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Optimization of mechanical oil extraction from *Jatropha curcas* L. kernel using response surface method

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ABSTRACT

Extraction of oil from *Jatropha curcas* L. kernel was investigated using a lab-scale hydraulic press. A face centered composite design of experiments was employed to study and optimize the effect of applied pressure, pressing temperature and moisture content on oil recovery. A quadratic polynomial model was generated to predict oil recovery and was found to cover 98% of the range for the factors studied, namely 10–20 MPa applied pressure, 60–90 °C pressing temperature and 3–5% (w.b.) moisture content. Among the process parameters studied, pressing temperature had the most significant effect on the recovery followed by applied pressure and quadratic of moisture content. Model validation experiments show good correspondence between actual and predicted values. The optimal extraction condition for oil yield within the experimental range of the variables researched was at 19 MPa applied pressure, 90 °C pressing temperature, and 3.8% (w.b.) moisture content. At this condition, the yield of oil was predicted to be 87.8%.

1. Introduction

One of the most promising renewable and independent energy sources in rural areas is *Jatropha* oil (Kumar and Sharma, 2008; Makkar and Becker, 2009). It is non-edible oil, thus it will not impair food security issues (Pinzi et al., 2009). As it grows well on dry marginal non-agricultural land, it will not compete with land needed for food production or with nature conservation (Achten et al., 2007; Makkar and Becker, 2009; Pinzi et al., 2009). Jatropha is considered a more sustainable feedstock for energy production than any other food-related crop such as palm, rapeseed, soybean or sunflower (Achten et al., 2007; Pinzi et al., 2009).

The extraction of the oil from the seed is done in different ways. Methods used are: solvent extraction, mechanical extraction, enzymatic extraction and aqueous extraction. For application in rural areas, mechanical extraction is considered to be the best option. In this extraction hydraulic presses are used to remove oil from the seeds. This method is generally preferred because of its lower initial and operational cost, and because it can be easily operated by semi-skilled personnel. It produces relatively good quality oil as compared to the solvent extraction process and it allows for the use of the cake residue (Olajide et al., 2007). However, a disadvantage of mechanical extraction is the lower oil recovery compared to solvent extraction. It has been reported that solvent extraction with *n*-hexane could achieve about 70–99% oil recovery, against a reported maximum of 60–80% for mechanical extraction (Achten et al., 2007).

Applied pressure, pressing temperature, and pressing time are important process parameters, while the adjustment of seed moisture content is shown to be the most important factor amongst pretreatments such as removal of hulls or shells, size reduction or heat treatment. Willems et al. (2008) reported higher oil yield for rapeseed, sesame, linseed, jatropha seed and jatropha kernel pressed at higher pressures and/or temperature. He also reported the 22% difference in oil recovery when pressed linseed at various moisture contents varied from 0 to 10%. Our previous study indeed shows that applied pressure, pressing temperature, and moisture content are important parameters that influence oil recovery. The rate of pressure is found to be optimum at 0.125 MPa/s (Subroto et al., 2014). This study indicated that the optimum oil recovery is within the range of 10–20 MPa, 60–90 °C and 3–5% (w.b.). This implies that maximizing the oil recovery is limited to the optimization of these process parameters. This research is aimed to study and model the effect of these variables and their interaction on the percentage of oil extraction. The model will be used to optimize the extraction, and the accuracy of the model will be tested.

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Table 1
Properties of Jatropha samples before moisture conditioning.

Properties	Jatropha Kalimantan	Jatropha Subang
Weight (% d.b.)		
Seed	100	100
Kernel	63.0	63.4
Shell	37.0	36.6
Oil content (% d.b.)		
Seed	36.8 ± 0.05	35.1 ± 0.06
Kernel	58.3 ± 0.02	55.3 ± 0.01
Shell	0.1 ± 0.01	0.1 ± 0.01
Moisture content (% w.b.)		
Seed	8.6 ± 0.18	8.5 ± 0.10
Kernel	7.04 ± 0.13	6.97 ± 0.17
Shell	11.3 ± 0.24	11.1 ± 0.21

2. Materials and methods

2.1. Material

Jatropha seeds used in the optimization experiment were obtained from Palangkaraya, Central Kalimantan, Indonesia. The mature fruits were harvested manually in March 2011. The seeds were dried under sun and stored in jute bags in a warehouse facility at temperatures between 20 and 30 °C and relative humidity of 80–90% for one month. In addition to Jatropha from Kalimantan, Jatropha from Subang was used for oil quality analysis. Jatropha seed from Subang was harvested manually during January 2011, dried under sun and stored in jute bags in a warehouse facility at temperatures between 20 and 30 °C and relative humidity of 70–80% for 3 months. After transport to the Netherlands in April 2011, both seeds were stored at room temperature (within a range of 18–22 °C) and relative humidity of 40–50%. The seeds were de-shelled manually and both the kernels and shells were analyzed for weight fraction, initial moisture and total oil content (see Table 1). The kernels were exposed to moisture conditioning pretreatment before being pressed (described below). The pretreated kernels were used directly in the pressing experiments to reduce the influence of storage time on oil quality. The oil analyses were conducted directly after pressing in May 2011 for both sources of Jatropha seeds.

Potassium hydroxide (pellets, 85%, Vetec), oxalic acid anhydrous (≥99%, Sigma–Aldrich), ethanol (95%, Sigma–Aldrich), diethyl ether (≥99%, Sigma–Aldrich), hexane (≥99, Sigma–Aldrich), Hydranal solvent (Fluka) and Hydranal titrant 5 (Fluka) were bought from Sigma–Aldrich (Amsterdam, The Netherlands).

For oil recovery measurement, the kernels were conditioned by oven drying. The drying temperatures were 35, 40 and 50 °C for desired moisture content of 5, 4 and 3% w.b., respectively. After drying, the kernel was wrapped tightly in a low density polyethylene bag of 25 μm thickness and then put inside a desiccator containing silica gel for a minimum of 1 day before being pressed. For oil quality analysis, the kernels were stored inside the desiccator containing silica gel until the desired moisture content was reached, and then wrapped in the polyethylene bag for equilibration.

The initial moisture content of the samples was determined by oven drying of 10 g of sample at 105 °C for 24 h. Duplicate measurements were performed for each sample and average values were taken. Moisture content after conditioning was determined by calculating the weight difference of the sample after and before conditioning.

2.2. Hydraulic pressing

A schematic representation of the hydraulic press is shown in Fig. 1. The pressing chamber was made from stainless steel with a diameter of 20 mm and a height of 70 mm. It is equipped with a perforated plate (diameter of 1 mm) covered with fine wire mesh (100 mesh). This was placed at the bottom of the pressing chamber acting as filter during extraction. An electrical-resistance heating ring attached around the pressing chamber is used to preheat the pressing chamber during operation within a temperature range of 60–90 °C. Pressures up to 20 MPa were applied by a hydraulic plunger. The press is completed with a thermocouple (±2.5 °C), pressure measurement (±1 MPa), and a level indicator (±0.01 mm), which measures the distance the plunger traveled.

Table 2
Actual and coded levels of the independent variables in the experimental design.

Independent variable	Symbol		Level	
	Actual	Coded	Actual	Coded
Applied pressure (MPa)	P	x_1	10	−1
			15	0
			20	1
Heating temperature (°C)	T	x_2	60	−1
			75	0
			90	1
Moisture content (% w.b.)	M	x_3	3	−1
			4	0
			5	1

Approximately 7 g of kernels was placed in the pressing chamber. Afterwards, the plunger is put on top of the kernels. The sample is preheated for 5 min without applying mechanical pressure. Subsequently, the mechanical pressure was increased linearly at a pressing rate of 0.125 MPa/s until the desired pressure is reached. Total pressing time was 10 min. For validation experiment, three replicate measurements were performed for each sample and average values were taken.

2.3. Statistical analysis

Levels for the independent variables, i.e. applied pressure, X_1, pressing temperature, X_2, and moisture content, X_3, were based on results obtained in a previous study (Subroto et al., 2014). A three-factor-three-level face centered central composite design (CCRD) was applied where the values of the independent variables X were coded as the variables, x in the range of −1 and +1 level (shown in Table 2). The mathematical transformation of any actual level of applied pressure, temperature, and moisture content into the coded level can be obtained, respectively, from the following equations:

$$x = \frac{(X - X_M)}{X_D} \quad (1)$$

$$X_M = \frac{(X_{max} + X_{min})}{2} \quad (2)$$

$$X_D = (X_{max} - X_M) \quad (3)$$

$$x_1 = \frac{(X_1 - 15)}{5}, \quad x_2 = \frac{(X_2 - 75)}{15}, \quad x_3 = X_3 - 4 \quad (4)$$

where X_M, X_D, X_{max}, and X_{min} is the mean value, interval of variation, maximum and minimum value of X, respectively. While, x_1, x_2 and x_3 are the coded values and X_1, X_2 and X_3 are the actual values for applied pressure, temperature and moisture content.

The experimental plan was designed and the results obtained were analyzed using Design Expert version 8.0.0 software (State-Ease Inc., Statistics Made Easy, Minneapolis, MN, USA) to build and evaluate models and to plot the three-dimensional response surface curves. The experimental data were analyzed for the response.

Twenty experiments were performed which consisted of eight factorial points, six extra points (star points) and six replicates for the center point. The six replicates for the center point were used to estimate the experimental error. An analysis of variance (ANOVA)

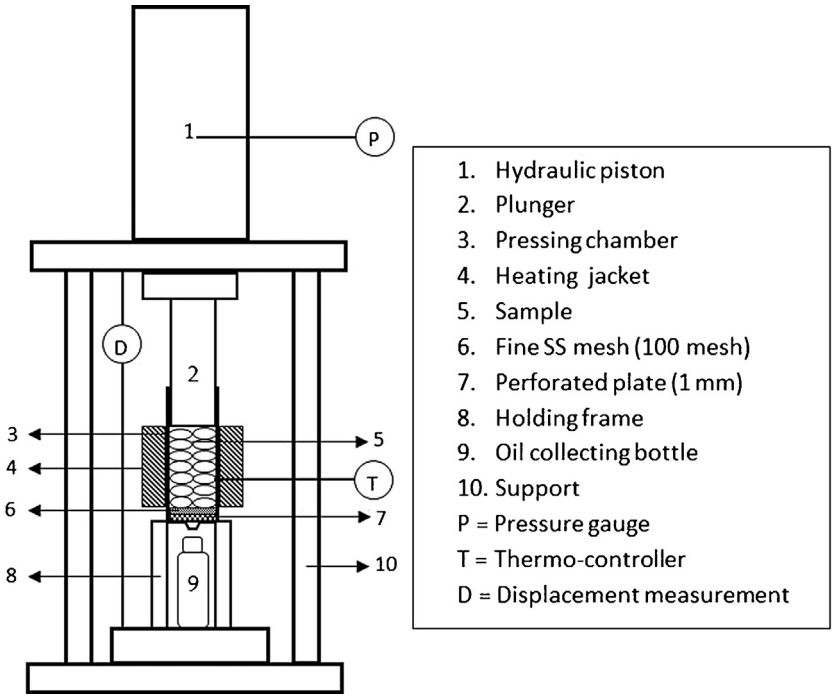

Fig. 1. Schematic representation of the hydraulic press.

and R^2 (coefficient of determination) statistic were used to check the adequacy of the developed model. Using an *F*-test, it was possible to test the variation of the data around the fitted model (lack of fit). The significance level was stated at 95%, with *p*-value 0.05. Confirmatory experiments were carried out to validate the equations using the combinations of independent variables. These were not part of the original experimental design, but within the experimental region. The optimal conditions for the Jatropha oil recovery were obtained using the software's numerical optimization function.

2.4. Total oil content and oil recovery

Total oil content of the kernels was determined by the weight of substances that are extracted by *n*-hexane according to standard Soxhlet extraction method. The kernels were dried overnight in an oven at 105 °C. The dried kernels were grinded using a coffee grinder (Princess 242195, Princess Household Appliances B.V., The Netherlands) and approximately 10 g were transferred into a cellulose extraction thimble (Whatman, I.D. × *H* 18 × 55 mm, GE). This was extracted with hexane at its boiling point for 24 h. The hexane–oil mixture was evaporated in a rotary vacuum evaporator while the cake was dried in the oven at 80 °C overnight. Duplicate measurements were performed and average values were taken. The total oil content was defined as weight of oil extracted over the dry weight of the sample taken. The oil recovery was defined as the ratio of the amount of oil expressed during mechanical pressing to the total oil content of the sample at dry weight basis.

2.5. Oil quality analysis

Hydraulic pressed oils which still contained some fine particles were centrifuged at 2000 × *g* for 10 min. The clear oils were recovered and used for oil analysis. Chemical and physical analyses of the samples were carried out according to the standard test methods: DIN EN 14111, DIN EN 14104, DIN EN ISO 12937, DIN EN 14112, DIN EN 14107 and DIN EN 14538 for iodine value, acid value, water content, oxidative stability, phosphorus content, and calcium and magnesium (Ca + Mg) content, respectively. Density analysis was carried out by measuring the mass of the samples with respect to their volume using a 10 mL pycnometer. The density at 15 °C was obtained by extrapolation of data collected from 30 to 100 °C, in 10 °C increments. Dynamic viscosity analysis was carried out by using a cone-and-plate viscometer AR 1000-N (40 mm 2° aluminum cone) at 40 °C with a shear rate of 40/s for 10 min. Kinematic viscosity was calculated from the corresponding dynamic viscosity and density at corresponding temperature. The flash point of the samples was measured according to the methods described in ASTM D6450 using a MINIFLASH FLP/H/L. Cloud point (CP) and pour point (PP) were measured using a Tanaka Scientific Limited Type MPC-102 L according to methods described in ASTM D6749 and ASTM D2500, respectively.

According to the German fuel standard DIN 51605: 2010-10 for pure plant oil, the acid value, phosphorus content, and water content should not exceed 2 mg KOH/g oil, 3 ppm, and 750 ppm respectively, and oxidative stability should be at least 6 h. Most chemical property analyses on the plant oils were conducted in our laboratory with the exception of phosphorus content, which was conducted by ASG Analytik-Service GmBH, Germany. Duplicate measurements were performed on each sample and average values were taken.

3. Results and discussions

3.1. Fitting the model and analysis of variance (ANOVA)

In order to determine the optimum process parameters, i.e. applied pressure, pressing temperature and moisture content for maximum oil recovery, the experiments were designed according to a face centered central composite design in three variables following the Response Surface Methodology (RSM). The experimental set-up and corresponding experimental responses are shown in Table 3. The oil recovery from the samples exhibited an increase with increasing temperature and pressure. The lowest value of 68.9% was obtained at 10 MPa pressure, 60 °C and 3% (w.b.) moisture; the highest value of 86.4% was obtained at 20 MPa, 90 °C and 3% (w.b.) moisture.

Table 3
Experimental layout of face centered central composite design and its corresponding observed values of oil recovery.

Run	Variable properties			
	Pressure (MPa)	Temperature (°C)	Moisture content (% w.b.)	Oil recovery (% d.b.)
1	10	60	3	69.6
2	20	60	3	78.6
3	10	90	3	79.1
4	20	90	3	86.4
5	10	60	5	73.7
6	20	60	5	78.6
7	10	90	5	79.0
8	20	90	5	82.3
9	10	75	4	78.2
10	20	75	4	84.6
11	15	60	4	79.8
12	15	90	4	86.1
13	15	75	3	80.5
14	15	75	5	79.3
15	15	75	4	83.3
16	15	75	4	84.2
17	15	75	4	83.2
18	15	75	4	83.7
19	15	75	4	84.7
20	15	75	4	82.6

Fitting of the data to various models (linear, two factorial, quadratic and cubic) and their subsequent analysis of variance shows that the hydraulic pressing of Jatropha kernel is most properly described with a quadratic polynomial model. The Adjusted R^2 of the quadratic model (0.9752) was higher than that of linear (0.5431) and two factorial (0.5186) models. The cubic model was found to be aliased. The second-order polynomial models used to express the oil recovery (Y) as a function of independent variables (Eqs. (5) and (6)) are shown below (in terms of coded and actual levels):

$$\text{Oil recovery(coded)} = 83.41 + 3.09x_1 + 3.26x_2 + 0.13x_3 - 0.41x_1x_2 - 1.01x_1x_3 - 1.04x_2x_3 - 1.71x_1^2 - 0.16x_2^2 - 3.21x_3^2 \quad (5)$$

$$\text{Oil Recovery(actual)} = 51.43 + 3.89X_1 + 0.68X_2 + 33.77X_3 - 0.005X_1X_2 - 0.20X_1X_3 - 0.07X_2X_3 - 0.06X_1^2 - 0.001X_2^2 - 3.21X_3^2 \quad (6)$$

Table 4 summarizes the ANOVA (F-test) and p-value that are used to estimate the coefficients of the model, to check the significance of each parameter, and to indicate the interaction strength of each parameter. It was observed from the ANOVA analysis that the confidence level was greater than 95% while the p-value of the model was less than 0.0001. The model with the p-value below 0.05 was statistically significant, which implied that the model was suitable for this experiment. Meanwhile, the "lack of fit" of this model was insignificant with the p-value being 0.76. The main effects, i.e. X_1 and X_2, and interation effects, i.e. X_1^2, X_3^2, X_1X_3, X_2X_3, are significant based on the calculated p-values. The effect of moisture content on oil recovery exhibited a p-value of 0.54. This exceeds a p-value level of 0.05 and indicates that the effect is not significant.

The coefficient of determination (R^2) and adjusted coefficient of determination (R^2_{adj}) were 0.987 and 0.975, respectively which indicated that the estimated model fits the experimental data satisfactorily. Lee et al. (2010) suggested that for a good fit of a model, R^2 should be at least 0.80. The R^2 for these response variables was higher than 0.80, indicating that the regression models explained the mechanism well.

Fig. 2a shows the experimental versus predicted oil recovery obtained from Eq. (6). A linear distribution is observed which is indicative of a well-fitting model. The values predicted from Eq. (6) were close to the observed values of oil recovery from Jatropha kernel. The normal probability plot is also presented in Fig. 2b. The plot indicates that the residuals (difference between actual and predicted values) follow a normal distribution and form an approximately straight line.

3.2. Model validation

The adequacy of the model equations to predict optimum response values was tested using the conditions shown in Table 5. The processing conditions for maximum recovery were used to experimentally validate and predict the values of the responses using the model equation. Close agreement exists between values calculated using the model equation and the experimental values of the response variables at the point of interest. The X^2 goodness-of-fit test was used to examine the validity of the model (Table 5) (Mooney and Swift, 1999). The test shows that there is not a significant difference between the predicted and actual values since the X^2 value (0.02) is much smaller than the cut-off value of X^2 for 95% confidence level for 3 degrees of freedom (7.81). This indicates that the generated model is valid at 95% confidence level.

From the established equation, the maximum oil recovery predicted is 87.8% at process parameters equal to 19 MPa applied pressure, 90 °C pressing temperature and 3.8% (w.b.) moisture content. The actual oil recovery obtained is 87.4 ± 0.5%.

3.3. Effect of independent processing parameters

The effect of the four independent variables on the oil recovery of Jatropha is shown in Fig. 3. Oil recovery improved with increasing pressure as shown in Fig. 3a; in particular when the applied pressure was increased from 10 to 15 MPa. However, upon increasing the applied pressure from 15 to 20 MPa, oil recovery seemed to level off. For example, for Jatropha kernel containing 4% (w.b.) moisture and pressed at 75 °C the oil recovery increased by 5 points from 78.6 to 83.4% by raising the applied pressure from 10 to 15 MPa. The oil recovery only increased by 1 point to 84.8% when the pressure was raised to 20 MPa. At higher pressures, the inter-kernel void is much smaller than at low pressures, which restricts the flow of oil. These results indicate that excessive pressure did not necessarily

Table 4
ANOVA for response surface quadratic model.

Source of variation	Degree of freedom	Sum of squares	Mean square	*F*-value	*p*-Value probability
Model	9	323.92	35.99	83.85	<0.0001[a]
Pressure, *P*	1	95.48	95.48	222.44	<0.0001[a]
Temperature, *T*	1	106.28	106.28	247.58	<0.0001[a]
Moisture, *M*	1	0.17	0.17	0.39	0.5444[b]
PT	1	1.36	1.36	3.17	0.1053[b]
PM	1	8.20	8.20	19.11	0.0014[a]
TM	1	8.61	8.61	20.06	0.0012[a]
P^2	1	8.03	8.03	18.71	0.0015[a]
T^2	1	0.07	0.07	0.16	0.6957[b]
M^2	1	28.32	28.32	65.98	<0.0001[a]
Residual	10	4.29	0.43		
Lack of fit	5	1.46	0.29	0.52	0.7563[b]
Pure error	5	2.83	0.57		
Correction total	19	328.22			
R^2	0.987				
Adj. R^2	0.975				

Values of '*p*-value' <0.0500 indicate model terms are significant.
[a] Significant.
[b] Not significant.

have a positive influence on oil recovery. A similar observation was observed by Willems et al. (2008) while pressing Jatropha seed and kernel from 20 to 70 MPa.

Pressing temperature significantly raised oil recovery as shown in Fig. 3b. This was observed for Jatropha kernel with 4% (w.b.) moisture content when pressed under 15 MPa, the oil recovery increased from 80.0 to 86.5% by increasing the temperature from 60 to 90 °C. Increasing the temperature coagulates the protein, softening the solid structure and decreases the oil viscosity. Kabutey et al. (2012) mentioned pre-heating temperatures of the jatropha seeds decreased seed hardness (N/mm), contrary, seed deformation (mm) increased in relation to the effect of pre-heating temperatures. Burubai et al. (2007) reported that seed deformation of African nutmeg increased with increasing temperatures. Increase in deformation related to the volume of oil releases from compressed cake. Tambunan et al. (2012) found that increasing pressing temperature from 50 to 80 °C increase oil recovery from 70 to 80.9% in pressing of Jatropha seed.

The effect of moisture content on oil recovery is shown in Fig. 3c. At a pressing temperature of 75 °C and an applied pressure of 15 MPa, oil recovery increased from 80.3 to 83.4% with an increase in moisture content from 3 to 4% (w.b.) but then decreased to 80.1% when the moisture content was raised to 5% (w.b.). Moisture content affects the hardness and compactness of the kernel. Sample with lower moisture content has higher hardness (Karaj and Müller, 2010) while at higher moisture contents plasticization promotes cake compaction and restricts the flow of oil. As reported by Kabutey et al. (2011) increasing moisture content from 1 to 10% reduces the deformation. The result shows that the value of 4% (w.b.) was found to be the optimum moisture content for Jatropha kernel. Acheheb et al. (2012) also found 3.95% as the optimum moisture content for pistachio nut.

3.4. Effects of interactive factors

The effect of interaction between factors, i.e. applied pressure, temperature and moisture content is shown in Figs. 4–6. Fig. 4 depicts the effect of applied pressure and pressing temperature on the pressing of Jatropha kernel with moisture content of 4%. The effect of temperature is more significant at lower applied pressure. For example at 10 and 20 MPa, the oil recovery increase from 74.8 to 82.1% and from 81.8 to 87.5%, respectively. This effect was observed by Mpagalile and Clarke (2005) when pressing coconut. The interaction between applied pressure and pressing temperature on oil recovery was explained by Bargale et al. (1999). The effect may be attributed to the interaction of temperature and pressure which at higher levels tend to become counteractive: increasing the temperature decreases the viscosity of the oil thereby increasing its fluidity through the compressed medium whereas an increase in pressure makes the cake harder which restricts the flow of oil.

A comparative study of the results showed that the oil recovery at any pressure was affected by the moisture content of the sample. The effect of applied pressure and moisture content on oil recovery is shown in Fig. 5. An increase in oil recovery is observed when the applied pressure was increased from 10 to 15 MPa. However, the oil recovery tends to level off at higher pressure levels from 15 to 20 MPa. This effect was observed for all levels of moisture content used in this study. For example, when Jatropha kernel with 3% (w.b.) moisture content is pressed at 75 °C, the oil recovery increased from 74.5 to 80.3% upon increasing pressure from 10 to 15 MPa and then slightly increased to 82.7% when the pressure is raised to 20 MPa. A similar trend was observed for Jatropha kernel with 5% (w.b.) moisture content: the oil recovery increased from 76.3 to 80.1% by increasing pressure from 10 to 15 MPa and then leveled off to 80.4% towards a pressure of 20 MPa. This leveling of the oil recovery

Table 5
Validation of model equation.

Run	Variable properties			Oil recovery (%)	
	P	*T*	*M*	Experimental (*E*)	Predicted (*P*)
1	19	90	3.8	87.4 ± 0.5	87.8
2	15	60	3	75.1 ± 1.1	75.9
3	15	60	5	77.7 ± 1.7	77.7
4	10	60	4	75.3 ± 0.6	74.8
5	20	60	4	81.2 ± 1.1	81.8

$X^2 = \sum \frac{(E-P)^2}{P} = 0.02$.

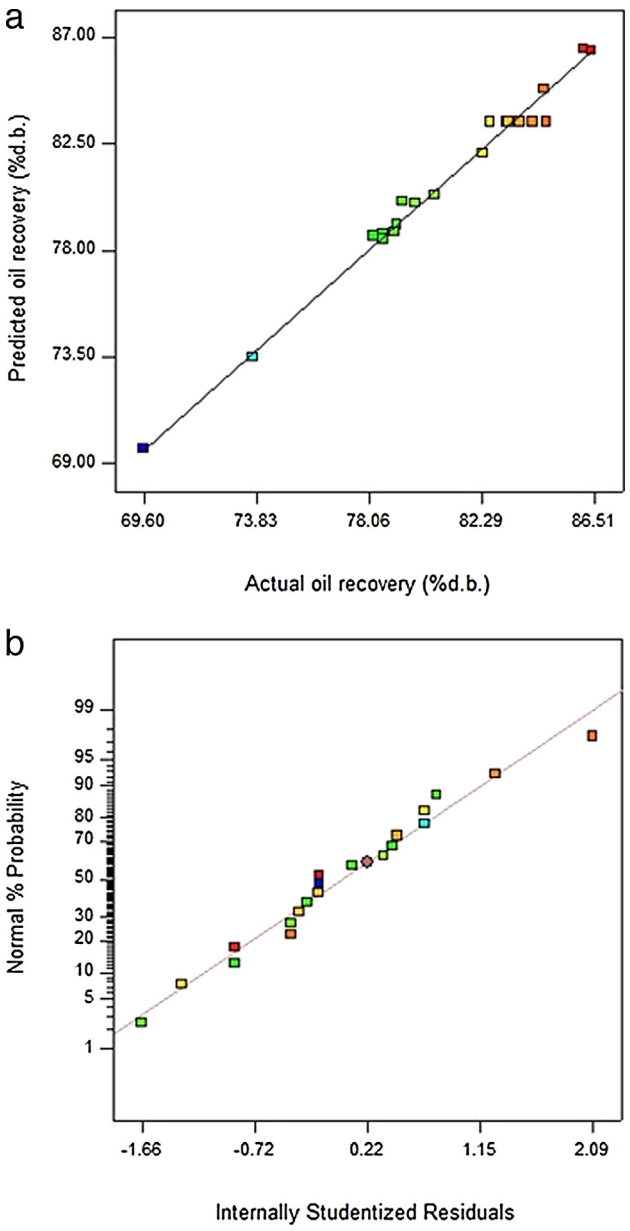

Fig. 2. Correlation of actual conversions and values predicted by the model (a) and normal probability of residuals (b).

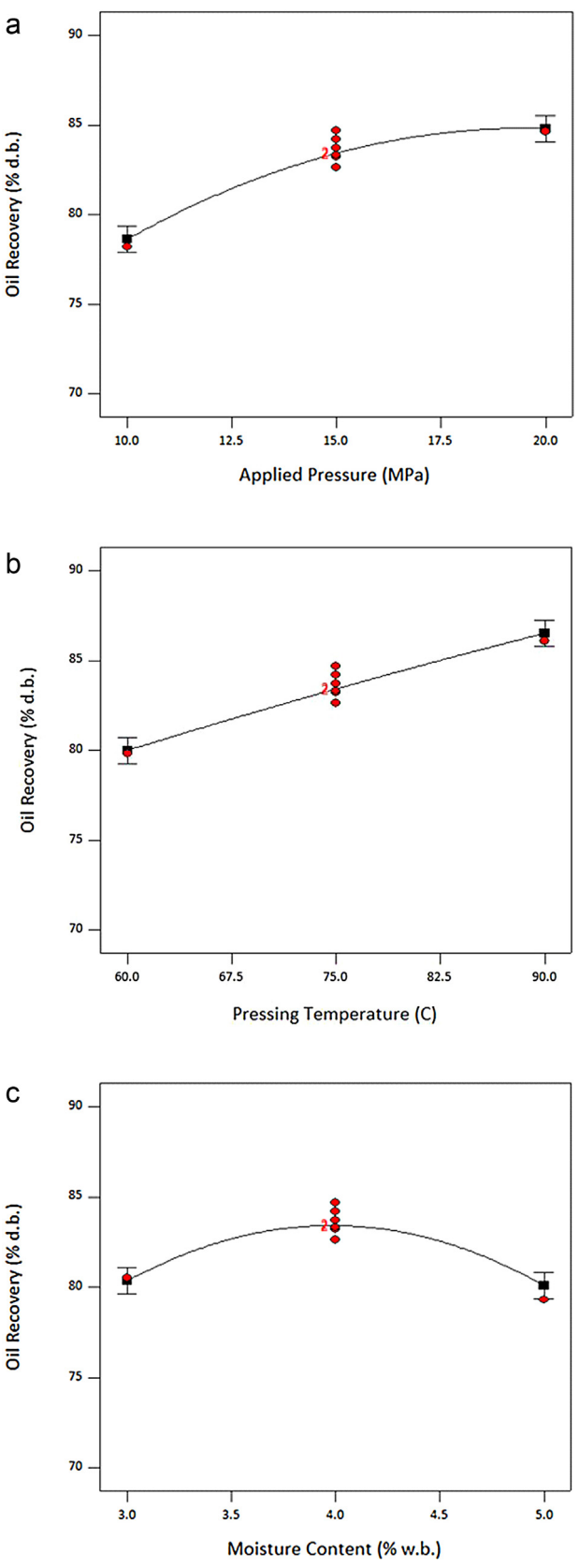

Fig. 3. Effect of various individual parameters: applied pressure (a), temperature (b), and moisture content (c), on the pressing of Jatropha kernel. One parameter is varied while the others are kept constant at their center points.

was appreciably noticeable in samples with higher moisture content, which shows that under such conditions high pressure is not effective in increasing the oil recovery. This effect was observed by Mpagalile and Clarke (2005) when pressing coconut.

Moisture content affects the hardness and compactness of the kernel. At lower moisture contents, evaporation causes the surface of the sample to harden, thus requiring a higher pressure to be used to overcome the hardened sample during pressing. Thus increasing pressure increases the oil recovery. At higher moisture contents, the presence of water will acts as plasticizer between the protein-rich cake and oil which forms paste-like plastized material. This promotes cake compaction and restricts the flow of oil. Thus increasing pressure at higher moisture content gave hardly noticeable changes or even some reduction in oil recovery.

The effect of temperature and moisture content on oil recovery is shown in Fig. 6. This was observed for Jatropha kernel with 3% (w.b.) moisture content when pressed at 15 MPa: the oil recovery increased from 75.9 to 84.5% by increasing the temperature from 60

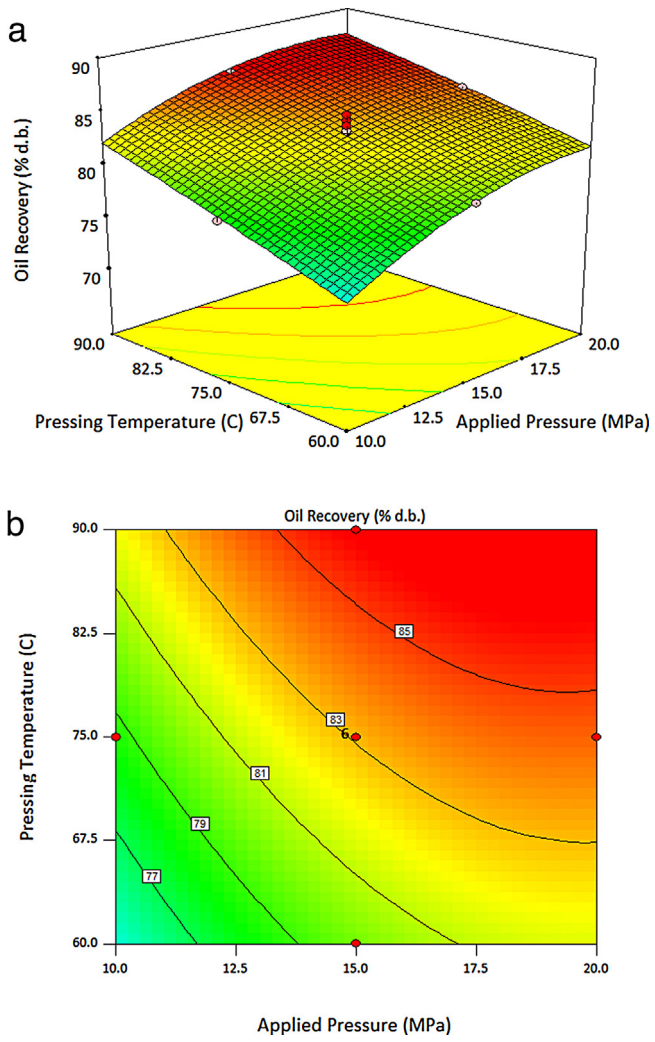

Fig. 4. Response surface (a) and contour plots (b) of oil recovery as function of applied pressure and temperature at moisture content of 4% (w.b.).

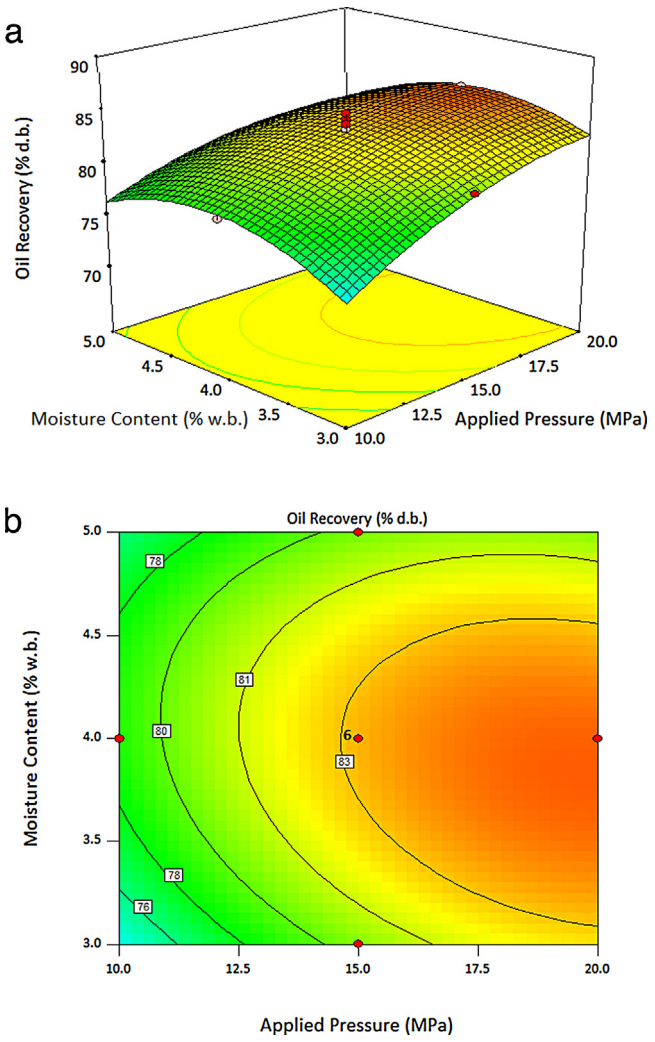

Fig. 5. Response surface (a) and contour plots (b) of oil recovery as function of applied pressure and moisture content at pressing temperature of 75 °C.

to 90 °C. A similar trend was observed for Jatropha kernel containing 5% (w.b.) moisture content: oil recovery increased from 77.7 to 82.1% by increasing pressing temperature from 60 to 90 °C. Increasing temperature at low moisture content gave a more noticeable effect on oil recovery than at higher moisture content.

The interactive effect between pressing temperature and moisture content can be explained as: increasing the temperature reduces the viscosity of the oil thereby increasing its fluidity through the compressed medium. An increase in moisture content makes the cake less compressible and restricts the oil flow. Thus increasing pressing temperature at higher moisture content gave less noticeable changes in oil recovery increment.

Manipulating moisture content enabled high oil expression efficiencies to be attained even at relatively low pressure or low temperature. Low moisture content samples need lower temperature or applied pressure to get the same oil recovery compared to higher moisture content. For example, oil recovery of 83.4% was attained at a pressure of 15 MPa and a temperature of 75 °C for kernels with 4% moisture content, while an oil recovery of 82% was obtained at a pressure of 20 MPa and temperature of 90 °C for kernels with 5% moisture content. Similar results were obtained by Mpagalile and Clarke (2005) and Ebewele et al. (2010) in the study on the effect of moisture content, applied pressure and pressing temperature on coconut grating and rubber seed expression.

3.5. Oil quality

Oil expression experiments were carried out with Jatropha kernels from different plantations. The best processing conditions showed to be: 19 MPa applied pressure, 90 °C pressing temperature and 3.8% (w.b.) moisture content. Data on the oils are summarized in Table 6. The quality of the obtained oil met the DIN 51605: 2010-10 standard for plant oil based fuel except on phosphorus content and group II metals. Acid value is a measurement of the hydrolytic degradation of oil during storage or processing, i.e. the hydrolysis of ester bonds in lipids by enzyme action or by heat and moisture, results in the liberation of FFAs. The oxidative stability index is associated with the oxidative degradation of oil which results in development of rancidity. A higher Oxidative Stability Index (OSI) expressed in hours Induction Period (IP) means more resistance to oxidation. Phosphorus content indicates the presence of phosphor-derived components in oil such as phospholipids and phytates. The iodine value is a measure of the degree of unsaturation of the oil (the higher the iodine value, the greater the degree of unsaturation). Jatropha oil from Kalimantan has a higher phosphorus content compared to Jatropha oils from Subang as is shown in Table 6. Soil nutrition or fertilizer is considered as a cause for this result with Jatropha from Kalimantan grown on more nutritious land than that from Subang. According to Lickfett et al. (1999), phosphorus content in seed is affected by the level of phosphorus

Table 6
Physical and chemical characteristics of *Jatropha curcas* L. oil.

Parameters	Test methods	Units	DIN 51605: 2010-10	Value
Flash point	ASTM D6450	°C	101 (min)	200[b]–204[a]
Cold point	ASTM D6749	°C		(−1)[b]–(−2)[a]
Pour point	ASTM D2500	°C		(−3)[b]–(−4)[a]
Iodine value	DIN EN 14111	g/100 g	125 (max)	108[a]–113[b]
Density (15 °C)	Pycnometer	kg/m^3	910–925	923[a]–925[b]
Kinematic viscosity (40 °C)	Viscometer	mPas	36 (max)	35.8[a]–37.2[b]
Oxidative stability (110 °C)	EN 14 112	hours	6 (min)	9.49[a]–13.99[b]
Acid value	DIN EN 14104	mgKOH/g	2 (min)	0.25[b]–0.6[a]
Phosphorus	DIN EN 14107	ppm	3 (max)	2.8[a]–25.9[b]
Group 2 metal (Ca + Mg)	DIN 51627-6	ppm	2 (max)	3.8[a]–31.2[b]
Moisture content	DIN EN ISO 12937	ppm	750 (max)	697[a]–747[b]

Note: Jatropha sample used for analysis are from Subang (a) and Kalimantan (b).

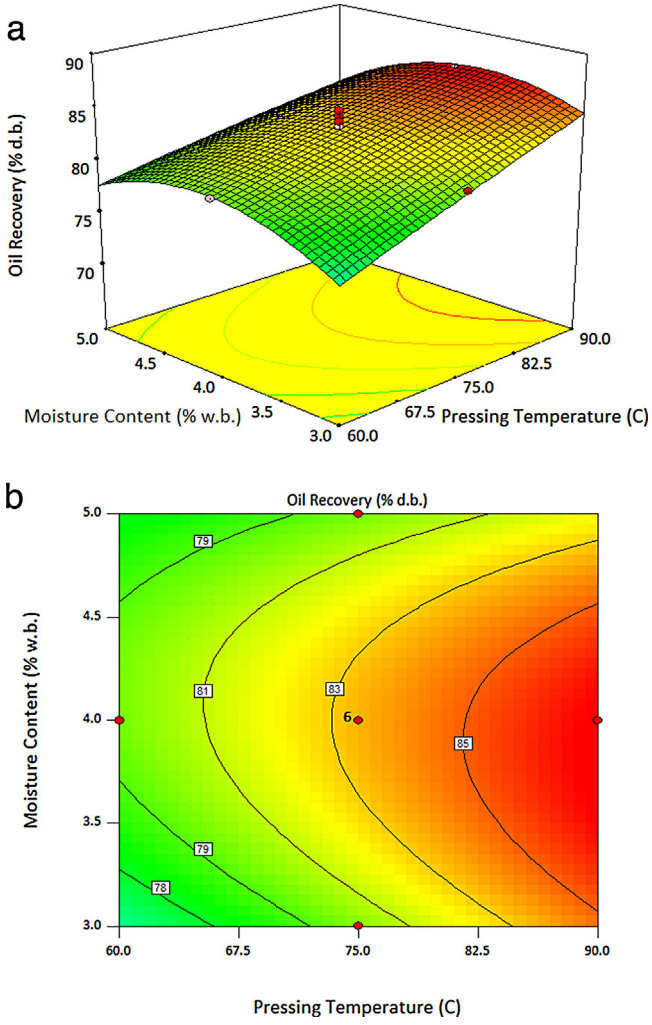

Fig. 6. Response surface (a) and contour plots (b) of oil recovery as function of temperature and moisture content at applied pressure of 15 MPa.

fertilizer supply or phosphorus content in soil. The higher oxidative stability of Jatropha oil from Kalimantan is attributed to the presence of phospholipids, phytates and other natural antioxidants. Phospholipid is known for its antioxidant properties and can work together with natural antioxidants such as tocopherols (Choe and Min, 2006).

4. Conclusions

A face centered central composite RSM design was used to determine the optimum conditions for the processing parameters applied in the extraction of oil from *Jatropha curcas* L. kernel by hydraulic pressing. It is found that applied pressure, pressing temperature, and the quadratics of applied pressure and moisture content, as well as interaction between applied pressure and moisture content and between pressing temperature and moisture content are significant factors affecting the oil recovery. The second order polynomial equation developed in this study shows a high correlation between observed and predicated oil recovery values. Response surface analysis was found to be a good approach for visualizing process-parameter interaction. The models developed by RSM shall be useful for predicting the optimum processing condition to achieve maximum oil recovery of *J. curcas* L. kernel pressing. Oil extraction executed at an applied pressure of 19 MPa, a pressing temperature of 90 °C and a kernel moisture content of 3.8% (w.b.) gave an actual oil recovery of 87.4 ± 0.5% which closely matches the predicted value of 87.8%. The oil quality satisfies the requirements set by the DIN 51605: 2010-10 norm for plant oil based fuels.

Conflict of interest

The authors have declared no conflict of interest on financial and or commercial application of this study.

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