Effect of Temperature on Oil Extraction of *Jatropha curcas* L. Kernel.

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ABSTRACT

This study investigated the effect of five different extraction temperatures on oil yield and quality of *Jatropha curcas* l. kernel in order to determine the optimum temperature for maximum oil yield and best oil characteristics. Oil was extracted from *Jatropha curcas* l. kernel by sohxlet extractor using normal hexane as solvent and at five temperatures (40, 50, 60, 70, and 80^{o}C). Based on physico-chemical analysis, the oil was characterized.

Oil yield, ash content, acid values, oleic acid values, iodine values, kinematic viscosity, density and hydroxyl values were measured at each temperature level. A 5×8 factorial in CRD experimental design with a total of 600 observations (5 temperature levels×8 levels of oil properties ×15 replications) was conducted. Data were analyzed using descriptive and inferential statistics with SPSS and Excel packages. The results show that temperature has significant effect on all the oil properties tested.

Oil yield was 33.35% by weight, at 40^{o}C, and it increased to 44.41% by weight at 60^{o}C. Thereafter oil yield fell to 41.55% by weight as extracting temperature rose to 80^{o}C. Ash content values in the oil were 0.0017% by weight, 0.014% by weight, 0.019% by weight, 0.0346% by weight and 0.05% by weight at extraction temperatures of 40, 50**,** 60**,** 70, and 80^{o}C, respectively. At a temperature of 40^{o}C the acid value of the oil was 3.11% by weight and increased by about 11.6%, 13.30%, 27.33% and 43.20% as extraction temperature rose to 50**,** 60**,** 70, and 80^{o}C, respectively. Temperature had significant effect on iodine value. Increase in temperature from 40^{o}C to 80^{o}C decreased iodine value from 3.05g/100g to 0.76g/100g. Kinematic viscosity was 41.62Cst at 40^{o}C, 42.43Cst at 50^{o}C, 43.30 at 60^{o}C, 45.57 at 70^{o}C, and 47.83 at 80^{o}C. The oil density and hydroxyl values were 0.91g/cm^3 and 0.16gmole, 0.90g/cm^3 and 0.11gmole, 0.90g/cm^3 and 0.069gmole, 0.91g/cm^3 and 0.066gmole and 0.91g/cm^3 and 0.078gmole at extraction temperatures of 40, 50**,** 60**,** 70, and 80^{o}C, respectively.

The value of oil yield at 70^{o}C is statistically the same with that at 60^{o}C. At the same temperatures most of the oil properties tested fell within international acceptable standard limit. Therefore it is advised that, contrary to general practice (that is extracting oil at temperature of / less than 40^{o}C normal atmospheric temperature), it is best to extract *Jatropha curcas* l. kernel oil at temperatures between 60 and 70^{o}C.

(Keywords: *Jatropha curcas* l., Euphorbiaceous, Sohxlet Extractor, oil yield, ash content, acid values, oleic acid values, iodine values, kinematic viscosity, density, hydroxyl, optimum temperature, biofuel, oil degradation)

INTRODUCTION

Jatropha plant, as it's generally called, belongs to the Euphorbiaceous family. The Euphorbiaceae family comprises approximately 8,000 species, belonging to over 321 genera. Jatropha is a genus of approximately 175 shrubs and trees. It also belongs to the tribe Joannesieae of Crotonoideae in the same family and contains over 170 species. One species *J. villosa*, is of Indian origin. *J. afrocurcas* and *J. macrophylla* are of East African origin, where as all other species are natives of America.

Jatropha plants are well known and cultivated throughout the tropics as ornamental plant. Its other names are Barbados nut, Physics nut, Tuba, Taua taua, Saboo dam, Jarak, Awla and Pourghere plant. The botanical name is *Jatropha curcas* L.

Jatropha curcas L., is generally believed to have originated from Central America and Mexico. It was spread as a valuable hedge plant to Africa

and Asia by Portuguese traders via Cape Verde Island (Gramage and Ahmed, 1988). *Jatropha curcas* L. is a draught-resistant perennial, growing well in marginal/poor soil. It is easy to establish, grow relatively quickly and lives, producing seeds for 50 years (CJP, 2007).

This highly draught-resistant species is adapted to arid and semi-arid conditions. The current distribution shows that the introduction has been most successful in the drier regions of the tropics with annual rainfall of 300-1000mm. It occurs mainly at lower altitudes (0-500meters) in areas with average annual temperature well above 20^{o}C but can grow at higher altitudes and tolerate slight frost, (CJP, 2007). *Jatropha curcas* L. is found to grow in many parts of Nigeria, rugged in nature and can survive with minimum impulse and easy to propagate. It grows wild in many areas of India and is even well adapted to infertile soil with good aeration.

Jatropha curcas L. has a wide range of uses and very beneficial to both human and industry. *Jatropha curcas* L. sap has proven effective in accelerating wound healing (Gubitz et al., 1999). The leaves are reported to be used as a remedy for malaria and high fever (Gubitz et al., 1999; Henning,1997) extracts from this plant have been show to have anti malaria tumor activity (Juan et. al., 2003) and the seeds are effective in the treatment of constipation.

Jatropha plant is often used as anti-erosion measures. It can also be used for soil enrichment manure, ornamental plant, and raw material for dye and pesticides, potential feedstock and more importantly as an alternative for bio diesel production. *Jatropha curcas* L. is an oil bearing seed. Among all the oil bearing crops *Jatropha curcas* L. plant has emerged the most promising in biofuel production.

Climate change, energy security and rural development are factors drawing much of the research and development in these areas. Jatropha has proven to be an inexpensive feedstock in biodiesel production that can be grown on a marginal agricultural land thus displacing much use of conventional fossil fuel. Jatropha diesel is a valuable renewable energy that can be used directly in any existing unmodified diesel engine.

Jatropha curcas L. plant has emerged as the focal point for the bio-fuel industry with rapid research and developments flowing into its cultivation, processing, oil extraction and conversion into biodiesel. The key to the future of biodiesel is finding inexpensive feedstocks that can be grown by farmers on marginal agricultural land and Jatropha is one of few plants that hold a great deal of promise. Jatropha proves to be a promising bio-fuel feed stock and could emerge as a major alternative to diesel thus reducing our dependence on oil imports and saving the precious foreign exchange besides providing the much needed energy security. Jatropha stacks up nicely compared with other feedstocks, as soybeans and rapeseed have a relatively low oil yield compared with Jatropha. However the oil yield still needs to be further improved by optimizing the extraction temperature.

Several methods can be used to obtain *Jatropha curcas* kernel oil. Methods like supercritical fluid extraction, mechanical method using either screw or hydraulic press and solvent extraction have been used. Before now mechanical methods have been widely used in extracting Jatropha kernel oil at normal atmospheric temperature. However oil produced with this method is usually low grade with poor quality and yield poor quality fuel.

The use of supercritical fluids, produce oil of superior quality but the investment cost is very high. Extraction using solvent is cheaper compared with the supercritical fluid extraction method and of better quality compared with the mechanical method.

Much of the work done in literature on oil extraction from *Jatropha curcas* L. kernel and other oil-bearing seeds were done in temperate regions at temperature less than 40^{0}C (atmospheric temperature). As a result of this about 10% of *Jatropha curcas* L. kernel oils are lost in the cake as well as the degradation of oil properties which jeopardizes biofuel efficiency. Hence considering 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 the need to explore the possibility of optimizing Jatropha oil yield by extracting at temperature range in other to ascertain optimum condition (temperature) for best oil yield in terms of quality and quantity.

MATERIALS AND METHODS

Sample

The fruits of *Jatropha curcas L*. are yellow at fruit stage (that is the mature stage for oil extraction) as shown in Figure 1. These were sourced from Ede Oballa, Nsukka in Enugu state, Nigeria. A day following the harvest, the hulls were taken off manually. Thereafter, the nuts (black) were cracked using pliers on the same day (Figure 2).

Figure 1: Mature *Jatropha curcas* L. Fruit Ready for Harvest.

Figure 2: (A) The Shell; (B) Kernel (White), and (C) Nut (Black) of *Jatropha curcas* l. after Cracking and Separation

The kernel (white) was dried to a save moisture content and maintained at a normal atmospheric temperature of 37^{o}C and stored in a polythene bag. About 160g sample of the Jatropha kernel was taken from the bag, weighed, dried in a hot air oven at five temperature levels (40, 50, 60, 70, and 80^{o}C) and ground manually using a mortar and pestle. After that, the sample meal (Figure 3) was dried in a hot air oven at same five temperature levels (40, 50, 60, 70, and 80^{o}C). The sample meal was removed from the oven when temperature was about 2-3^{o}C above set value. The drying was done in batches but from the same variety and field, oil was extracted by sohxlet extractor using normal hexane and oil properties analyzed.

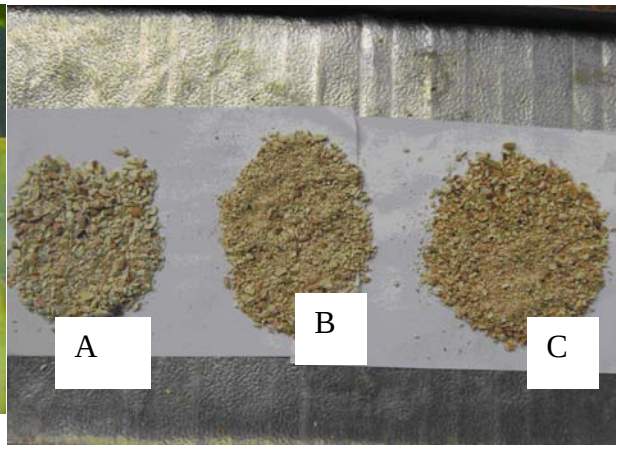

Figure 3: *Jatropha curcas* l. Kernel Meal before (A), During (B) and After (C) Oil Extraction at the Five Temperature Levels.

Jatropha curcas L. Kernel Oil Extraction

About 160g of grounded *Jatropha curcas* L. kernel were subjected to solvent extraction using n-hexane according to AOCS (2001). The extraction was continued for 4 hours. In this process, about 1500cm^3 of normal hexane was introduced into a 2000cm^3 quick fit round bottom flask (Figure 4).

The grounded jatropha kernel was introduced into the soxhlet extractor. Before this was done, a glass wool or cotton wool was used to block the inner capillary of the extractor. This prevented the sample meal or the oil from being sucked into the flask. With the condenser introduced, a rubber hose/ pipe was connected to a water tap through the inlet while another hose was connected to a reservoir through an outlet. After this, the heating mantle was switched on and the temperature regulated to 69^{o}C which was about the boiling point of normal hexane. Then the solvent will boil, vaporize, condense and fall back into the flask through the lower capillary with some quantity of oil and extracting solvent. This process continued

and was stopped when the inner capillary became colorless- indicating that oil has been completely extracted from the kernel meal. There after a rotary evaporator was used to separate the oil from the solvent under reduced pressure of about 760mmHg.

Determination of *Jatropha curcas* L. Kernel Oil Properties

The extracted oil properties of the heat treated jatropha curcas l kernel that were analyzed as shown below.

Determination of oil yield: The oil yield (OY % by wt) was calculated by Equation 1.

$$OY = 100\left(\frac{M_O}{M_K}\right) \quad \textbf{(1)}$$

Where M_o is mass of oil in grams, M_k is mass of kernel in grams.

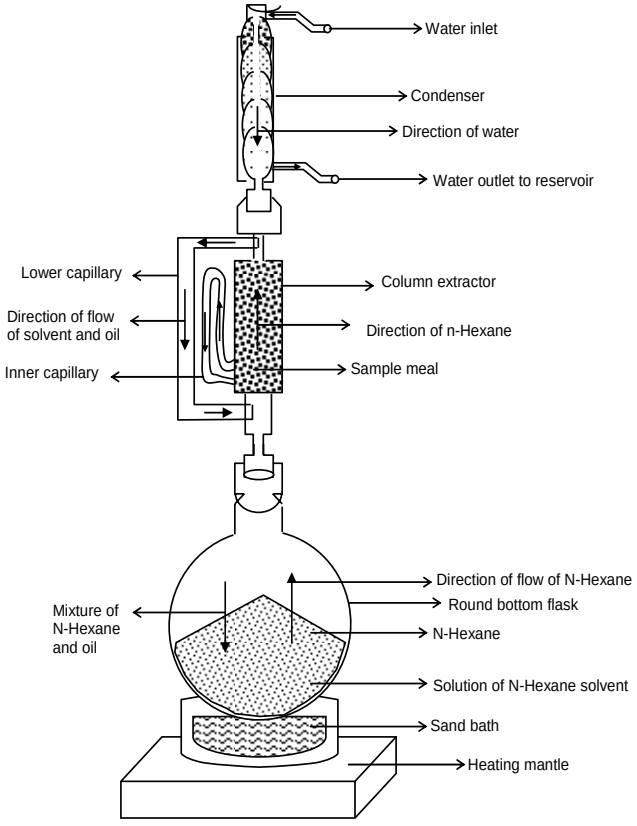

Figure 4: A Sohxlet Extractor.

Determination of Oleic Acid Value: The oil obtained was assessed by AOCS (1997) for oleic acid content (OA, % by wt), using Potassium Hydroxide and Phenolphthalein as indicator. The oleic acid value was then calculated using Equation 2.

$$OA = \frac{V_{KOH} \times N_{KOH} \times 56.1}{M_O} \quad \textbf{(2)}$$

where V is volume of KOH in ml, N is concentration in (normality), M_o is mass of oil in grams.

The constant 56.1 was derived from the molar mass of Potassium Hydroxide to obtain the respective acid in % by weight.

Determination of Acid Value: The acid value was calculated from Equation 3.

$$AV = 2(OA) \quad \textbf{(3)}$$

Determination of Ash Content Value: The ash content was evaluated by burning about 6g of the oil in a platinum bowl, (25mm deep, 75mm diameter at the top and 65mm at the base) with the normal heat until the sample became dark. After that the sample was burnt in an oven with no ventilation at 750^{o}C for 10 minutes. The bowl with the sample was then kept in a dessicator to cool down and then weighed with an electronic balance (Mettler Toledo AX 205, Switzerland, resolutiopn 0.01mg). The ash content in % was calculated with equation 4 which was developed by AOAC (2006).

$$AC = 100\left(\frac{M_A}{M_O}\right) \quad \textbf{(4)}$$

where M_A is mass of ash in grams, M_o is mass of oil in grams.

Determination of Oil Kinematic Viscosity: The viscosity of the oil was measured using a kinematic viscometer (Model K21590, Koehler Instrument Company, incorporated, NY, USA). The extracted oil, viscometer and water were conditioned at a temperature of 20^{o}C in water bath. Water volume of about 9cm^3 was poured into the viscometer and drawn above the upper mark using suction. As the water drops in the tube, the time at which the meniscus passes the

lower mark was noted using a stopwatch. Then oil sample added into the viscometer, the time for the passage of the oil between meniscus was determined as above. The kinematic viscosity (centistokes) was then calculated using equation 5 (AOAC, 2006).

$$\text{Kinematic Viscosity(Cst)} = \frac{\text{Flow time of sample} \times 1.0038}{\text{Flow time of water}} \quad \textbf{(5)}$$

where oil time flow and water time flow are all in seconds.

Determination of Iodine Value: Iodine value was determined by AOAC (2006) method. In this process, about 0.5g sample of the Jatropha oil was weighed, 10cm^3 ethanol and 5cm^3 wij's solution added. Thereafter about 10cm^3 of 10% potassium iodide solution was added and shaken vigorously. This was to ensure thorough mixing of the solution. Then about 2cm^3 of 1% starch solution was added and stirred as well. The entire solution was titrated with about 0.1N sodium thiosulphate (vi). Iodine value was calculated using equation 6.

$$\text{Iodine Value} = \frac{(\text{B - S}) \times N \times 0.1269 \times 100}{W_S} \quad \textbf{(6)}$$

where B is the blank value, S is the titre value, N is Normality and W_S is the weight of oil sample.

Determination of Oil Density: A clean and dry density bottle was weighed on a chemical balance with its stopper. Then filled completely with water, the excess wiped with a cloth and reweighed. The bottle content was emptied, the inside dried, filled with the Jatropha oil and weighed. The relative density of the oil was calculated using equation 7 (AOAC, 2006).

$$\text{Relative density of oil} = \frac{\textit{Mass} \text{ of oil}}{\textit{Mass} \text{ of equal volume of water}} \quad \textbf{(7)}$$

But relative density of the oil is equal to the density of the oil (g/cm^3).

Determination of Hydroxyl Value: Two thoroughly cleaned and dried conical flask were marked A and B. about 1g of extracted *Jatropha curcas* L. kernel oil was added into flask B while flask A was left empty. About 10cm^3 mixture of 20% acetic anhydride in pyridine was added into the flasks, water condenser attached and heated for 30 minutes on a boiling water bath. The flasks were removed from the bath and kept for 20 minutes then distilled water was added into them and stirred vigorously. After cooling, the solution in the flasks were titrated with 0.5N potassium hydroxide (KOH) using phenolphthailein. Hydroxyl value was calculated using Equation 8.

$$\textit{Titre} \text{ value} = \text{Titre value of blank - Titre value of oil sample} \quad \textbf{(8)}$$

where N is normality.

Statistical Analyses of Data

A 5x8 factorial in CRD experimental design with a total of 600 observations (5 temperature level x 8 levels of oil properties ×15 replications) was conducted. Data were analyzed using descriptive and inferential statistics with SPSS and Excel packages. Analysis of variance (ANOVA), F-test, Duncan multiple range test, LSD and regression analyses were carried out.

RESULTS AND DISCUSSION

Summary result of the effect of temperature on oil yield and other oil properties using Duncan Multiple Range Test format is presented in Table 1.

Oil yield: Analysis of variance showed that effect of temperature on Jatropha curcas l. kernel oil yield and some other oil properties were significant at 5% probability level. At 40^{o}C the mean oil yield was 33.35% by weight, 38.88% by weight at 50^{o}C, 44.40% by weight at 60^{o}C, 43.83% by weight at 70^{o}C and 41.50% by weight at 80^{o}C.

Jatropha curcas L. kernel oil yield was maximum at 60^{o}C and thereafter decreased to 41.50% by weight when extraction temperature rose to 80^{o}C. Therefore 60^{o}C is the optimum extraction temperature of *Jatropha curcas* L. kernel for maximum oil yield. Statistical analysis of variance showed that effect of temperature on jatropha curcas L kernel oil yield was significant at 5% probability level. At 5% probability level the effect of temperature on oil yield was highly significant.

When treatment means were analyzed statistically using Duncam Multiple Range Test, significant differences exist among these means

(Table 1) except for oil yield at 60°C and 70°C which are statistically the same. Statistically, there is no difference between oil yield at 60°C and yield at 70°C. Oil yield at 40°C, 50°C and 80°C are all significantly different from each other. They are also significantly different from yield at 60°C and 70°C. But oil yield at extraction temperature of 60°C and 70°C are statistically the same.

Ash Content: Ash content value of *Jatropha caucas l. kernel* oil increased with extraction temperature. Ash content was maximum at 80°C (0.05% by weight), minimum at 40°C (0.00167% by weight), a value of 0.014% by weight, 0.0187% by weight and 0.034% by weight at temperatures of 50°C, 60°C and 70°C respectively. All the value for ash content at temperature levels of 40°C, 50°C and 60°C fell within international acceptable standard level of 0.02% by wt. for ash in biodiesel oil. This suggests a better usage quality. Increase in ash level in *Jatropha caucas l. kernel* as temperature increased could be attributed to oil degradation. Statistically, extraction temperature of *Jatropha caucas l. kernel* oil has significant effect on ash content of the oil. Analyzing the results by Duncan multiple range test, significant differences exist among the ash content values at the various extraction temperatures. Ash content values at 40°C, 70°C and 80°C are all significantly different from each other and also different from the values at 50°C and 60°C. No differences were observed between ash content values at 50°C and at 60°C (Table 1).

Acid Value: All the acid values at all the five temperature levels were above internationally acceptable standards. However at extraction temperatures of 40, 50, 60, and 70°C, the oleic acid values fell within international acceptable ranges. The international acceptable ranges of acid in biodiesel oil is between 0-2% by weight. Acid values of *Jatropha caucas l. kernel* oil increased with temperature. The values were 3.11% by weight, 3.47% by weight, 3.58% by weight, 3.96% by weight and 4.45% by weight at temperature levels of 40, 50, 60, 70, and 80°C respectively. This could be attributed to oil degradation. Oleic acid values in the oil were 1.56% by weight, 1.74% by weight, 1.80% by weight, 1.98% by weight and 2.22% by weight at same temperature levels of 40, 50, 60, 70, and 80°C, respectively.

Statistically temperature has significant effect on acid value and oleic acid values of *Jatropha caucas L. kernel* oil. As shown in Table 1, acid value and oleic acid value of Jatropha oil at the five extraction temperature levels of 40, 50, 60, 70, and 80°C were all significantly different from each other. Therefore *Jatropha caucas L. kernel* oil has better quality for biodiesel production at extraction temperature below 70°C. Higher oleic acid value indicates higher free fatty acid, which can cause serious abrasion and corrosion on metal parts. This is a deleterious property of oil for biofuel production.

Table 1: Summary Results of Oil Yield and Oil Properties of *Jatropha curcas* L. Kernel at Five Different Temperatures using Duncan Multiple Range Test Format.

Oil Properties	**Temperature (°C)**				
	40°C	**50°C**	**60°C**	**70°C**	**80°C**
Oil yield, % by wt	33.35_a	38.88_b	44.41_d	43.83_d	41.56_c
Ash content, % by wt	0.0017_a	0.014_b	0.019_b	0.035_c	0.05_d
Acid value, % by wt	3.11_a	3.47_b	3.58_c	3.96_d	4.45_e
Oleic acid value, % by wt	1.56_a	1.74_b	1.80_c	1.98_d	2.22_e
Kinematic viscosity, Cst	41.62_a	42.43_b	43.30_c	45.57_d	47.83_e
Iodine value, g/100g	3.05_a	1.95_b	1.28_c	0.99_d	0.76_e
Hydroxyl value, gmole	0.16_d	0.11_c	0.069_{ae}	0.066_a	0.078_{be}
Oil density, g/cm^3	0.91_b	0.90_a	0.90_a	0.91_b	0.91_c

Kinematic Viscosity: The kinematic viscosity at the five temperature levels were higher than the international standard value of 39 Centistoke (Cst) for biodiesel oil. The values were also higher than the ranges for the conventional fossil fuel, which is 2-4.5Cst at 40^{o}C (Mittelbach and Remschmidt, 2004). Oil extraction at 40^{o}C gave kinematic viscosity of 41.62Cst. A value of 42.43 Cst was calculated at 50^{o}C.

The kinematic viscosity value of *Jatropha caucas L. kernel* oil was 43.30Cst at extraction temperature of 60^{o}C. It was 45.57Cst at 70^{o}C and 47.84Cst at 80^{o}C. It is apparent from Table1 that the kinematic viscosity of *Jatropha caucas L. kernel* oil increased as extraction temperature increased. Statistically, temperature had significant effect on the kinematic viscosity of *Jatropha caucas L kernel* oil with 95% level of confidence. The mean values (41.62Cst, 42.43Cst, 43.29Cst, 45.57Cst and 47.83Cst) of kinematic viscosity of *Jatropha caucas l kernel* oil at the different temperature levels (40, 50, 60, 70, and 80^{o}C) respectively, were all significant at 5% level of probability.

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. This problem can be avoided. The viscosity of Jatropha oil can be lowered by blending it with conventional diesel or by using it as transesterified oil (biodiesel). *Jatropha curcas* L. kernel oil should be extracted at a temperature of about 60^{o}C in other to obtain oil with fairly lower kinematic viscosity.

Oil Density: The density of Jatropha *kernel* oil is lower than the density of water. Jatropha *kernel* oil density was highest with a value of 0.91g/cm^{3} at extraction temperatures of 40, 70, and 80^{o}C. The value was lowest (0.90g/cm^{3}) at extraction temperature of 50^{o}C and 60^{o}C. At 5% probability level temperature significantly affected *Jatropha caucas L.,* kernel oil density. Jatropha oil density at 60^{o}C is the same with the value of oil density at 50^{o}C. Oil density at 70^{o}C is the same with oil density value at 40^{o}C. From Table 1, oil density value at extraction temperature of 80^{o}C is statistically different from the values at 40, 50, 60, and 70^{o}C.

Iodine Value: Iodine values in the oil were 3.05g/100g, 1.95g/100g, 1.28g/100g, 0.99g/100g and 0.76g/100g at extraction temperatures of 40, 50, 60, 70, and 80^{o}C, respectively. Iodine value decreased with temperature. The iodine values at all the five temperature levels were far below the allowable international standard level (100-200g/100g) of iodine in biodiesel oil products. Temperature has significant effect on the iodine value of the kernel oil. At the five temperature levels (40, 50, 60, 70, and 80^{o}C), the values of iodine in *Jatropha caucas* L. kernel oil were all significantly different from each other as shown in Table 1.

Hydroxyl Values: The hydroxyl value was 0.16g mole at 60^{o}C, 0.066g mole at 70^{o}C and 0.078g mole at 80^{o}C. Hydroxyl value of 0.066g mole at 70^{o}C and 0.069g mole at 60^{o}C are statistically the same. The value of 0.069g mole at 60^{o}C and 0.078g mole at 80^{o}C are also statistically the same. The values of 0.1587g mole at 40^{o}C and 0.11g mole at 50^{o}C are statistically different from each other and also different from hydroxyl values at 60, 70, and 80^{o}C respectively (Table 1).

CONCLUSIONS AND RECOMMENDATION

The following conclusions can be drawn from this work:

1. Temperature had significant effect on *Jatropha curcas* L. kernel oil yield. As extraction temperature increased, oil yield rose from 33.35% by weight to a maximum value of 44.41% by weight at 60^{o}C. Further increase in extraction temperature to 80^{o}C reduced oil yield to 41.5% by weight. Optimum temperature conditions for maximum Jatropha oil yield and quality was obtained at 60^{o}C.

2. Ash content increased as temperature increased. Temperature level of 60^{o}C is the most desirable for high quality *Jatropha curcas* oil with ash content within international standard limit. Acid value increased as temperature increased.

3. The viscosity values of *Jatropha curcas* L. kernel oil were extremely high. Much higher than the value for fossil fuel. However the extracted oil can be used as transesterified oil (biodiesel) or as blend with conventional diesel.

4. *Jatropha curcas* L. kernel oil is less dense than water.

5. Iodine values decreased as extraction

temperature increased.

6. Hydroxyl value was 0.16gmole at 40^{o}C, 0.11gmole at 50^{o}C, 0.069gmole at 60^{o}C, 0.066gmole at 70^{o}C and 0.078gmole at 80^{o}C.

7. It is recommended that for future studies, more temperature levels should be used. Normal hexane was used as the solvent for this work. Other solvents should be used and the results compared.

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SUGGESTED CITATION

Asoiro, F.U. and C.O. Akubuo. 2011. "Effect of Temperature on Oil Extraction of *Jatropha curcas* L. Kernel". *Pacific Journal of Science and Technology*. 12(2):456-463.

Pacific Journal of Science and Technology