Industrial Crops and Products 83 (2016) 614–619

Contents lists available at ScienceDirect

Industrial Crops and Products

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

# 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*

ARTICLE INFO

*Article history:*
Received 13 September 2015
Received in revised form
17 December 2015
Accepted 21 December 2015
Available online 6 January 2016

*Keywords:*
Conditioning
*Jatropha curcas* L.
Mechanical oil extraction
Response Surface Methodology
Seeds

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).

http://dx.doi.org/10.1016/j.indcrop.2015.12.062
0926-6690/

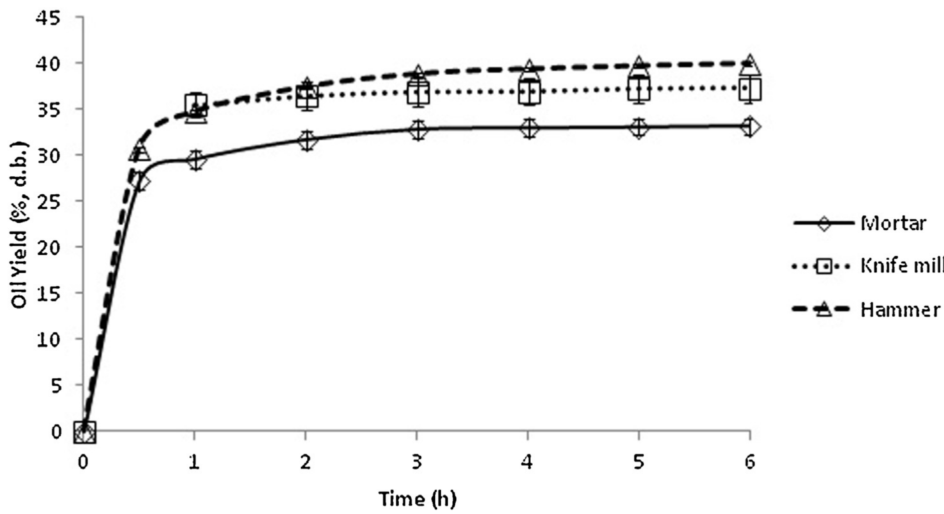


**Fig. 1.** Average amount of oil extracted (%, dry basis) over time for different types of milling (mortar, hammer and knife mill).

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