40	39.0±0.01	1.5±0.06	5.6±0.14	122.0±0.28	81.0±2.12	14.7±0.15	0.9±0.01
3	-1	1	130	60	30.4±0.07	2.6±0.13	5.6±0.14	125.0±1.13	84.0±2.12	15.1±0.13	0.8±0.03
4	1	1	150	60	31.2±0.07	1.9±0.08	6.3±0.14	121.0±0.85	78.0±0.71	14.5±0.13	0.9±0.01
5	0	1.4	140	70	31.6±0.14	2.9±0.25	5.6±0.21	122.0±1.77	82.0±1.41	14.5±0.28	0.9±0.01
6	0	-1.4	140	30	30.4±0.14	2.7±0.08	5.6±0.07	126.0±1.41	83.0±0.71	15.5±0.21	0.9±0.01
7	1.4	0	160	50	27.0±0.14	2.0±0.16	6.5±0.14	120.0±1.41	77.0±1.41	14.5±0.16	0.8±0.03
8	-1.4	0	120	50	31.0±0.21	1.8±0.16	4.9±0.07	128.0±1.41	84.0±1.06	15.5±0.19	0.9±0.01
9	0	0	140	50	34.8±0.21	1.2±0.13	6.3±0.21	124.0±2.12	82.0±0.85	15.0±0.21	0.9±0.01
10	0	0	140	50	34.8±0.21	1.2±0.13	6.3±0.21	124.0±2.12	82.0±0.85	15.0±0.21	0.9±0.01
11	0	0	140	50	34.8±0.14	1.2±0.13	6.3±0.21	124.0±2.12	82.0±0.85	15.0±0.21	0.9±0.01
12	0	0	140	50	34.8±0.14	1.2±0.13	6.3±0.21	124.0±2.12	82.0±0.85	15.0±0.21	0.9±0.01
13	0	0	140	50	34.8±0.14	1.2±0.13	6.3±0.21	124.0±2.12	82.0±0.85	15.0±0.21	0.9±0.01

Table 2. Summary of Analysis of Variance for Response Surface Models

Parameter	Sum of square	df	Mean square	F value	p-value
OY (%)	109.42	12	15.63	44.27	0.0003
FFA (%)	4.04	12	0.81	12.59	0.0022
PV(mEq/kg)	2.96	12	0.59	5.74	0.0203
IV(g/100g)	50.26	12	10.05	139.10	0.0001
SV(ml)	56.33	12	28.17	207.26	0.0001
KV(mm/s)	1.22	12	0.61	47.67	0.0001
SG(g/ml)	0.019	12	$2.32x10^{-3}$	$6.37x10^{7}$	0.0000

Data Source: Lab Experiment

$$OY = +1268.62 - 25.88X_1 - 9.25X_2 + 0.45X_1X_2 + 0.12X_1^2 - 0.42X_2^2 - 2.70x10^{-3}X_1^2X_2 + 2.90\,x10^{-3}X_1X_2^2 \quad (1)$$

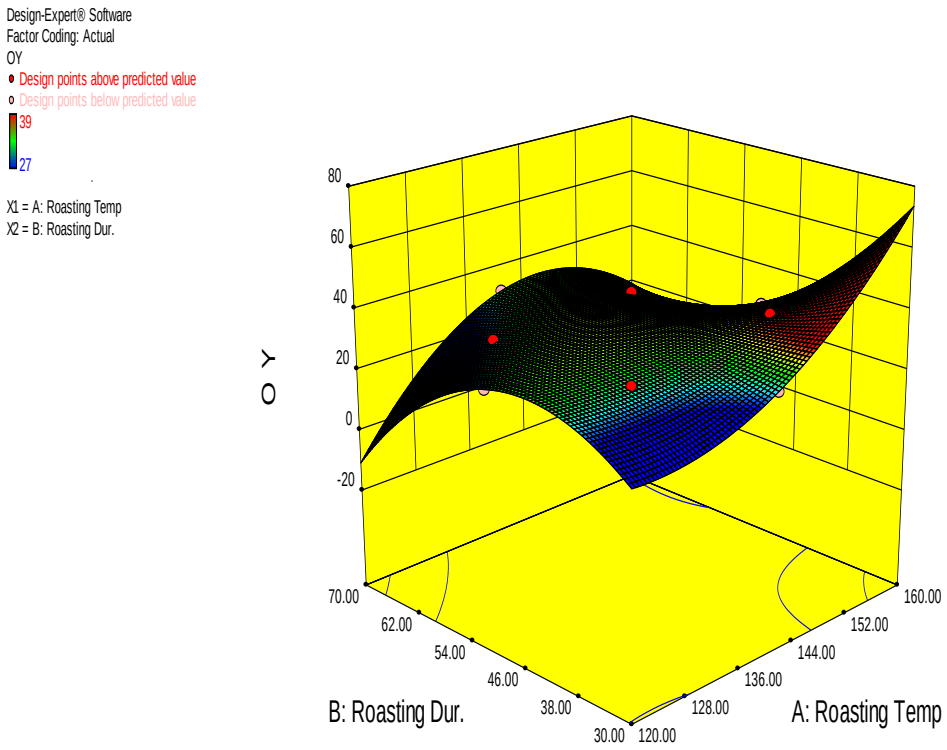

Figure 1: Plot of oil yield against roasting temperature and duration

Effect of roasting conditions on free fatty acid (FFA)

FFA ranged from 1.21-2.86% (Table 1) with a mean value of 1.76±0.25%. Free fatty acid is formed due to the hydrolysis of triglyceride and is responsible for off flavor development during storage. Inappropriate conditions such as high temperature, moisture and presence of active lipase are responsible for the formation of FFA in fat containing raw materials or oils (Kumar *et al.*, 2009). Moisture content and lipase activity in the oilseed could be controlled by thermal process such as roasting (Hamilton, 1995). Increase in FFA content of the oil with increase in roasting temperature and duration may be due thermal oxidative decomposition of oil during roasting. Inactivation of lipase enzyme at roasting temperature of 120^{o}C contributed to moderate level of FFA development in jatropha oil which could favour the shelf life extension of the oil, compared to the high FFA (5.23%-22.6%) in oil extracted from unroasted jatropha seed (Tint and Mya 2009) . From the response surface plot, a quadratic model was used to represent the relationship (Eq. 2) and model satisfied lack of fit test $p<0.05$. Coefficient of determination R^2 is 0.90.Taking the coefficient of determination into a generalized model, a second order polynomial equation generated for FFA was stated below;

$$FFA = +1.24 - 0.045X_1 + 0.28X_2 - 0.65X_1X_2 + 0.67X_1^2 + 1.59X_2^2 \quad (2)$$

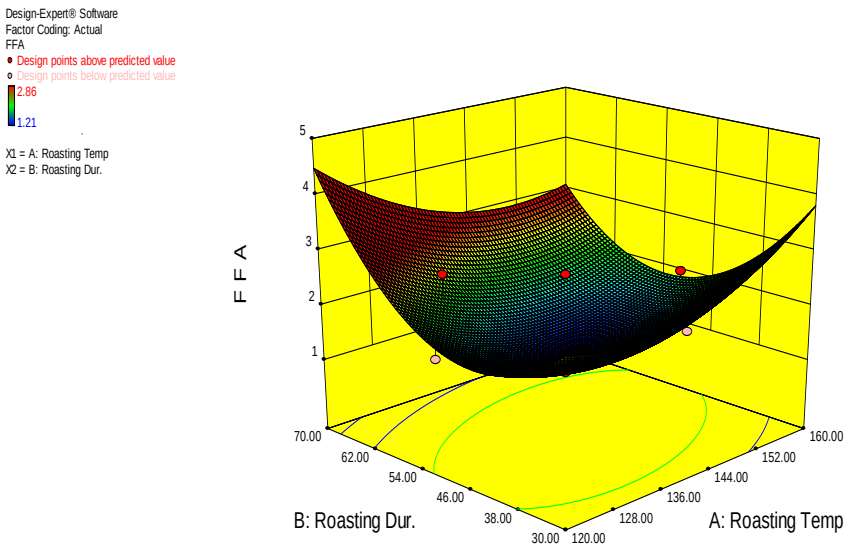

Figure 2. Plot of free fatty acid (FFA) against roasting temperature and duratio

Effect of roasting condition on specific gravity (SG)

The SG of oil is directly related to the relative density and it is very important in determining the quality of oil and efficiency of the extraction procedures. Recorded SG ranged from 0.80 to 0.92 with a mean value of 0.89±0.039 g/ml.From Figure 3, the SG was higher in samples that were roasted at 136-152^{o}C for 38-54mins,this is relative to many edible oils (Rapeseed oil, 0.906-0.914g/ml and sunflower oil,0.894-0.899g/ml)(Salunkhe *et al*.,1992).This value also fell in the range reported for olive (0.910-0.920), coconut (0.908-0.921), rapeseed (0.910-0.920), and canola (0.914-0.920) oils (Nichols and Sanderson, 2003). Further increase in roasting temperature and duration resulted in lower specific gravity. From the response plot a quadratic model was used to represent the relationship (Eq.3). Higher value of coefficient of determination (1.00)indicated that the model was a good fit.

$$SG = +0.92 - 0.035X_1 + 0.04X_1X_2 - 0.055X_1^2 - 0.02X_2^2 - 0.32\,X_1^2X_2 + 0.26\,X_1X_2^2 - 0.5\,X_1^2X_2^2 \quad (3)$$

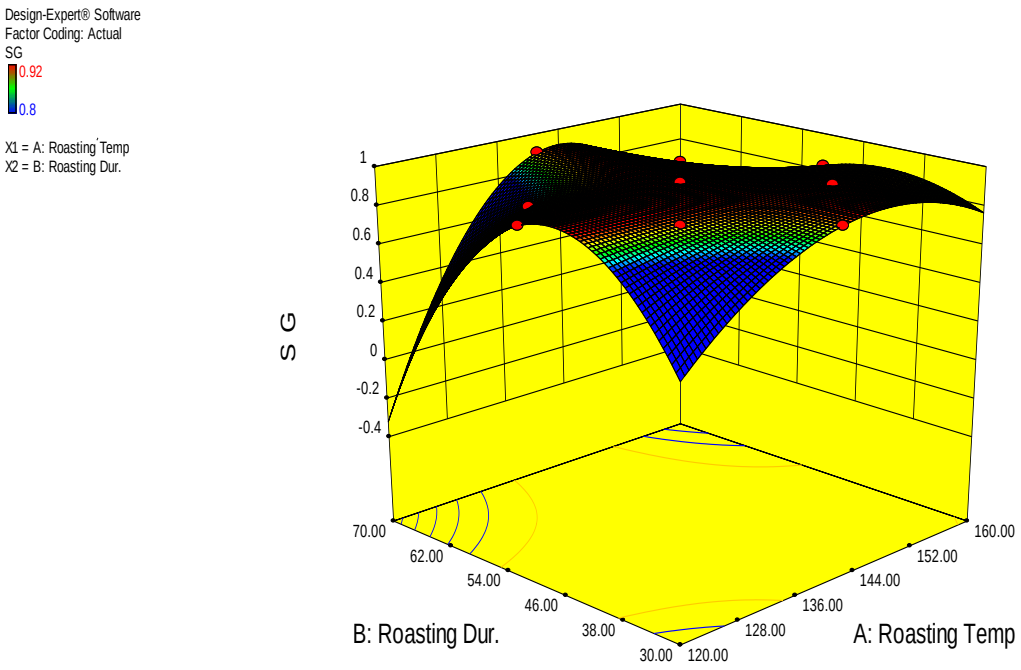

Figure 3; Plot of specific gravity against roasting temperature and duration

Effect of roasting conditions on Peroxide Value (PV)

Peroxide value obtained ranged from 4.90-6.50 mEq/Kg, with a mean value of 5.88±0.32 mEq/Kg. Quadratic model was found appropriate to express the relationship (Eq. 4). The model has high coefficient of determination R^2 (0.98) and satisfied lack of fit test at $p<0.05$.

$$PV = +6.20 + 0.77X_1 + 0.23X_2 - 0.063X_1^2 - 0.73X_2^2 \quad (4)$$

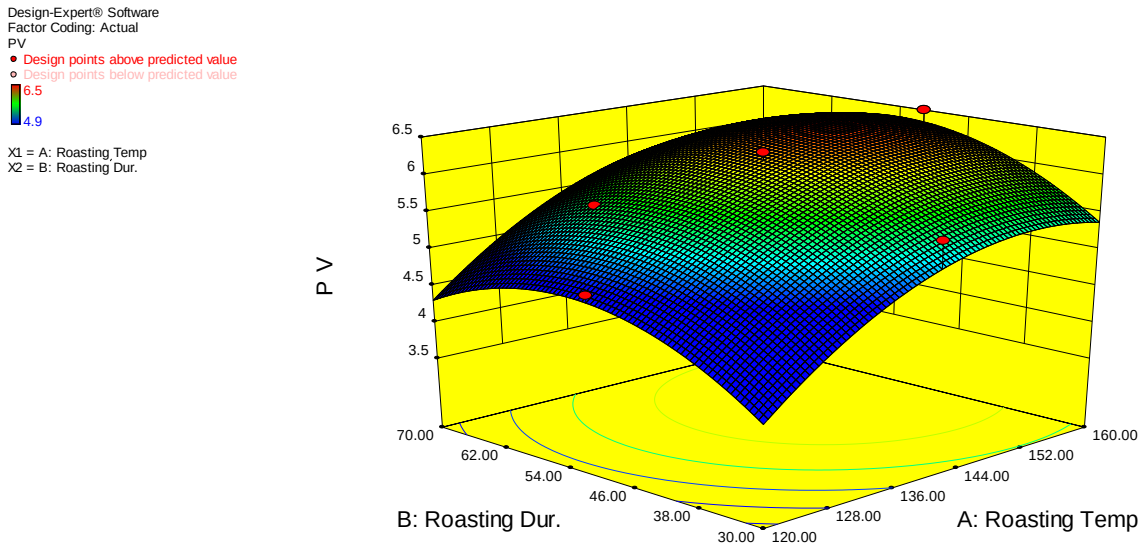

Figure 4. Plot of peroxide value (PV) against roasting temperature and duration

From Figure 4, it could be seen that that the peroxide value was lower in the samples that were roasted at 120-140^{o}C for 30-40 mins. At higher temperature and longer duration, a significant increase in the PV was noticed. This is caused by thermal oxidative transformation of free radicals, which initiated the degradation of unsaturated fat producing volatile compound (Wang *et al*., 1995). On the other hand, according to the Codex Alimentarius Commission, the peroxide value for unrefined vegetable oil may be maximum 20 meq/kg oil (Markovic and Bastic, 1975). Therefore, the jatropha

oil obtained was unrefined and its PVs were within the reported limits.

Effect of roasting conditions on Iodine Value (IV)

The jatropha seed oil obtained had an iodine value of 77-84g/100g (Table 1) with a mean value of 81.69±0.27, indicating a high degree of unsaturation. From Fig. 5, it was observed that the iodine value decreased as the roasting temperature and duration increased**.** The iodine values at all the roasting temperature and duration level combinations were a little below the allowable international standard level (100-200g/100g) of iodine in biodiesel oil products (Felix and Clement, 2011).The mean value was higher than 80.0 reported by Esuoso *et al.*(1998), and lower than 123.0 of Younis *et al.*(2000), 116.0-133.4 of Markovic and Bastic (1975) for Cucurbita species. It also fell below the range reported for cottonseed, canola, rapeseed, and corn oils (Nichols and Sanderson, 2003).The ANOVA revealed a significant effect ($p<0.05$) in quadratic relationship (Table 2).Coefficient of determination R^2 of the model was 0.99 and satisfied lack of fit test at $p<0.05$.

$$IV = +81.93 - 3.67X_1 - 0.67X_2 - 4.00X_1X_2 - 1.52X_1^2 + 0.48\,X_2^2 \quad (5)$$

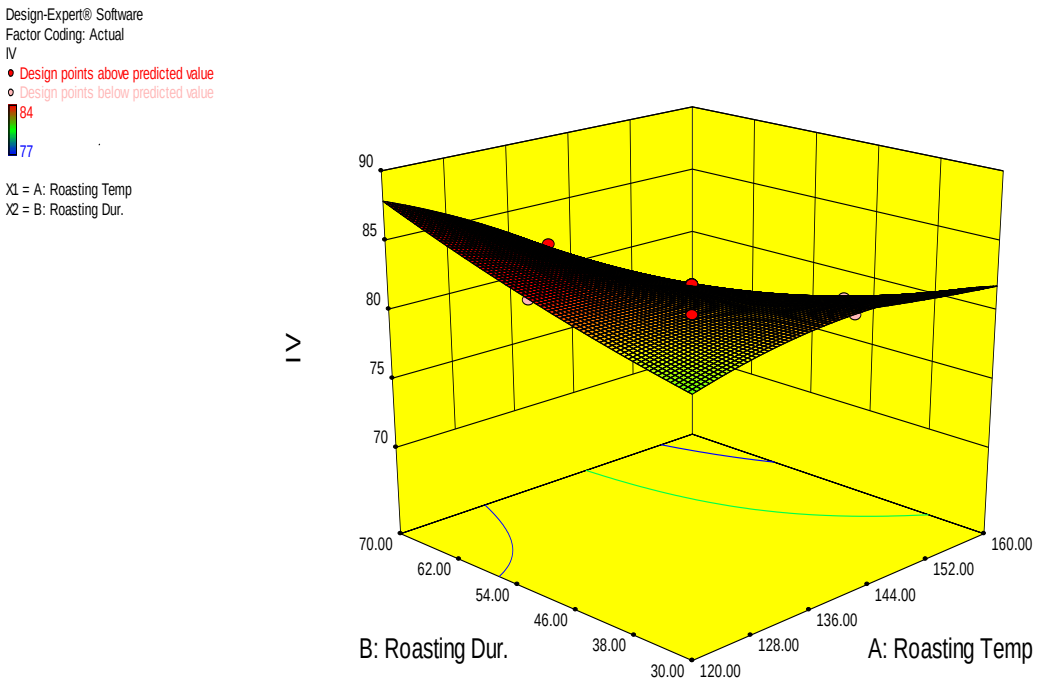

Figure 5. Plot of Iodine Value (IV) against roasting temperature and duration

Effect of roasting conditions on Saponification Value (SV)

The saponification value obtained ranged from 120 -128ml (Table 1) with a mean value of123.85±0.37. Response surface plot shown in Fig. 6, best described the relationship between saponification value of the oil and the roasting conditions that jatropha seeds were subjected to. All the model terms were significant, and the linear model satisfied lack of fit test at $p<0.05$.The coefficient of determination R^2 of the model was 0.98 and this indicated that the model had a good fit.

$$SV = +123.85 - 4.00X_1 - 1.67X_2 \quad (6)$$

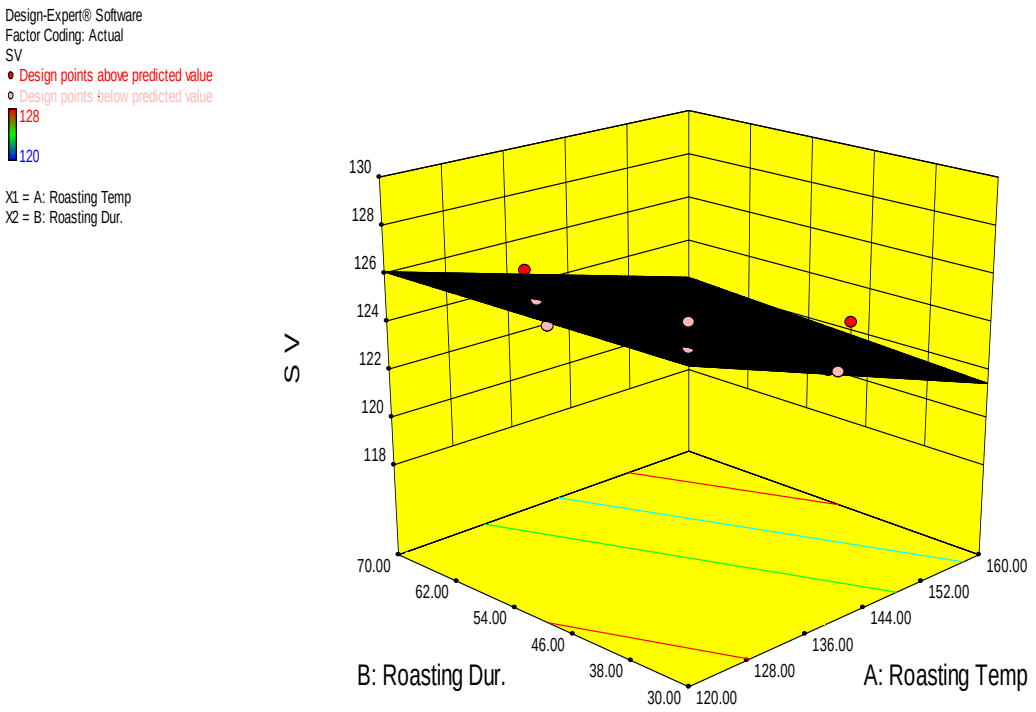

Figure 6: Plot of Saponification Value (SV) against roasting temperature and duration

Effect of roasting conditions on Kinematic Viscosity (KV)

The kinematic viscosity value obtained was in the range of 14.-15.53mm/s with a mean value of 14.97±0.11.It is apparent from Fig.7 that the kinematic viscosity of *Jatropha caucas* L. seed oil decreased as the roasting temperature and roasting duration increased. Statistically, the roasting conditions had a significant linear effect (Eq. 7) on the kinematic viscosity of *Jatropha caucas* L seed oil with 95% level of confidence. The coefficient of determination R^2 was 0.91,which suggested that the model had a good fit. The kinematic viscosity at all levels were lower than the international standard value of 39 mm/s for biodiesel oil and higher than the ranges for the conventional fossil fuel, which is 2-4.5mm/s at 40^{o}C (Felix and Clement, 2011). A very high kinematic viscosity indicates poor oil quality. It hinders the ability of the oil injector to spray oil properly and also to obtain a mist. It also prevents proper feeding of the oil into the combustion chamber of engines thereby hindering complete combustion. The viscosity of Jatropha oil can be lowered by blending it with conventional diesel or by using it as trans-esterified oil (biodiesel) (Felix and Clement, 2011). The Kinematic viscosity obtained from all combinations had good biomass attributes.

$$KV = +14.97 - 0.52X_1 - 0.37X_2 \quad (5)$$

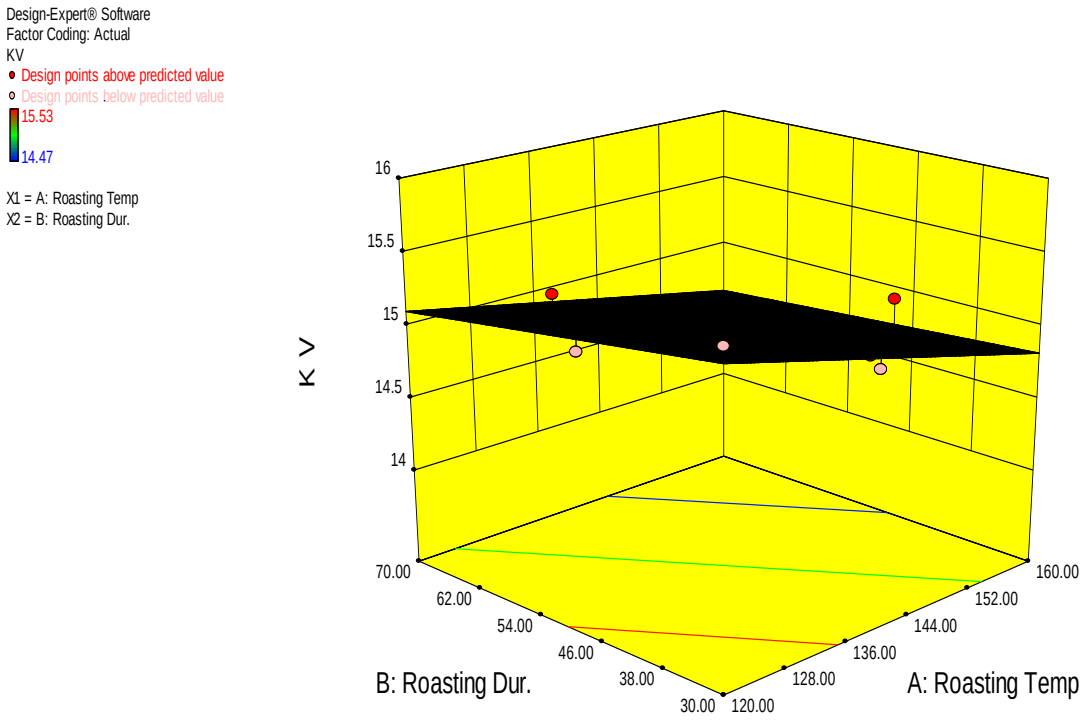

Fig.7; Plot of Kinematic Viscosity (KV) against roasting temperature and duration

Optimization of the processing conditions

An overlay plot of the seven responses (Figure 8) was carried out to determine the specific roasting temperature and duration that would give optimum oil yield and better quality. Two possible optimum solutions were found with desirability ranging from 0.609 to 0.623. Roasting at 148.18^{o}C for 37.34 mins was preferred and it gave optimum oil yield (39.0%) and good quality attributes (FFA=1.96%, SV=123.27mg/ml, PV=5.97meq/kg, IV = 81.83g /100g, KV = 14.99mm^2/s and SG = 0.916g/ml).

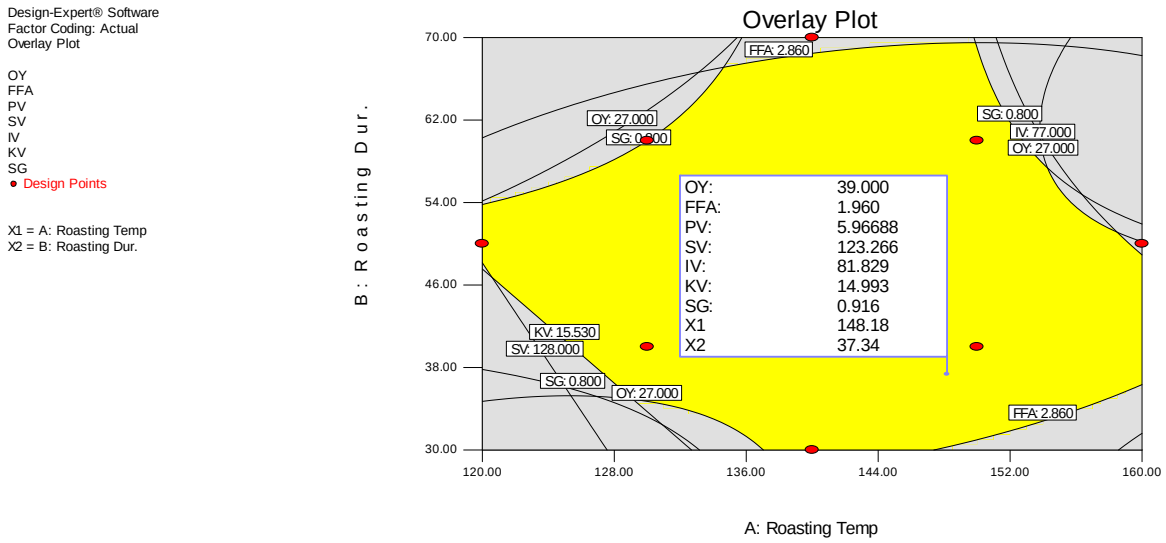

Figure 8: Overlay plot of the seven response plots

CONCLUSIONS

The results obtained in this study revealed that:

1. Roasting duration and temperature combinations influenced both oil yield and biomass quality of oil extracted from jatropha seeds significantly at 95% confidence level.
2. Predictive models were developed for jatropha seeds Oil Yield (OY), Free Fatty Acid (FFA), Saponification Value (SV), Peroxide Value (PV), Iodine Value (IV), Kinematic Viscosity (KV) and Specific Gravity (SG), as influenced by roasting temperature and duration, and the response models had good fits.
3. Two possible optimum solutions were found with desirability ranging from 0.609 to 0.623.The best of the two conditions was roasting at 148.18^{o}C for 37.34 mins,which gave optimum oil yield (39.0%) and good quality attributes (FFA=1.96%, SV=123.27mg/ml, PV=5.97meq/kg, IV = 81.83g /100g, KV = 14.99mm^{2}/s and SG = 0.916g/ml). This treatment has desirability of 0.623, a close value to 1.
4. The optimum condition produced a relatively high oil yield and good storage stability.

5. The oil obtained at this level might be used as biokerosine in jatropha oil stove or processed into biodiesel with minute or tolerable emlusion problem.
6. Further study should be carried out on the design and construction of combined roaster-expeller with temperature regulator and timer for jatropha oil production.

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