

## Full Length Research Paper

# Effect of roasting temperature on the physicochemical properties of *Jatropha curcas* Kernel oil extracted with cold hexane and hot water

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The industrial application of a vegetable oil is determined by the oil properties. This work was undertaken to alter the properties of *Jatropha curcas* Kernel oil and possibly find new applications for it. The seed kernels were roasted to different temperatures (140 to 230°C) and the physicochemical properties of the cold hexane and hot water extracted oils were studied. The oil yield, saponification, iodine, acid and peroxide values were affected by roasting temperature and method of extraction and these showed significant difference ( $p < 0.05$ ). The calculated fuel properties: cetane number and calorific value were improved upon roasting, with cold hexane extracted oils yielding better results. Roasting improved the properties of the oil as diesel substitute but its suitability for use in paints and surface coatings formulation was reduced. The hot water extracted oil showed improved properties for soap production.

**Key words:** *Jatropha curcas*, roasting temperature, oil extraction, physicochemical properties.

## INTRODUCTION

Vegetable oils continue to play an important role in the manufacturing industry, because of their suitability for application in food, lubricants, hydraulic fluids, fuel, in production of soap and shampoos, alkyd resin polishes, varnishes, paints and other surface coatings (Demirbas, 2009; Akbar et al., 2009; Akintayo, 2004; Alamperese et al., 2009; Kyari, 2008; Knothe et al., 2004). They can be sourced from seeds (such as soybean, canola), fruit coat (palm and olive), and seeds kernel (coconut, sunflower, *Jatropha*, palm kernel, etc). Oil content in seeds is

usually within 10 to 52% (Zapata et al., 2012; Eromosele and Pascal, 2003; Schinas et al., 2009; Kesari et al., 2010; Elleuch et al., 2007; Nehdi et al., 2010).

The application of oil is determined by the oil property and this has been reported to be a function of oil composition, most especially the fatty acid profile/composition (Aluyor et al., 2009; Erhan, 2005). A lot of research has been directed to modify oils to meet specific applications: as biodiesel (Lim and Lee, 2011; Sarin et al., 2010; Belewu et al., 2010, Rashid et al., 2010;

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Yusup and Khan, 2010), as lubricants and hydraulic fluids (Erhan and Asadauskas, 2000; Erhan, 2005; Adhvaryu et al., 2004) and for paint application (Nakayama, 1998; Ahmad et al., 2005; Dutta et al., 2009).

*Jatropha curcas* is a large shrub growing up to a height of 5 to 7 m (Achten et al., 2008). This drought-resistance plant belongs to the Euphorbiaceae family and has native distributional range in south and central America but is now abundantly found in tropical and subtropical regions throughout Africa and Asia (Jongschaap et al., 2007; Achten et al., 2008). Its seeds are called by names like Barbados nut, Physics nut, Tuba, Taua taua, Saboo dam, Jarak, Awla and Pourghere plant (Asoiro and Akubuo, 2011).

The seeds make up approximately 70% of the total weight of the fruit and comprise of 41% shell and 59% kernel (Joshi et al., 2011b; Jongschaap et al., 2007). The kernel has up to 66.4% oil (Adebowale and Adedire, 2006) and moisture content of about 5% (Jongschaap et al., 2007). The fatty acid composition of *J. curcas* oil showed that oleic acid (44.70 to 43.32%) is the most abundant followed by linoleic acid (36.70 to 32.80%) and palmitic acid (14.20 to 13.19%) (Abdullah et al., 2013; Akbar et al., 2009). *J. curcas* is not edible and the seed cake is not readily suitable for use as animal feed because it contains some toxicants like phorbol esters (Joshi et al., 2011b), curcins, phytates and protease inhibitors (He et al., 2011; Achten et al., 2008).

## MATERIALS AND METHODS

### Seeds collection

The *J. curcas* seeds used for this work were collected from trees at different locations in South Western Nigeria. Matured and dried fruits were collected from the plants and the coats and shells were removed manually. About 4 kg of kernels were recovered (Figure S1). This was oven dried at 105°C for 4 h in batches to remove the moisture. The moisture was calculated as the percentage loss in weight between the fresh kernel and the dried kernel.

### Seed processing

The dried kernels were subjected to different roasting temperatures for 30 min. Three hundred grams (duplicate) were heat-treated in an oven at four different roasting temperatures: 140, 170, 200 and 230°C. The weight loss upon roasting was noted. The roasted kernels were cooled in a desiccator and pulverized using a blender and stored in black polythene bags before oil extraction.

### Oil extraction

The oil was extracted using the hot water and cold hexane extraction methods. In the hot water extraction method, 100 g of pulverized kernel was boiled in 700 ml of distilled water (1:7 w/v) for 1 h. The mixture was allowed to cool. The oil floated on the water, while the hydrated cake settled at the bottom. The oil-water mixture was decanted and centrifuged (10,000 rpm, 20°C, 10 min) using Hitachi high speed refrigerated centrifuge (Himac CR 21GII). The oil

layer was separated and mixed with excess anhydrous sodium sulphate and centrifuged again to obtain pure oil while sodium sulphate formed a lump at the base.

In the cold hexane extraction, 100 g of pulverized roasted ground kernel was transferred into 200 ml hexane (1:2 w/v) in air-tight glassware. The mixture was left for 16 days with daily mixing. After 16 days, the hexane-oil was decanted. The oil was recovered using a rotary evaporator (BUCHI rotavapor R-210) on a heating bath (BUCHI B-491). The oil recovered was then dried by treating with anhydrous sodium sulphate. The mixture was shaken vigorously and centrifuged (10,000 rpm, 10 min, 20°C) to obtain pure oil. The oil yield was calculated as percentage weight of oil to weight of the kernel flour.

## Physicochemical properties

### Acid value

Oil (1000 mg) was weighed into 250 ml conical flask and ethanol (10 ml) was added and the flask heated on a steam bath for 3 min. The content was titrated, while hot with 0.112N alcoholic potassium hydroxide using 1 ml phenolphthalein as indicator. The end point was indicated by the appearance of a pink color which persisted for about 30 s. Acid value was calculated as in Equation 1:

$$\text{Acid value} = \frac{\text{Normality of alcoholic KOH} \times \text{Volume of alcoholic KOH (mL)} \times 56.1}{\text{Weight of oil (g)}} \quad (1)$$

56.1 = KOH molecule weight.

### Iodine value

To the oil (100 mg), 10 ml of chloroform was added, followed by 10 ml of Wijs (Iodine monochloride) solution. Thereafter, 10 ml of 15% KI was added and shaken vigorously to ensure thorough mixing. This was left in the dark for 1 h with intermittent shaking. To this was added 1 ml of fresh starch solution and stirred. The resulting solution was titrated with sodium thiosulphate (0.1N). The iodine value was calculated as in Equation 2:

$$\text{Iodine value} = \frac{(\text{Blank titre} - \text{Sample titre}) (\text{mL}) \times \text{Normality of } \text{Na}_2\text{S}_2\text{O}_3 (\text{N}) \times 126.9}{\text{Weight of oil (g)}} \quad (2)$$

126.9 = gram-equivalent of iodine.

### Saponification value

Oil (500 mg) was transferred into a conical flask and 7.5 ml of 0.475N KOH solution was added. This was refluxed for 60 min and cooled before 4 drops of phenolphthalein was added. The resulting mixture was titrated with 0.482N HCl until the pink color disappeared. The same process (but without the oil) was conducted to determine blank. Saponification value was calculated as in Equation 3:

$$\text{Saponification value} = \frac{(\text{Blank titre} - \text{Sample titre}) (\text{mL}) \times \text{Normality of HCl (N)} \times 56.1}{\text{Weight of sample (g)}} \quad (3)$$

### Peroxide value

Oil (500 mg) was weighed into a 250 ml quick-fit glass stoppered conical flask and 5 ml of acetic acid-chloroform mixture (3:2 v/v)

was added and the content of the flask was swirled before it was carefully warmed on hot plate for 30 s. 0.5 ml of saturated KI solution was then introduced and the flask was stoppered and swirled for 1 min. After this time, 30 ml of distilled water was added and the mixture was shaken vigorously to liberate the iodine from the chloroform layer. The mixture was titrated with 0.1N sodium thiosulphate until the amber color lightens before 1 ml of 1% starch solution was added as indicator. Titration was continued until the blue coloration that appeared upon introduction of starch disappeared from the aqueous layer. The peroxide value was calculated as in Equation 4:

$$\text{Peroxide value} = \frac{(\text{Sample titre} - \text{Blank titre})(\text{mL}) \times \text{Normality of thiosulphate (N)} \times 1000}{\text{Weight of sample (g)}} \quad (4)$$

#### Cetane number and calorific value

The cetane number (CN) and calorific value (CV) were calculated from saponification value (SV) and iodine value (IV) using the equations used by Azam et al. (2005) Equation and Demirbas (1998) Equation 6:

$$\text{CN} = 46.3 + \left(\frac{5458}{\text{SV}}\right) - 0.225(\text{IV}) \quad (5)$$

$$\text{CV} = 49.43 - (0.041(\text{SV}) + 0.015(\text{IV})) \quad (6)$$

#### Statistical analysis

One way analysis of variance was used to compare means of values. Values are considered statistically different at  $p < 0.05$ .

## RESULTS AND DISCUSSION

### Moisture content and loss upon roasting

The loss on drying at 105°C for *Jatropha curcas* Kernel was  $6.8 \pm 0.04\%$ . This loss is attributed to moisture. Jongschaap et al. (2007) has reported a value of 5% for *Jatropha curcas* Kernels. Roasting resulted in a gradual increase in weight loss (3.10 to 4.14%) with increase in temperature from 140 to 200°C (Figure 1). There is no statistical difference ( $p = 0.78$ ) in weight loss between 140 and 230°C. This loss is attributed to thermal decomposition and loss of some volatile constituents of the kernel.

### Oil yield and appearance

Figure 2 shows that the oil yield increased with roasting temperature and was maximum at 200°C (13.7%, cold hexane and 6.3%, hot water). However, the yield declined above 200°C. This could be as a result of destruction or disruption of the oleosomes to release more oil. The decrease in oil yield observed above 200°C might be as a result of decomposition of the oil. Yields

obtained by these methods are very low compared to 66.4% obtained by soxhlet extraction (Adebowale and Adedire, 2006). This indicates a good percentage of the oil could not be extracted. The unroasted kernel oils were light yellow, while the oils from roasted kernel appeared deeper and darker (Figure S2). The deeper color at very high temperatures could suggest some seed decomposition products were extracted. The oils from cold hexane extraction appeared more viscous than those from hot water extraction.

### Acid value

The cold hexane extracted oil showed a decline in acid value from 6.45 to 2.72 mg KOH/g of oil as temperature increased from 105 to 230°C, while for hot water extracted oil it was relatively unchanged (~2.24 mg KOH/g oil at 105 to 170°C and increased at 200°C (3.98 mg KOH/g oil) and then decreased (Figure 3). The acid values showed significant difference ( $p < 0.05$ ) with method of extraction. The obtained acid values in this work 2.24 to 6.45 mg KOH/g of oil are in the range of values 0.6 to 36.46 mg KOH/g reported for *Jatropha curcas* oil (Akbar et al., 2009; Akintayo, 2004; Foidl et al., 1996; Sarin et al., 2010; Joshi et al., 2011a; Cheng-Yuan et al., 2012). Acid values of many edible oils are less than 3.0 mg KOH/g of oil (Bello et al., 2011; Akubugwo et al., 2008; Akubugwo and Ugbogwu, 2007). While edible oils may have low acid values, the decreased acid values obtained upon increased roasting temperature in this study does not necessarily imply edibility of the oil as no validation study of such was carried out. However, this could be indicative of reduced toxicity. For example, curcin, a toxicant commonly found in *J. curcas* seed, is a ribosome inactivating protein which like most proteins could be denatured or experience loss of function at high temperatures. Also, high temperature of 260°C and 3 mbar has been reported to completely degrade phorbol esters present in *J. curcas* (Makkar et al., 2009).

### Iodine value

Generally, increase in roasting temperature caused a decrease in iodine value (degree of unsaturation) (Figure 4) and these values varied significantly ( $p < 0.05$ ) with method of extraction. A possible reason for this trend is the oxidation of the fatty acids at the points of unsaturation. This decrease is more pronounced in oils extracted with cold hexane than hot water. This indicates that roasting of *Jatropha* kernels reduces the drying property of the oil and suitability for use in paints, varnishes and polishes industries. The iodine values of the unroasted oils are 100.2 mg I<sub>2</sub>/g of oil and 115.5 mg I<sub>2</sub>/g of oil for water and cold hexane extracted oils, respectively; this places them in the range of semi-drying

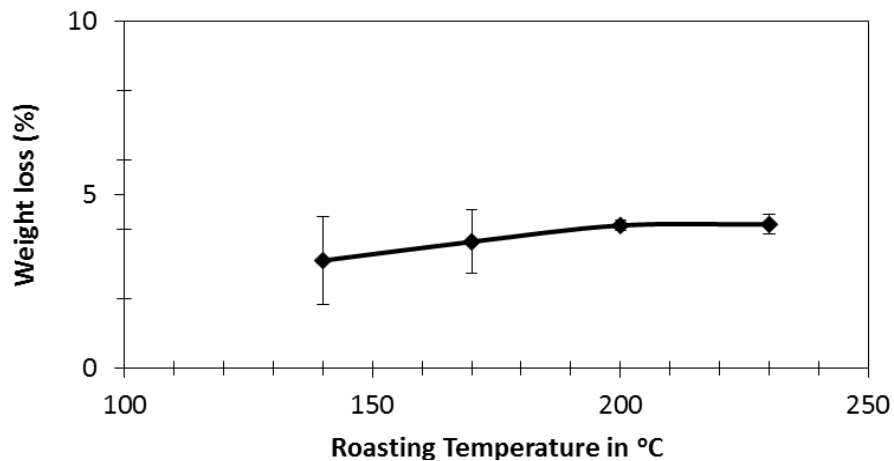


Figure 1. Weight loss upon roasting.

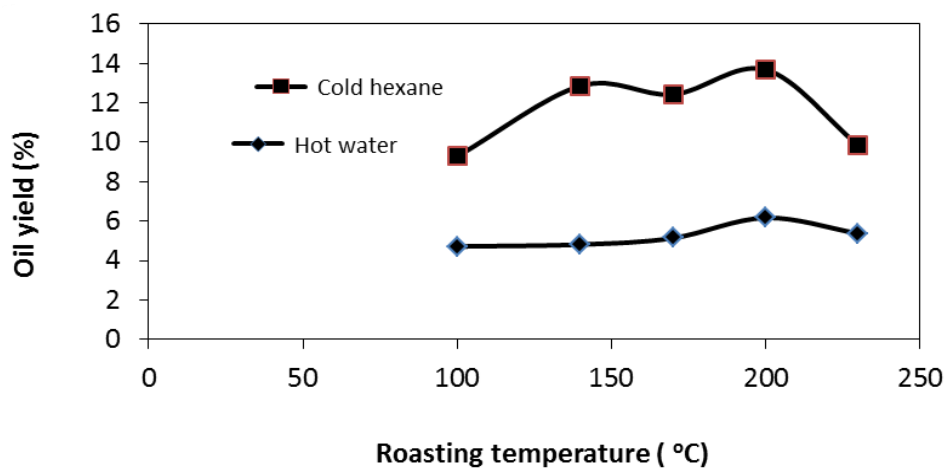


Figure 2. Oil yield variation with temperature and extraction method.

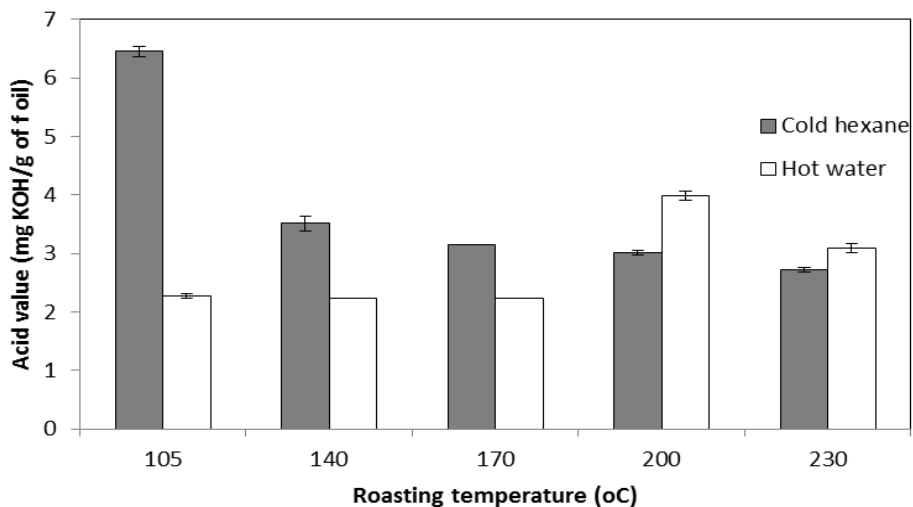


Figure 3. Variation of acid value with roasting temperature and extraction method.

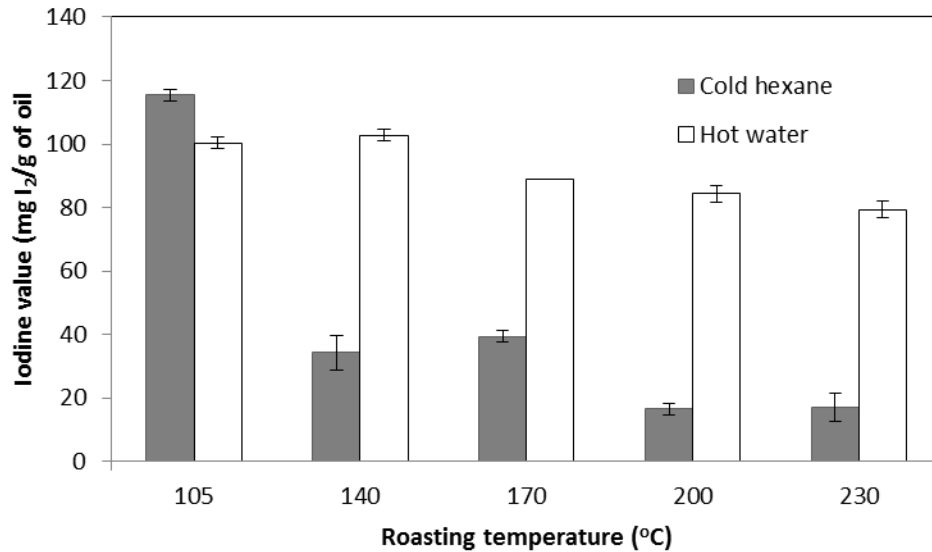


Figure 4. Iodine value variation with temperature and extraction method.

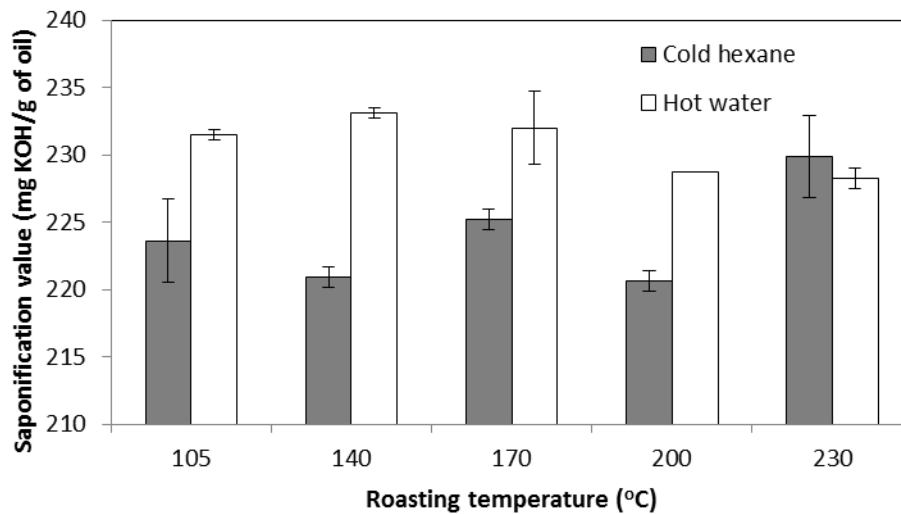


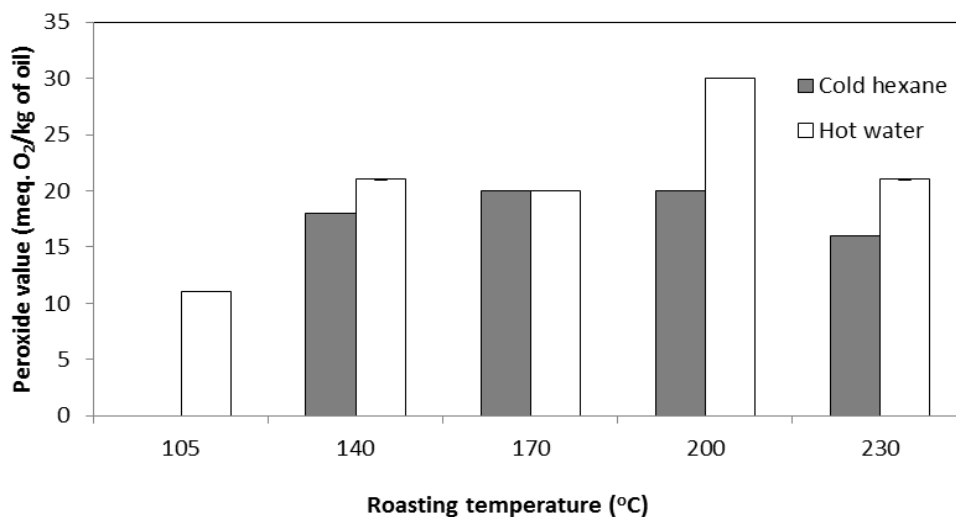
Figure 5. Saponification value variation with temperature and extraction method.

oils. These values are consistent with 92 to 106.6 mg/g reported for *J. curcas* oil in the literature (Akbar et al., 2009; Akintayo et al., 2004; Foidl et al., 1996; Sarin et al., 2010; Pramanik, 2003).

### Saponification value

High saponification value suggests presence of normal triacylglycerides (Akintayo, 2004). Water extracted oils have saponification values between 228 mg KOH/g oil to 233 mg KOH/g of oil which are significantly different ( $p < 0.05$ ) from 221 mg KOH/g of oil to 230 mg KOH/g oil

obtained for cold hexane extracted oils (Figure 5). This difference is probably as a result of hydrolysis which occurred during hot water extraction which has broken the triacylglycerides, thereby reducing the molecular weight. The obtained values in this work are higher than those reported in the literature for *J. curcas* oil which ranged between 188.2 and 198.85 mg KOH/g of oil (Joshi et al., 2011a; Akbar et al., 2009; Akintayo, 2004; Cheng-Yuan et al., 2012; Pramanik, 2003; Gopinath et al., 2010). The high saponification values in all the oils indicate that the fatty acids in the oils have low molecular weight and short chain length. These low saponification values suggest that the number of ester bonds is less



**Figure 6.** Peroxide value variation with temperature and extraction method.

than normal. All the obtained oils from the two extraction methods will find good application in the production of soaps and shampoo as a result of this. Oils from hot water extraction will however be preferred in this regard.

### Peroxide value

Peroxide value is an indication of oxidative stability. The lower the peroxide value, the better the stability of oil to oxidation. Ezech et al. (2012) reported that oils become rancid when the peroxide value ranges from 20.0 to 40.0 meq.O<sub>2</sub>/kg oil. In this study, the obtained peroxide value for cold hexane extracted oil from unroasted (105°C) kernel was an unusual value of 0.0. Generally, the peroxide values of both hot water and cold hexane extracted oils increased with increase in roasting temperature and a slight decrease was observed when roasted at 230°C (Figure 6). The peroxide values of oils extracted with hexane were significantly different ( $p < 0.05$ ) from values obtained by hot water extraction. The decrease in oxidative stability (increase in peroxide value) is as a result of reduction in the anti-oxidants present. This reduction could be as a result of thermal degradation or microstructural changes in seeds that take place at elevated temperatures, while increase in oxidative stability is generally attributed to the increase in extractability of tocopherols (or other anti-oxidants) by the thermal degradation of cellular structure (Durmaz and Gokmen, 2011). Another possible reason for the variation of oxidative stability with temperature increase is generation and accumulation of anti-oxidants activity which could develop antagonistic or synergistic effects among themselves or with other constituents in oil (Miranda et al., 2010). The obtained peroxide values in this work showed that hexane extracted oils are more

stable to oxidation/rancidity.

### Cetane number

Cetane number is an indication of ignition quality. Higher cetane value means better ignition quality (Atabani et al., 2012) that is higher cetane number in fuels will facilitate easy starting of compression ignition engines. Cetane number is affected by degree of saturation and chain length. Cold hexane extracted oils have higher cetane number (44.7 to 67.32) than hot water extracted oils (46.58 to 52.32) and cetane number increased with increase in roasting temperature as shown in Figure 7. The cetane numbers for oils extracted with hexane and hot water at 105°C were not significantly different ( $p = 0.09$ ) but the values for oils extracted from *Jatropha* kernels roasted at higher temperatures were significantly different ( $p < 0.05$ ). The obtained values in this work are higher than 38 which was reported by Pramanik (2003) for *J. curcas* oil but close to the cetane number of *J. curcas* methyl esters which ranged between 51 and 58.4 (Ong et al., 2011; Foidl et al., 1996; Qian et al., 2010; Sarin et al., 2010; Rashid et al., 2010; Gopinath et al., 2010; Kumar and Sharma, 2008). This result shows that hexane extracted oils are more saturated than hot water extracted oils and have better ignition quality. This is being supported by the peroxide value result which showed better oxidative stability for hexane extracted oils. Hexane extracted oils will find better application as diesel substitute than hot water extracted oils.

### Calorific value

The higher the heating value or calorific value, the more

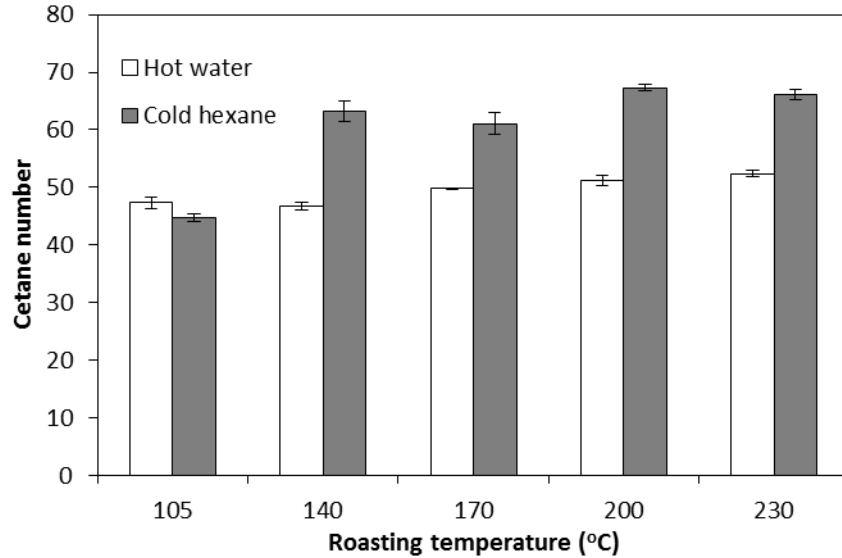


Figure 7. Cetane number variation with temperature and extraction method.

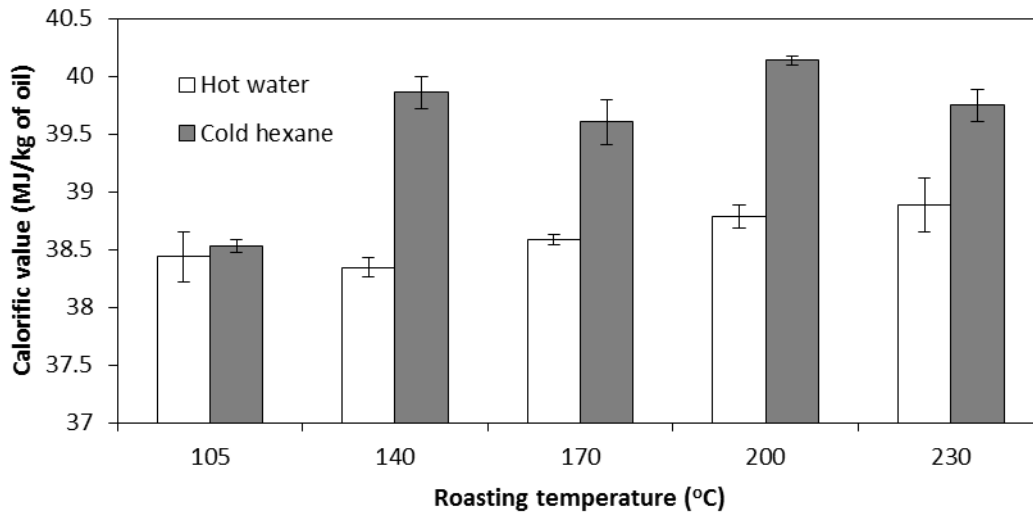


Figure 8. Variation of calorific value with temperature and extraction method.

energy released per unit mass of oil (Gopinath et al., 2010). The heating values of oils from both extraction methods increased with increase in roasting temperature (Figure 8). This suggests decrease in unsaturation as roasting temperature increased. Oils extracted from kernels using hexane will release more heat upon burning than their corresponding oils obtained using water extraction. The heating values for oils extracted by the different extraction methods from kernels roasted a 105°C did not show any significant difference ( $p = 0.62$ ); however, those extracted from samples roasted at higher temperatures differed significantly ( $p < 0.05$ ). The obtained values in this work: (38.5 to 40.1 MJ/kg for oil

extracted using cold hexane and 38.3 to 38.9 MJ/kg for hot water extracted oils) are consistent with those reported in the literature (38.2 to 39.66 MJ/kg) (Karaj et al., 2008; Pramanik, 2003).

## Conclusion

Roasting temperature has significant effect on iodine values of cold hexane extracted oils, potentially making them a more desirable feedstock in surface coating applications. Also, extraction method (hot water extraction) slightly improved the oil's potential for use in

soaps and shampoo production. Oil yield was highest in the kernels roasted at 200°C for the two extraction methods employed. Increase in roasting temperature slightly improved both ignition quality and the energy released on burning a specific mass of oil. However, the stability of *J. curcas* oil to oxidation and the degree of unsaturation of the fatty acids decreased with increase in roasting temperature as reflected in the peroxide and iodine values.

## CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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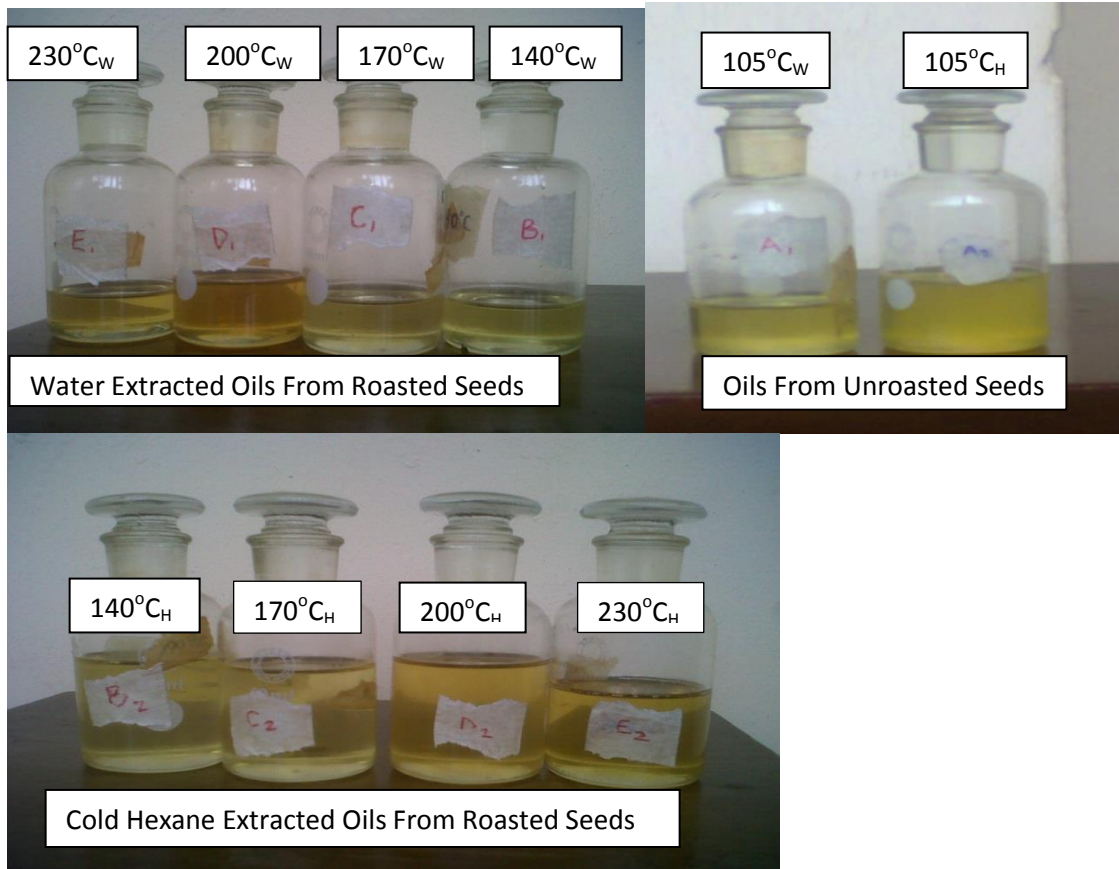


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Supplementary Figures



Figure S1. *Jatropha curcas* seeds and kernels.



**Figure S2.** Appearance of oils. 'W' and 'H' represent water and cold hexane extracted oils, respectively.