Industrial Crops and Products 63 (2015) 294–302

Contents lists available at ScienceDirect

Industrial Crops and Products

journal homepage: www.elsevier.com/locate/indcrop

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

Erna Subroto [a], Robert Manurung [b], Hero Jan Heeres [a], Antonius Augustinus Broekhuis [a,*]

[a] Department of Chemical Engineering, Rijksuniversiteit Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands
[b] School of Life Sciences and Technology, Institut Teknologi Bandung, Jl. Ganesha 10, Bandung, Indonesia

ARTICLE INFO

Article history:
Received 28 January 2014
Received in revised form 13 August 2014
Accepted 14 August 2014
Available online 14 November 2014

Keywords:
Jatropha curcas L.
Oil extraction
Hydraulic press
Face-centered composite design
Response surface methodology
Process optimization

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.

* Corresponding author. Tel.: +31 50 363 4918; fax: +31 50 363 4479.
E-mail address: a.a.broekhuis@rug.nl (A.A. Broekhuis).

http://dx.doi.org/10.1016/j.indcrop.2014.08.050
0926-6690/

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)