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Modeling and optimization of laboratory-scale conditioning of *Jatropha curcas* L. seeds for oil expression

Joana Rodrigues[a], Isabel Miranda[b], Jorge Gominho[b], Manuel Vasconcelos[c], Gonçalo Barradas[c], Helena Pereira[b], Fernando Bianchi-de-Aguiar[c], Suzana Ferreira-Dias[a,*]

[a] Universidade de Lisboa, LEAF, Instituto Superior de Agronomia, Tapada da Ajuda, 1349-017 Lisboa, Portugal
[b] Universidade de Lisboa, CEF, Instituto Superior de Agronomia, Tapada da Ajuda, 1349-017 Lisboa, Portugal
[c] GALP Energia, Unidade de Desenvolvimento de Biocombustíveis, Rua Tomás da Fonseca—Edifício GALP—Torre C, 1600-209 Lisboa, Portugal

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ABSTRACT

The effect of conditioning of *Jatropha curcas* L. seeds on the yield of oil extracted by screw pressing was studied. A pressing test was performed with seeds without heating treatment and the extraction yield was 84.0%. Modelling dry conditioning of seeds and optimization of operation conditions were performed using Response Surface Methodology with a total of 11 dry conditioning experiments, following a central composite rotatable design as a function of temperature (52–108 °C) and time (8–92 min). An increase in the extraction yield occurred in all the experiments, with a maximum of 5.6% (w/w) increase, indicating the need to perform seed conditioning before mechanical extraction. The minimum oil contents in the press cakes, and therefore the highest extraction yields (c.a. 88%, w/w) were obtained with seeds treated at higher temperatures and shorter periods of time (e.g., 90 °C/10–20 min; 80 °C/30 min) or at lower temperatures and longer periods of time (e.g., 60 °C/50–60 min). Steam treatment at temperatures of 100, 110 and 120 °C resulted in seed softening and press clogging and no oil could be extracted.

1. Introduction

The scarcity of fossil fuels and the political instability in oil producing countries have stimulated research into alternative renewable energy sources, such as biofuels. One of the major advantages of this biomass derived fuels is that they can be produced anywhere in the world from local raw materials.

However, the relationship between biofuels production and food security has been controversial. Second-generation biofuels, also known as advanced biofuels, have developed to circumvent this issue since they are produced from non-food biomass, including by-products, wastes and dedicated feedstocks. Unlike first-generation biofuels, they do not compete with food production and usually can grow in marginal lands e.g., in arid and semiarid regions. Production of second generation biofuels is being encouraged by gradually phasing in mandatory targets for their use in the transport sector (Committee on Industry, Research and Energy, 2013).

The oil from seeds can be obtained either by mechanical or by solvent extraction processes. Solvent extraction (usually using *n*-hexane or petroleum ether) produces higher yields of oil than mechanical extraction methods: 99.3% versus 75–85% (Ofori-Boateng et al., 2012). However, solvent extraction is only economically viable when large amounts of seed can be processed (a minimum processing capacity of 100 t/day for continuous-feed processes), while mechanical extraction by expellers are economically feasible for smaller quantities (Adriaans, 2006). Furthermore, solvent extraction is only suitable where storage, transportation, power, water and solvent supply are available; and when training for safety and health at work occurs (Adriaans, 2006). Also, due to long extraction time, solvent extraction consumes much more energy than mechanical extraction.

The mechanical extraction or expression involves the use of mechanical forces to remove oil from the seed, such as batch hydraulic pressing or continuous mechanical pressing (screw presses). Screw pressing is now experiencing a renewed interest as an alternative process to solvent extraction in developing countries. This is mainly due to the simplicity and robustness of the equipment, which make this type of press suitable to be controlled by semi-skilled workers. In addition, a screw press can be adapted

* Corresponding author. Fax: +351 213653200.
E-mail address: suzanafdias@mail.telepac.pt (S. Ferreira-Dias).

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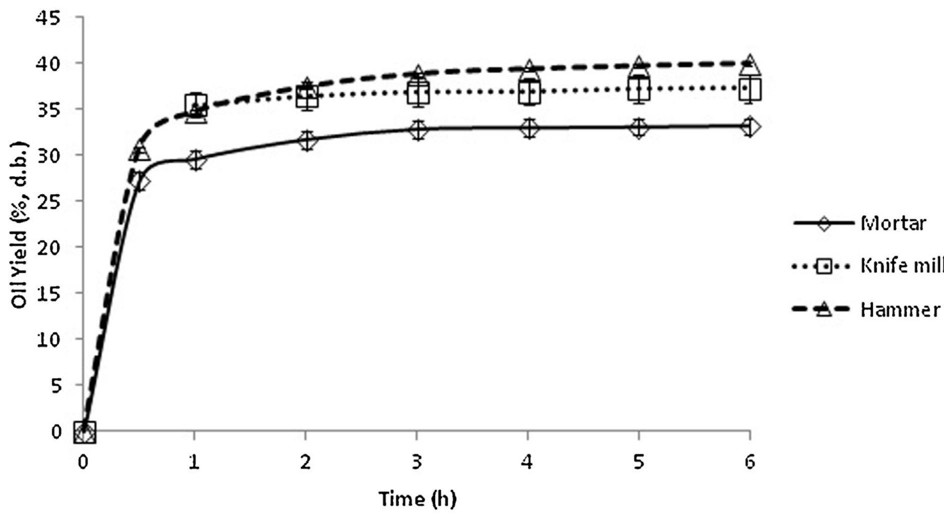

Fig. 1. Average amount of oil extracted (%, dry basis) over time for different types of milling (mortar, hammer and knife mill).

quickly for processing different kinds of oil seeds in a multi-purpose plant (Pradhan et al., 2011). The possibility of using non-toxic seed cake resulting from the pressing, as fertilizer or animal feed, since it is free of toxic solvents is also advantageous, when compared to more efficient solvent extraction equipment (Bargale and Singh, 2000; Fitch-Haumann, 1997).

Optimization of the continuous mechanical pressing consists of (i) defining the optimum conditions related with the preparation of the raw material and/or (ii) adjustments on the press, in order to reach maximum yields of oil, using a minimum value of pressure applied by the press (Pighinelli and Gambetta, 2012).

After seed cracking and before mechanical oil extraction, a thermal treatment (cooking or conditioning) can be performed in order to improve oil yield extraction and quality (Erickson, 1990). This thermal treatment has the following purposes: (1) rupture of cell walls to facilitate the outflow of oil; (2) reduction of oil viscosity, which allows the oil to flow more easily; (3) adjustment of moisture content to the optimal value for pressing; (4) coagulation of proteins; (5) inactivation of enzymes and destruction of some microorganisms; and (6) insolubilization of phosphatides in the cake, which helps to minimize subsequent refining losses (Pradhan et al., 2011; Singh et al., 2002).

However, an excess in the heating time and/or temperature will result in a cake with lower nutritional value and oil with lower quality (Walkelyn and Wan, 2006).

The seed moisture content at the time of pressing is reported to be the most important factor affecting cake residual oil content, using either hydraulic or screw presses. For sunflower seed and rapeseed, optimal moisture contents of 6% and 7%, respectively, have been reported, to be optimal in hydraulic pressing (Bargale and Singh, 2000; Zheng et al., 2003).

The drought resistant tropical and sub-tropical shrub, *Jatropha curcas* L. is considered one of the best sustainable second generation biofuel sources, due to the high oil content of its seeds (27–42%) (Achten et al., 2007; Rodrigues et al., 2013, 2015). Jatropha oil, which can be extracted from the seeds either by mechanical expression or chemically by organic solvents, has a great potential to be used as raw-material for biodiesel production (Rodrigues et al., 2013, 2015; Shivani et al., 2011). The obtained by-products (e.g., fruit husks, glycerol and cake) can also be valorized. Therefore, the extraction of the oil from its seeds by mechanical screw pressing is preferred considering economical, safety and environmental aspects. However, the mechanical oil extraction of *Jatropha curcas* seeds was reported as suboptimal due to lack of knowledge about the best operation conditions (Shah et al., 2005). Thus, the optimization of mechanical extraction of Jatropha seed oil must be performed.

The aim of this study was to model and optimize, via Response Surface Methodology (RSM), the thermal treatment of Jatropha seeds (heating temperature and time), as a conditioning step prior to the mechanical oil extraction in order to maximize oil yield, at laboratory scale. The total amount of oil in the seeds was quantified after optimization at laboratory scale regarding oil extraction time and grain dimensions.

2. Material and methods

2.1. Raw material

Jatropha curcas L. seeds were collected during 2013 from healthy and ripe fruits harvested at the GalpBúzi field trial (19°56′S; 34°24′E) in Sofala province, in central Mozambique. These seeds belong to a *Jatropha curcas* accession brought from Brazil to Mozambique. Only good-condition and whole seeds were selected to ensure higher uniformity of experimental results. The moisture content of the seeds was determined by drying at 100–105 °C, until constant mass, in an oven (WTB Binder 7200) in triplicate measurements.

2.2. Size reduction and fractioning of seeds

In order to select the best size reduction method, the seeds were grinded using (i) a household knife mill, (ii) a mortar and (iii) a hammer. The particles obtained with the knife mill and the hammer were sieved with a 2 mm diameter sieve and the fraction under 2 mm diameter was used.

Granulometric fractioning of the seed particles obtained with each size reduction method was carried out after oil removal by petroleum-ether for 6 h in a Soxtec apparatus (non-extracted seeds clogged the sieve screens) using 20, 40, 60 and 80 mesh sieves.

2.3. Oil solvent extraction

The milled not dehulled Jatropha seed samples (4.5 g) were put into a cellulose extraction thimble mixed with sand (2 g) to act as an extraction adjuvant to prevent the formation of a compact cake. The filled thimble was loaded inside a flat bottom flask with 180 mL petroleum-ether as extraction solvent. Extractions were carried

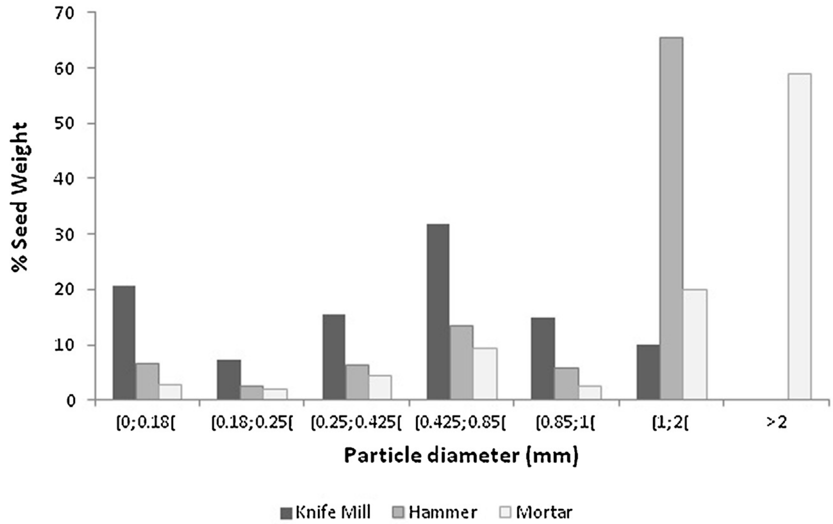

Fig. 2. Histogram of the different granulometric fractions of seeds in the milling tests performed with a knife mill, a hammer and a mortar.

out, in a Soxhlet apparatus, up to 6 h to ensure complete oil extraction. The oil solution (miscella) was recovered after the first 30 min extraction and every hour until the end of the test. The solvent was replaced after each extraction time. The miscella was filtered under vacuum (Millipore glass base and funnel) to remove suspended solids and the solvent was evaporated using a rotary vacuum evaporator. The oil was collected and weighted. Each extraction was carried out in triplicate.

2.4. Conditioning of Jatropha seeds and oil expression

Jatropha whole seeds (not dehulled) were crushed with a hammer and the fraction smaller than 2 mm diameter was subjected to two types of conditioning treatments at different temperatures and times, prior to mechanical oil extraction: steam conditioning and dry conditioning. About 400 g of seeds were used in each assay.

Seeds were treated with saturated steam during 30 min in an autoclave under the following conditions: 100 °C (101 kPa); 110 °C (143.3 kPa); and 120 °C (198.5 kPa).

The dry conditioning was made in an oven and Response Surface Methodology (RSM) was used to find the best operation conditions. With RSM, several variables are tested simultaneously with a minimum number of trials, using special experimental designs that enable to find interactions between the variables which cannot be identified with classical approaches (Gacula and Singh, 1984; Haaland, 1989).

The experiments were carried out following a central composite rotatable design (CCRD), as a function of temperature (52–108 °C) and time (8–92 min). A total of 11 experiments were carried out (Table 1): 4 factorial points (experiments 1–4); 4 star points (experiments 5–8) and 3 central points (experiments 9–11). The repetition of the central points is used to determine the experimental error, which is assumed to be constant along the experimental domain. The experiments were performed randomly in order to avoid systematic errors.

Immediately after seed conditioning, the oil was mechanically extracted in a screw press, Täby Press type 20 (Skeppsta Maskin AB, Sweden), equipped with an outlet nozzle with 9 mm internal diameter. Oil extraction assays were also performed for seeds without any thermal treatment.

The seeds and the pressing cake were weighed and analysed for oil (c.f. 2.3) and moisture contents (in triplicate). The oil pressing yield (%) was calculated by a mass balance considering the initial oil content in the seeds and the residual oil content in the pressing cake (in dry basis).

3. Results and discussion

3.1. Optimization of solvent extraction

The oil extraction kinetics from seed particles obtained with the different size reduction methods is presented in Fig. 1. Seed moisture content was 7.74% (d.b.; STD = 0.23). After 4 h of extraction, the percentage of extracted oil (d.b.) becomes nearly constant and after 6 h, the extraction yield was 33, 37 and 40%, for the seeds crushed with a mortar, a knife mill or a hammer, respectively (Fig. 1).

The particle-size distribution of Jatropha seeds crushed with a household knife mill, a mortar or a hammer is shown in Fig. 2. The majority of the particles obtained with the mortar have a size greater than 2 mm, representing about 60% of the particles, and 20% of the particles have dimensions between 1 and 2 mm. The presence of a large amount of big particles may explain the lower extraction yield obtained when the seeds were crushed with the mortar. When a household knife mill was used, about 30% of the material had a size dimension between 0.425 and 0.85 mm and 20% correspond to very small particles (<0.18 mm). These very small particles may form aggregates in the extraction thimble, which may difficult the oil extraction. In addition, some fine seed particles were observed in the miscella which had to be previously vacuum filtered, before extract concentration and solvent recovery. Higher extraction yield was obtained when Jatropha seeds were crushed with a hammer. The majority of the particles (~65%) had a diameter between 1 and 2 mm. These results show that a size reduction to 2 mm or lower is determinant for an efficient extraction but the presence of large amounts of fines (<0.18 mm) will hinder the extraction.

Extraction with petroleum ether for 4 h of seeds ground with a hammer and with addition of sand within the extraction thimble was the selected method to extract oil from Jatropha seed samples.

3.2. Modeling seed conditioning and optimization

A pressing test was performed with seeds (7.2% moisture content, d.b.; 39.5% oil content, d.b.) without heating in order to compare with the results obtained with the treated seeds (Table 1). The oil extraction yield was 83.3% and the pressing cake presented an average residual oil content of 9.8% (d.b.).

Table 1
CCRD: Coded and decoded levels of the experimental factors used.

	Coded levels		Decoded levels		Points
Experiment	Temperature	Time	Temperature (°C)	Time (min)	
1	−1	−1	60	20	Factorial points
2	−1	1	60	80	
3	1	−1	100	20	
4	1	1	100	80	
5	$-\sqrt{2}$	0	51.7	50.0	Star points
6	$\sqrt{2}$	0	108.3	50.0	
7	0	$-\sqrt{2}$	80.0	7.6	
8	0	$\sqrt{2}$	80.0	92.4	
9 (c)	0	0	80	50	Central points
10 (c)	0	0	80	50	
11 (c)	0	0	80	50	

The seeds treated for 30 min with steam at 100, 110 and 120 °C lost their rigidity and became soft, causing clogging of the press. It was not possible to extract the oil from these seeds by pressing. The higher moisture content increased plasticity, thereby reducing the level of compression and acting as a lubricant in the barrel (Singh et al., 2002). Therefore higher moisture content resulted in insufficient friction during pressing.

The experiments for modelling dry conditioning and optimization of treatment conditions (temperature and time) were carried out following a CCRD. The results are shown in Table 2. In all the experiments an increase in the extraction yield occurred, up to about 5.6%. Therefore the dry conditioning of Jatropha seeds is a very important operation to increase oil extraction yield by pressing.

In order to analyze the influence of temperature (T) and heating time (t) of seed conditioning on oil extraction, linear and quadratic effects of each factor, as well as their linear interaction, were calculated for the residual oil in the cake, the oil extraction yield and the increase in extraction yield, compared with the extraction with untreated seeds. Only the linear interaction between time and temperature has a statistically significant effect ($p < 0.05$): positive for the residual oil in the cake and negative for oil extraction yield and yield increase (data not shown). Thus, a simultaneous increase in extraction time and temperature will promote an increase in the amount of residual oil in the cake and, therefore, a reduction in oil extraction yield. An intense heat treatment will probably promote the modification of cell structure with reduction in cell porosity, hindering oil expelling.

However the practical significance of an experimental factor may not coincide with its statistical significance (Haaland, 1989). It is better then to accept factors with higher p-values rather to take the chance of missing an important factor.

Experimental data could be fitted to 3-dimensional response surfaces (Fig. 3), as a function of heating time (t; min) and temperature (T; °C), namely a concave surface for residual oil in cake (Oil_{cake};%, w/w), and convex surfaces for the oil extraction yield (Y;%, w/w) and the increase in extraction yield ($Y_{increase}$;%). These surfaces can be described by the following second order polynomial models, using decoded variables:

$$Oil_{cake} = 14.43 - 0.130T + 0.0006T^2 - 0.081t + 0.0003t^2 + 0.0008(t \times T) \quad (1)$$

$$(R^2 = 0.763; R^2\text{ajust} = 0.525)$$

$$Y = 74.1 + 0.251T - 0.001T^2 + 0.154t - 0.0005t^2 - 0.0014(t \times T) \quad (2)$$

$$(R^2 = 0.785; R^2\text{ajust} = 0.570)$$

$$Y_{increase} = -9.89 + 0.282T - 0.0014T^2 + 0.176t - 0.0006t^2 - 0.0016(t \times T) \quad (3)$$

$$(R^2 = 0.765; R^2\text{ajust} = 0.530)$$

In these polynomial models, the factors with significant effects ($p < 0.05$) and those having a confidence range smaller than the value of the effect, or smaller than the standard deviation (data not shown) were included in the model (Haaland, 1989). In fact, these later effects have a lower probability, but their values are not small enough to be neglected. When these factors were removed, a lack of fit of the model was observed. All models have determination coefficients (R^2) higher than 0.75, indicating a good fit to the experimental data (Haaland, 1989).

Response surfaces presented in Fig. 3 show that the minimum oil contents in the press cake, and consequently the highest oil

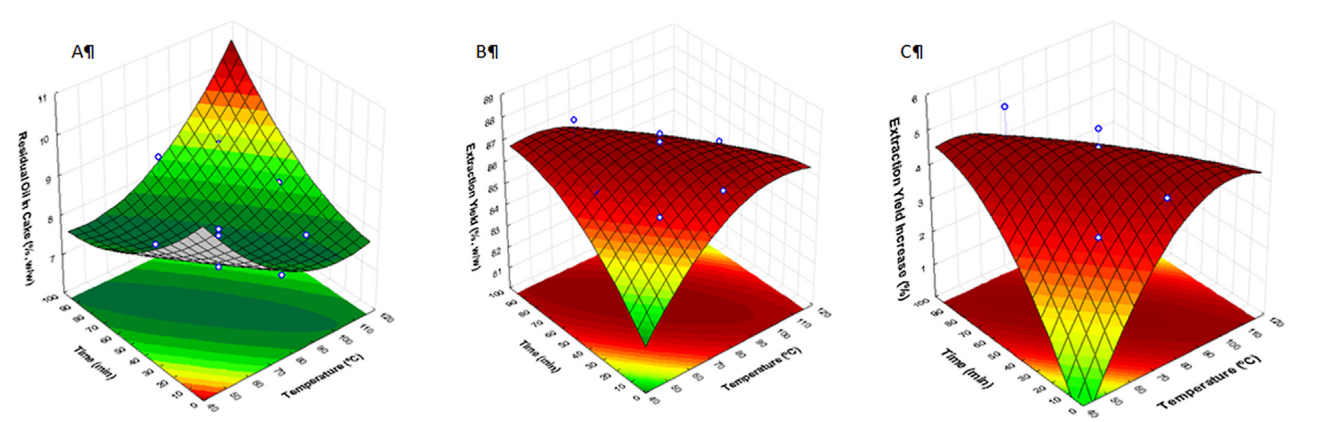

Fig. 3. Surface responses fitted to (A) the residual oil content in the pressing cake (%, w/w), (B) extraction yield (%, w/w) and (C) extraction yield increase (%) as a function of temperature and time of conditioning.

Table 2
CCRD: experimental conditions used in each conditioning experiment and the results obtained.

Experiment	Temperature (°C)	Time (min)	Residual oil(%, w/w)	Oil extractionyield (%)	Oil extraction yield increase (%)
1	60	20	8.2	86.3	3.6
2	60	80	7.3	88.0	5.6
3	100	20	8.0	86.8	4.2
4	100	80	8.8	85.2	2.3
5	51.7	50	8.2	86.3	3.6
6	108.3	50	8.3	86.1	3.4
7	80	7.6	7.8	87.1	4.5
8	80	92.4	8.7	85.5	2.6
9 (C)	80	50	7.5	87.7	5.3
10 (C)	80	50	7.7	87.2	4.4
11 (C)	80	50	7.9	87.0	4.7

yields (ca. 88%, w/w), are obtained with treatments at higher temperatures and shorter periods of time (e.g., 90 °C/10–20 min; 80 °C/30 min) or at lower temperatures and longer periods of time (e.g., 60 °C/50–60 min).

3.3. Comparison with other studies

Oil extraction from Jatropha seeds by mechanical screw presses has been studied by several authors. In most of these studies, no seed conditioning was performed and the optimization was focused on press type and/or pressing conditions (Beerens, 2007; Evon et al., 2013; Kabutey et al., 2010; Karaj and Müller, 2011; Tambunan et al., 2012).

Tambunan et al. (2012) observed that crushing the kernel of Jatropha seeds before extracting the oil in specially designed laboratory-scale mechanical extractor increased oil yield and extraction efficiency. In our study, seeds were previously crushed by a hammer before mechanical extraction.

Beerens (2007) tested two mechanical screw presses, a mechanical cylinder press and a strainer press, for oil extraction from Jatropha seeds. Maximum oil recovery was 79% for screw press (single pass) and 88% for strainer press, after dual passing. Karaj and Müller (2011) used Jatropha seeds for an oil extraction optimization study with a screw press. In this study, two different screws with two different press cylinders were tested using three different nozzles and three rotational speeds (low, medium and high). The highest oil recovery was 89.4% (w/w) and obtained at the lowest rotation speed (Karaj and Müller, 2011).

In another study carried out by Evon et al. (2013), tests were performed for the expression of Jatropha seeds using a co-rotating twin-screw extruder. Maximum oil recovery of 71% was achieved under 153 rpm screw rotation speed, 5.16 kg/h inlet flow rate of Jatropha seeds, and 80 °C pressing temperature.

Dried Jatropha seeds were used in a study carried out by Kabutey et al. (2010) on the effect of moisture content of seeds on extraction yield. Two batches of Jatropha seeds with different moisture content (8.9% d.b. and 11.1% d.b.) were used with a mechanical screw press. The results showed that *Jatropha curcas* seeds with low moisture content produced higher amount of oil compared to seeds with high moisture content.

In our study, the oil extraction yield attained with seeds without conditioning and 7.2% moisture content (d.b.) and using a laboratory screw press (83.3%, w/w) is similar or higher than the values reported in the literature.

With dry heat conditioning of Jatropha seeds, Pradhan et al. (2011) investigated the effects of initial moisture content of seeds, conditioning temperature and time on the oil yield obtained using a screw press. When seeds with a moisture level of 9.7% d.b. were treated at 110 °C for 10 min, the highest oil yield (73.1%) was obtained representing an oil recovery 7% higher than that of untreated seeds.

In our studies, the maximum oil extraction yield increase (ca. 6%) was observed for heat treatments at temperatures ranging from 60 to 90 °C. This is not very different from the results obtained by Pradhan et al. (2011) at 110 °C, although the lower conditioning temperatures will preserve the quality of the extracted oil.

4. Conclusions

Mechanical extraction of oil from *Jatropha curcas* L. seeds is the most adequate technique in developing countries. To optimize oil extraction yield, the seeds must be previously crushed with a hammer mill to particles smaller than 2 mm diameter and submitted to a dry heating treatment (conditioning). High extraction yields of 88% can be attained with treatments at high temperatures and short time periods (e.g., 90 °C/10–20 min; 80 °C/30 min) or at low temperatures and longer time periods (e.g., 60 °C/50–60 min). The decision about the binomial time/temperature conditions to be used may be based on economical issues.

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