Optimization of Jatropha curcas pure plant oil production
Subroto, Erna
Chapter 3

Mechanical extraction of oil from *Jatropha curcas* L. kernel: effect of processing parameters

Erna Subroto, Robert Manurung, Hero Jan Heeres, Antonius Augustinus Broekhuis

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Abstract

Mechanical extraction is considered to be the best option for oil expression of *Jatropha curcas* in rural areas. Lab scale hydraulic pressing experiments were conducted to investigate the effect of process parameters on oil recovery from jatropha kernel. The ranges of pressing parameters investigated were: compression speed, 0.05–2.5 MPa/s; applied pressure, 5–25 MPa; moisture content, 1–6%; pressing temperature, 25–105 °C; pressing time, 1–30 min; shell removal, 0–100%; preheating time, 0–30 min; and particle size, fine, coarse and whole kernel. Chemical analyses such as acid value, phosphorus content, oxidative stability index and water content were carried out to determine the quality of the oil. Moisture content was found to influence oil recovery at any applied pressure and pressure rates. Oil recovery increased to some extent with an increase in temperature or pressing time. The preferred moisture content was found to be about 4% w.b. The presence of jatropha shell and size reduction of the kernel reduce oil recovery. The optimum oil recovery with respect to time of pressing and oil quality was obtained when jatropha kernel was pressed at 15 MPa, 90 °C, 4% w.b. moisture content for 10 min of pressing. The oil recovery obtained at these processing conditions was 86.1%.

**Keywords:** *Jatropha curcas* L., oilseed extraction, hydraulic press, oil recovery, oil quality, process parameters
3.1 Introduction

People living in the rural areas of developing regions often lack access to energy sources. An approach to provide the required energy is to enable the generation of energy from local resources. One of the most promising renewable and independent energy sources in rural areas is jatropha oil [1-3]. As it is a non-edible oil, it will not impair food security issues [4], and, as it can grow well on marginal non-agricultural land, it will not compete with the land needed either for food production or with nature conservation [3-5]. Jatropha is considered a more sustainable feedstock for energy production than any other food-based crop such as palm, rapeseed, soybean and sunflower [4,5].

Different techniques have been used to extract oil from Jatropha curcas seed or kernel, the most notable being mechanical pressing [6,7], solvent extraction [8], enzyme assisted extraction [9], and more recently, supercritical fluid extraction [10]. Mechanical pressing is considered as the preferred method to extract oil from seed especially in the rural areas of developing regions [5]. The other methods provide too much complexity for application in rural areas.

Previous research on other plant oil seeds has shown that both seed pretreatment and pressing parameters influence oil recovery [11,12]. 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 [12]. The effect of moisture content on oil recovery also appears to be related to the type of feedstock, as each oilseed seems to have a specific optimum moisture content [11-13].

Acid value, phosphorus content and oxidative stability are the properties used to determine the quality of the oil expressed. Each of these can be related to pretreatment conditions or to the pressing operation which leads to the general conclusion that deleterious effects such as long processing times, contact with oxygen, exposure to high temperatures, light and catalysts should be avoided [14]. Willems et al. (2008) [12] reported oil yields for jatropha seed and kernel pressed at various pressures and temperature. While, Tambunan et al. (2012) [15] studied the influence of crushing, preheating time and pressing temperature on yield from jatropha seed and kernel. Both studies reported a higher oil recovery from kernel than from complete seeds.

Removal of shell and oil extraction from jatropha kernel is necessary because the shell absorbs oil during expression which results in lower oil recovery when pressing the whole seed [12]. Jatropha shell does not contain significant amounts of oil (0.5–1.4% d.b.) [16] and the seed has higher hardness and energy for rupture (50–300 N/mm; 25–95 N mm) than the kernel (10–40 N/mm; 7–28 N mm) [17]. Thus expression of seed will required more energy
in comparison with kernel. Extraction from kernel produces a light-colored, low-fiber, and protein-rich cake. In addition, there are several potential applications of jatropha seed shell, for example as particle board [18], activated carbon [19], pyrolysis oil [20], fuel for combustion units [21], and gasifier feedstock [22] which will add some economic value. Literature covering the influence of pretreatment and operating conditions during hydraulic pressing of jatropha oil from kernel is limited. This research therefore is aimed to study the influence of process parameters on oil recovery from jatropha kernel in more detail. Compression speed, applied pressure, moisture content of sample, pressing temperature, duration of pressing, feedstock size reduction, shell removal and preheating time were studied as variables, and the quality of the obtained oils was evaluated.

3.2 Material and methods

3.2.1 Materials

The experiment was conducted using *Jatropha curcas* L. seeds 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. Jatropha shells were removed manually and both the kernels and shells were analyzed for initial moisture and total oil content. In addition to jatropha from Kalimantan, jatropha from Cape Verde was used for oil quality analysis (see Table 1).

**Table 1** Properties of jatropha sample before moisture conditioning

<table>
<thead>
<tr>
<th>Properties</th>
<th>Jatropha Kalimantan</th>
<th>Jatropha Cape Verde</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight fraction, % d.b.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Seed</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>kernel</td>
<td>63.6</td>
<td>62.9</td>
</tr>
<tr>
<td>shell</td>
<td>36.4</td>
<td>37.1</td>
</tr>
<tr>
<td>Oil content, % d.b.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>seed</td>
<td>37.1</td>
<td>36.5</td>
</tr>
<tr>
<td>kernel</td>
<td>58.3</td>
<td>58.0</td>
</tr>
<tr>
<td>shell</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Moisture content, % d.b.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>seed</td>
<td>8.6</td>
<td>6.9</td>
</tr>
<tr>
<td>kernel</td>
<td>7.0</td>
<td>5.3</td>
</tr>
<tr>
<td>shell</td>
<td>11.3</td>
<td>9.6</td>
</tr>
</tbody>
</table>
The harvesting and post harvesting history for the jatropha from Cape Verde is unknown. The seeds were imported into the Netherlands in February 2009. After transport to the Netherlands, both seeds were stored at a temperature in the range of 15–25 °C and at a relative humidity of 40–50%. The seed shells were removed manually and the kernels were exposed to some pretreatments before being pressed (described below). The pretreated kernels were used directly in the pressing experiments to reduce the influence of storage time and condition on oil quality. The oil analyses were conducted directly after pressing in May 2011 and September 2011 for jatropha Kalimantan and Cape Verde, respectively. Figure 1 shows the representative of seed, kernel and shell of jatropha.

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).

Figure 1 Parts of jatropha: a. seeds, b. kernels, c. shells

Kernel pretreatments

Jatropha kernels were given some pretreatments before being pressed. Those were moisture conditioning, shell removal and/or size reduction for kernel.

Shell removal

In the experiments to study the effect of shell removal, the total weights of samples (shells and whole kernels) were approximately 7 g each. The moisture content of kernel and shell were 4 and 8.4% w.b., respectively. At these moisture contents, seed contains 62.5% w.b. kernel and 37.5% w.b. shell. The stated percentage of shell removal is the percentage of shell removed from the original sample. 0% removal means that the experiment uses 100% seeds or corresponds to a shell content of 37.5% w.b., while 100% removal means that the experiment uses 100% kernels or corresponds with a shell content of 0% w.b. The oil recovery was calculated on the basis of oil content and weight of kernel (shell contains no
Experiments were carried out with removal of 0, 20, 40, 60, 80 and 100% of the shell. Unless stated otherwise, the samples consist of whole kernel or 100% shell removal.

**Moisture conditioning**

For quantitative analysis, the whole kernels were conditioned by oven drying. The drying temperature were 30, 35, 40, 50, 60, and 70 °C for desired moisture content of 6, 5, 4, 3, 2 and 1% w.b., respectively. After drying, the kernels were wrapped tightly in a low density polyethylene bag of 25 μm thickness and then put inside a desiccator containing silica gel for 24 h before being pressed. For oil quality analysis, the kernels were equilibrated inside the desiccator containing silica gel or water until they reach the desired moisture content and then wrapped in the polyethylene bag. The initial moisture content of the sample was determined by using the standard oven method at 105 °C for 24 h. Moisture contents after conditioning were determined based on the weight difference of the sample after and before conditioning. Unless stated otherwise, whole kernel with a moisture content of 4% w.b. was used.

**Size reduction**

After moisture conditioning, the whole kernels were grinded into two particle sizes: a finely ground sample [1.17-2.36 mm] and a coarsely ground sample [3.33-4.75 mm]. Unless stated otherwise, whole kernel was used.

### 3.2.2 Hydraulic pressing

A specially designed laboratory hydraulic press was used to study the effect of processing parameters on oil recovery from the jatropha samples. A schematic representation of the press is shown in Figure 2. The pressing chamber is made from stainless steel with a diameter of 20 mm and a height 70 mm. It is equipped with a perforated plate (diameter hole of 1 mm) covered with stainless steel (SS) wire mesh (100 mesh) placed at the bottom of the pressing chamber acting as filter during extraction. An electrical-resistance heating ring being attached around the pressing chamber is used to preheat the pressing chamber during operation within a temperature range of 30–105 °C. For expression at 25 °C, the heating ring was removed. Pressures up to 25 MPa were applied by a hydraulic plunger. The press is equipped with a thermocouple (±1 °C), pressure measurement (±1 MPa), and a level indicator (±0.01 mm), which measures the distance the plunger traveled.
Approximately 7 g of pretreated sample was placed in the pressing chamber. Afterwards, the plunger is put on top of the sample. Unless stated otherwise, the sample is preheated at the desired pressing temperature for 5 min. Preheating time is the duration of the sample at the pressing temperature, before it was pressed for actual oil extraction. The pressure is increased linearly at the predefined compression speed until the desired pressure is reached. Unless stated otherwise, the sample is pressed at a compression speed of 0.125 MPa/s. Total pressing time is 10 min, except for the experiments showing the influence of pressing time. Three replicate measurements were performed for each sample and average values were taken.

3.2.3 Total oil content and oil recovery

The oil content of the sample was determined using standard Soxhlet extraction methods. The kernels were dried overnight in an oven at 105 °C before analysis. The dried kernels were grinded using a coffee grinder (Princess 242195, Netherland) and approximately 10 g samples were transferred into a cellulose extraction thimble (Whatman, I.D.xH 18x55 mm, GE). This was extracted with 100 ml n-hexane at its boiling point for 24 h. The solvent was removed using a rotary vacuum evaporator (100 mbar 40 °C). The total oil content is
calculated on a dry basis of the sample. The oil recovery is defined as the ratio of the amount of oil expressed to the total oil contained in the sample on dry weight basis.

### 3.2.4 Oil quality analysis

Hydraulic pressed oils which still contained some fine particles were centrifuged at 2,000 x G for 10 min. The clear oils were recovered and used for oil analysis. Chemical analyses of the samples were carried out according to the standard test methods: DIN EN 14104, DIN EN ISO 12937, DIN EN 14112 and DIN EN 14107 for acid value, water content, oxidative stability and phosphorus content, 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 of chemical property analysis of plant oil samples was conducted in our laboratory with the exception of phosphorus content analyses which were conducted by ASG Analytik-Service GmbH, Germany. Duplicate measurements were performed on each sample and average values were taken.

### 3.3 Results and discussions

#### 3.3.1 Influence processing factors on oil recovery

Jatropha kernel from Kalimantan used in this experiment has a 58.3% d.b. oil content and an original moisture content of 7% d.b. (see Table 1) The ranges of parameters investigated were: applied pressure, 5–25 MPa; moisture content, 1–6% w.b; pressing temperature, 20–105 °C; pressing time 2–30 min; shell removal, 0–100%; preheating time, 0–30 min; and, particle size, fine, coarse and whole kernel.

**Effect of compression speed**

Industrial expression generally applies a linear pressure increase during the early phase of pressing followed by a period of constant pressure. The compression speed for jatropha extraction is studied at three levels, namely 2.5, 0.125 and 0.05 MPa/s. The compression speed in this study is defined as the speed until reaching the final pressure. The time needed to reach its final pressure of 10 MPa at a compression speed of 2.5, 0.125 and 0.05 MPa/s were 4, 80 and 200 s, respectively. Increasing the speed from 0.05 to 2.5 MPa/s reduced the oil recovery from 78.2 to 67.2% d.b. for jatropha kernel with a moisture content of 4% w.b (see Table 2).
At higher compression speeds, the oil initially released into the inter-kernel voids experiences resistance to flow through low-porosity compressed cake due to compaction of the cake at the beginning of pressing operation. This reduces the porosity and restricts the flow of oil. Increasing pressures can help to push the oil out. As shown in Table 2, when the applied pressure is increased from 10 to 20 MPa at high compression speeds (2.5 MPa/s), an increase in oil recovery was observed from 67.2 to 73.2% d.b. At low compression speed, slow deformation maintains the porosity needed for free oil flow until it reaches the maximum consolidation point. By tuning the compression speed, a high oil recovery at lower applied pressure can be achieved.

Table 2 The effect of compression speed at various applied pressures

<table>
<thead>
<tr>
<th>Compression speed (MPa/s)</th>
<th>Oil recovery (% d.b.)</th>
<th>10 MPa</th>
<th>15 MPa</th>
<th>20 MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td></td>
<td>78.2±1.3</td>
<td>81.4±1.0</td>
<td>81.9±1.2</td>
</tr>
<tr>
<td>0.125</td>
<td></td>
<td>75.3±0.6</td>
<td>79.8±1.1</td>
<td>81.2±1.2</td>
</tr>
<tr>
<td>2.5</td>
<td></td>
<td>67.2±1.3</td>
<td>71.6±1.4</td>
<td>73.2±1.7</td>
</tr>
</tbody>
</table>

*Jatropha kernel with moisture content of 4% w.b. pressed at 60 °C for 10 min.*

The oil recovery profiles for pressing jatropha kernel with moisture contents of 4% w.b. are recorded as a function of pressing time at three different compression speeds (Figure 3). Oil recovery slowly increased and finally reached the maximum value when jatropha kernel was being pressed at 15 MPa with compression speed of 2.5 MPa/s. Meanwhile, almost 90% of oil (from the total oil expressed after 10 min of pressing) was recovered after 4 min of pressing at 0.125 MPa/s. When jatropha kernel is being pressed at a compression speed of 0.05 MPa/s, there is no oil recovered during the first minute of pressing. The oil recovery profile seems similar for the compression speeds of 0.125 MPa/s and 0.05 MPa/s, except for 0.05 MPa/s where there was 1 min delay due to the longer time needed to reach the oil point pressure. Oil point pressure is defined as the value of applied pressure at which oil flows out of the kernel particles or the minimum pressures need for mechanical oil expression [23-24]. There is no significant difference in oil recovery for pressing jatropha kernel at compression speeds of 0.125 MPa/s and 0.05 MPa/s.
Figure 3 Oil recovery profiles of jatropha kernels pressed at different compression speeds
(moisture content of kernels: 4% w.b.; applied pressure: 15 MPa; pressing temperature: 60
°C; 10 min pressing)

The effect of compression speed was evaluated at three different moisture contents,
namely 1, 4 and 6% w.b. At a moisture content of 6% w.b., oil recovery reduced from 71.4 to
41.8% d.b. with increasing the compression speed from 0.05 to 2.5 MPa/s. While at the
moisture content of 4% w.b, increasing the speed from 0.05 to 2.5 MPa/s reduced the oil
recovery from 81.4 to 71.6% d.b. However, this oil recovery reduction did not happen when
the dried kernel was pressed, for example at 1% w.b., the oil recovery remained more or
less constant at a non-optimum level of around 63-64% d.b. as shown in Table 3. Our data
indeed confirmed work reported by Willems et al. (2008) [12], who concluded that the
effect of compression speed did not affect oil recovery when using relatively dry samples
(moisture content of 0% w.b.). Yet, the conclusion appeared not to be valid for higher
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(moisture content of 0% w.b.). Yet, the conclusion appeared not to be valid for higher moisture contents, as found in this study.

Table 3 The effect of compression speed at various moisture contents

<table>
<thead>
<tr>
<th>Compression speed (MPa/s)</th>
<th>Oil recovery (% d.b.)</th>
<th>1% w.b.</th>
<th>4% w.b.</th>
<th>6% w.b.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td></td>
<td>63.5±1.5</td>
<td>81.4±1.0</td>
<td>71.4±1.2</td>
</tr>
<tr>
<td>0.125</td>
<td></td>
<td>64.5±1.3</td>
<td>79.8±1.1</td>
<td>65.1±0.7</td>
</tr>
<tr>
<td>2.5</td>
<td></td>
<td>62.9±1.3</td>
<td>71.6±1.4</td>
<td>41.8±1.7</td>
</tr>
</tbody>
</table>

a Jatropha kernel pressed at applied pressure of 15 MPa at 60°C for 10 min.

The effect of moisture content on the oil recovery could be explained by the ability of oil and water to wet the surface of the seed particles. Water is known to act as an interfacial agent between the protein-rich cake and the oil, which forms paste-like plastized material. A lower compression speed helps in continuous oil and water removal from the seed and prevents excessive mixing of these fractions with the protein-rich cake. High moisture content of kernel needs slower deformation for optimum oil expression.

Effect of moisture content and applied pressure

The effects of applied pressure and moisture content on oil recovery are shown in Figure 4. A comparative study of the results showed that the oil recovery at any pressure was affected by the moisture content of the sample.

The oil recovery increases from a low value at the moisture contents of 1% w.b. to reach a maximum value at moisture contents of between 4 and 5% w.b. for all applied pressures. Thereafter the oil recovery decreases with a further increase in moisture content for all extraction pressures. For example, at an applied pressure of 15 MPa, oil recovery increases from 64.5 to 79.8% d.b. with an increase in moisture content from 1 to 4% w.b. and then decreased to 65.1% d.b. with a further increase of moisture content to 6% w.b. In addition, it was observed that at a moisture content of 6% w.b., the oil was contaminated with fine particles containing water which made the obtained oil cloudy. As reported by Karaj and Müller (2010) [17], the presence of moisture influences the hardness of jatropha kernel with a higher hardness at lower moisture content. However, at excess moisture level, the presence of water will act as plasticizer between the protein-rich cake and oil which forms paste-like plastized material. It seems that optimum moisture levels shift to lower values as pressures increase. At low pressure, the optimum moisture content is 5% w.b. while at
higher pressure the optimum moisture content is 4% w.b. This observation is in agreement with the work carried out by Mpagalile and Clarke (2005) [11]. In order to get higher recoveries it would be preferable to extract oil at moisture contents of jatropha kernel close to 4% w.b. Acheheb et al. (2012) [13] found 3.95% as optimum moisture content for pistachio nut.

Figure 4 Effect of applied pressure and moisture content on oil recovery of jatropha kernel pressing at 60 °C for 10 min

Effect of moisture on oil recovery is more pronounced at low pressure. As the applied pressure increases, the effect of moisture content becomes more negligible. At applied pressure of 5 MPa, the oil recovery varied between 44.0 to 67.9% d.b. for kernel with moisture content of 1 and 5% w.b., respectively. While at 25 MPa, the oil recovery ranged from 74.7 to 81.6% d.b. for kernel with moisture content of 1 and 4% w.b., respectively. Except for pressing high moisture content of 6% w.b., where the effect of pressure is negligible. Due to the low pressure used, oil recoveries are influenced by hardness of the kernel, giving higher oil recovery at higher moisture. As the pressure increases, maximum cake consolidation is achieved regardless of moisture content used. Pressing at 25 MPa is almost insensitive to moisture (below 5% w.b.) which makes it ideal for commercial operations as the moisture content of the feedstock may vary over a wider range while still
a high recovery can be achieved. This observation is in agreement with the work carried out by Mpagalile and Clarke (2005) [11] who studied the effect of moisture content on oil recovery from coconut at various applied pressures.

Generally, there was an increase in oil recovery as pressure increased from 5 MPa to 25 MPa. Increasing the applied pressure leads to the increase of oil recovery when jatropha kernel was pressed up to 3% w.b. moisture content. Beyond this moisture content, the oil recovery increases as the pressure increases to 15 MPa and either leveled off or decreased when pressure is raised to 25 MPa. A reduction in oil recovery with a pressure increase to 25 MPa was observed and the effect is more noticeable in samples with high moisture contents. At higher pressure, the empty voids between particles from which the oil could flow were becoming smaller and sealed, and thus restricted the flow of oil. These results indicate that excessive pressure does not necessarily have a positive influence on oil recovery.

The oil recovery increment as the result of pressure increase varied with moisture content. At a moisture content of 1% w.b., increase pressure from 5 MPa to 25 MPa gave an oil recovery increment as much as 30.7%; i.e. from 44.0 to 74.7%. Meanwhile, at moisture content of 6% w.b, the oil recovery shifted from 62.5 to 65.1% as pressure increased from 5 to 15 MPa and decreased to 61.8% as pressure increased to 25 MPa. Moisture affects the hardness and compactness of the kernel. At lower moisture contents, moisture loss causes the surface of the sample to harden, and it requires a higher applied pressure to break the hardened surface during pressing. Consequently, the increasing pressure leads to the increase of the oil recovery. Meanwhile, at higher moisture contents, the water acts as a interfacial agent between the protein-rich cake and oil which forms paste-like plastized material thus restricting the flow of oil. Increasing pressure at higher moisture content increased the compactness of cake, thus reduce the oil recovery.

**Effect of pressing temperature and time**

The effect of pressing temperature and time on oil recovery is presented in Figure 5. The effect of these parameters on the oil recovery was studied at 15 MPa and moisture content of 4% w.b. Temperature is found to affect the oil recovery of jatropha oil which is in agreement with earlier studies on the effect of temperature on oil recovery from oilseeds [11,12,25]. As temperature increases, oil recovery increases but tends to level off at higher temperature. For example at a pressing time of 10 min, oil recovery increase from 58.6 to 87.2% as temperature rise from 25 to 105 °C. It was observed that at 105 °C, the color of oil became darker and the resultant cake was dry and hard. Increasing temperature raised oil recovery due to breakdown of cell walls, caused protein denaturation and coagulation, and reduced oil viscosity, thus facilitating the release of the oil from the cells into the inter-
kernel voids [12]. However, increasing pressing temperature reduced the oil recovery increment. This phenomenon could be attributed to the fact that water evaporates faster at higher temperature causing substantial moisture loss; the compressed cake becomes hard and dry thus reducing the oil flow through the compressed cake. This observation is in agreement with the work carried out by Mpagalile and Clarke (2005) [11] and Willems et al. (2008) [12].

![Graph showing oil recovery vs. pressing time and temperature](image)

**Figure 5** Effect of pressing temperature and time on oil recovery from jatropha kernel with moisture content of 4% w.b. pressing at 15 MPa

The influence of pressing time is shown in Figure 5. In general, oil recovery increased with increasing pressing time at all pressing temperatures. Oil recovery increased progressively when the pressing time was increased from 1 to 10 min. Upon increasing pressing time up to 30 min, oil recovery gradually leveled off to a constant value depending on the pressing temperature used. For example at temperature of 90 °C, oil recovery increased to 86.1% after 10 min and finally reached 94.1% after 30 min of pressing. The amount of oil extracted increased with pressing time with more than 50% (of the total expressed oil after 30 min of pressing) recovered in 1 min and about 90% after 10 min of pressing. It is likely that an increase in the pressing time at the applied pressure led to slow cake deformation and compaction of the cake. This caused restricted flow of the oil from the cake and a reduction of the oil expression [11].
Effect of shell removal

Mechanical separation of shell from kernel potentially could not achieve full separation. Therefore, the effect of shell is investigated to determine whether its presence has an effect on oil recovery. A comparative study showed that an increase in the amount of shell reduced oil recovery (see Table 4).

Table 4 Effect of shell removal on oil recovery

<table>
<thead>
<tr>
<th>Shell removal (% w.b.)</th>
<th>K (g)</th>
<th>So(^b) (g)</th>
<th>S(^c) (g)</th>
<th>Kernel (% w.b.)</th>
<th>Shell (% w.b.)</th>
<th>Oil recovery (% d.b.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>7.00</td>
<td>4.20</td>
<td>0</td>
<td>100</td>
<td>0</td>
<td>79.8±1.1</td>
</tr>
<tr>
<td>80</td>
<td>6.25</td>
<td>3.75</td>
<td>0.75</td>
<td>89.3</td>
<td>10.7</td>
<td>81.7±1.1</td>
</tr>
<tr>
<td>60</td>
<td>5.65</td>
<td>3.39</td>
<td>1.36</td>
<td>80.6</td>
<td>19.4</td>
<td>76.1±0.6</td>
</tr>
<tr>
<td>40</td>
<td>5.15</td>
<td>3.09</td>
<td>1.85</td>
<td>73.5</td>
<td>26.5</td>
<td>73.8±1.4</td>
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<tr>
<td>20</td>
<td>4.73</td>
<td>2.84</td>
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<td>67.6</td>
<td>32.4</td>
<td>68.3±1.4</td>
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<tr>
<td>0</td>
<td>4.38</td>
<td>2.63</td>
<td>2.63</td>
<td>62.5</td>
<td>37.5</td>
<td>54.2±0.7</td>
</tr>
</tbody>
</table>

Note:
\(^a\) Jatropha sample (moisture content of seed: 5.6% w.b., kernel: 4% w.b., shell: 8.4% w.b.) pressed at 15 MPa at 60 °C for 10 min.
\(^b\) Originally Shell weight (So) = 37.5/62.5 x kernel weight (K).
\(^c\) Shell weight after removal (S) = (100 - % shell removal) x originally shell weight / 100.

Extraction of neat kernel (100% shell removal) gave a 25.6% higher oil recovery than extraction of seed (0% shell removal). However, the highest oil recovery is achieved at 80% shell removal. Similar results were obtained by Willems et al. (2008) [12], with oil recoveries of 33-52% for dried jatropha seeds and 62-75% for dried jatropha kernels pressed at 20 to 70 MPa, 40 °C for 10 min. According to this author, jatropha shell contains no oil; the presence of a significant quantity of shell may lead to absorption of oil by the fiber in the shell, thereby reducing oil recovery. In addition, when using seeds, the shells consume part of the pressure, whereas for kernel, all the pressure is used to express the oil. The presence of small quantities of shell helps to maintain porosity in the sample thus facilitates the flow of oil through the compressed cake.

Effect of particle size

Three sizes of samples were used to investigate the effect of size reduction on oil recovery, i.e. whole, coarsely ground (3.33-4.7 mm), and finely ground kernel (1.17-2.36 mm); Table 5
summarizes the effect of size reduction of jatropha kernel. The oil recoveries from whole, coarsely and finely ground kernel are 74.5, 71.8 and 69.3% d.b. at 10 MPa and 81.3, 77.5 and 75.1% d.b. at 20 MPa, respectively. Whole jatropha kernel gave a higher oil recovery than coarsely and finely ground at all applied pressure levels. Conflicting results have been reported by Tambunan et al. (2012) [15] with a significantly higher oil recovery for coarse samples than for whole jatropha samples. The higher oil recovery for whole jatropha kernel compared to the other sizes can be explained by the bigger voids formed by the whole kernel samples compared to those of the coarsely and finely ground ones. Despite the larger surface area exposed to pressure and heat treatment and the increased number of ruptured cells, which should result in a high oil concentration at the particle surface, the smaller inter-particle voids restrict the flow of oil through the compressed cake. Pressing whole kernel provides a better porosity in the cake which enables better oil flow.

**Table 5** Effect of particle size on oil recovery

<table>
<thead>
<tr>
<th>Applied pressure (MPa)</th>
<th>Oil recovery (% d.b.)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>whole</td>
</tr>
<tr>
<td>10</td>
<td>75.3±0.6</td>
</tr>
<tr>
<td>20</td>
<td>81.2±1.2</td>
</tr>
</tbody>
</table>

*a* Jatropha whole kernel with moisture content of 4% w.b. pressed at applied pressure of 10 and 20 MPa at 60 °C for 10 min.

**Effect of preheating time**

To evaluate the effect of preheating, jatropha kernel is preheated inside the pressing chamber. In this experiment, it is observed that 5 min of preheating is sufficient to increase oil recovery from 77.8 to 79.8%. Further increases of the time to 30 min did not significantly raise the oil recovery (see Table 6). Initial moisture content before pre-heating was 4% w.b. while moisture content after pre-heating were 3.92, 3.80 and 3.66% w.b. for 5, 15 and 30 min preheating, respectively. Apparently, longer preheating at this condition has no significant influence on oil recovery due to hardening of the kernel surface and moisture loss. This is in agreement with the results obtained by Tambunan et al. (2012) [15] in their study on the effect of preheating time on jatropha pressing.
Table 6 Effect of preheating time on oil recovery

<table>
<thead>
<tr>
<th>Preheating time (min)</th>
<th>Oil recovery (% d.b.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>77.8±0.6</td>
</tr>
<tr>
<td>5</td>
<td>79.8±0.6</td>
</tr>
<tr>
<td>15</td>
<td>80.7±0.5</td>
</tr>
<tr>
<td>30</td>
<td>81.2±0.8</td>
</tr>
</tbody>
</table>

*a* Jatropha whole kernel with moisture content of 4% w.b. pressed at applied pressure of 15 at 60 °C for 10 min.

### 3.3.2 Quality parameters

Some important oil quality criteria for jatropha oils are presented in Table 7. Acid value reflects the free fatty acids (FFAs) and other possible organic acids. FFAs are a result 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. The oxidative stability index is associated with the oxidative degradation of oil which results in rancidity development. A higher Oxidative Stability Index (OSI) expressed in hours Induction Period (IP) means that the oil is more resistance to oxidation. Phosphorus content indicates the presence of phosphor-derived components in oil such as phospholipids and phytates.

In general, jatropha oil from Kalimantan has a higher phosphorus content compared to jatropha oils from Cape Verde as shown in Table 7. The soil nutrition or fertilizer is considered to be contributing to this result. According to Lickfett et al. (1999) [26], phosphorus content in seed is affected by the level of phosphorus fertilizer supply or phosphorus content in soil. It is possible that jatropha from Kalimantan is planted on better nutritious land than the one from Cape Verde.

The acid values of jatropha oils from Cape Verde were higher than those for jatropha oils from Kalimantan, while the oxidative stability of the former was lower. This is probably due to the longer storage time of seed. Jatropha Kalimatan is freshly harvested seed, while jatropha from Cape Verde has been stored at ± 7% w.b. moisture content, within a temperature range of 15-25 °C and at a relative humidity of 45% for minimum 2.5 years. According to Murthy et al. (2003) [27], the quality of oil depends on the way the seeds are dried, treated and stored. Storage conditions, such as moisture content of seed during storage and temperature also have been reported to contribute to the final oil quality. Worang et al. (2008) [28] reported the acid value increased from 0.1 to 1.1 mg KOH/ g oil after jatropha seed is stored at 8% moisture content, a temperature of 26 °C, and a relative humidity of 70% for 6 months. Other studies showed similar results, with hydrolytic and oxidative degradation related to time of storage and storage conditions [29,30]. Factors
such as initial moisture content, storage temperature and/or humidity increase the rate of seed deterioration.

Table 7 Jatropha oil quality at different processing conditions and varieties

<table>
<thead>
<tr>
<th>No</th>
<th>Processing conditions</th>
<th>Oil quality</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>AV (mg KOH/gr oil)</td>
<td>P content (ppm)</td>
<td>OSI (hours)</td>
<td>Water (ppm)</td>
</tr>
<tr>
<td>A</td>
<td>Jatropha Kalimantan</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>whole kernel, 4% w.b., 10 MPa, 25 °C, 10 min</td>
<td>0.21</td>
<td>15.8</td>
<td>12.70</td>
<td>672</td>
</tr>
<tr>
<td>2</td>
<td>whole kernel, 2% w.b., 10 MPa, 60 °C, 10 min</td>
<td>0.17</td>
<td>19.6</td>
<td>11.55</td>
<td>406</td>
</tr>
<tr>
<td>3</td>
<td>whole kernel, 6% w.b., 10 MPa, 60 °C, 10 min</td>
<td>0.36</td>
<td>26.5</td>
<td>12.01</td>
<td>933</td>
</tr>
<tr>
<td>4</td>
<td>whole kernel, 4% w.b., 20 MPa, 60 °C, 10 min</td>
<td>0.29</td>
<td>23.5</td>
<td>13.81</td>
<td>720</td>
</tr>
<tr>
<td>5</td>
<td>whole kernel, 4% w.b., 20 MPa, 90 °C, 10 min</td>
<td>0.25</td>
<td>25.9</td>
<td>13.99</td>
<td>747</td>
</tr>
<tr>
<td>B</td>
<td>Jatropha Cape Verde</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>whole kernel, 2% w.b., 15 MPa, 60 °C, 10 min</td>
<td>1.03</td>
<td>1.4</td>
<td>8.93</td>
<td>357</td>
</tr>
<tr>
<td>2</td>
<td>whole kernel, 6% w.b., 15 MPa, 60 °C, 10 min</td>
<td>1.19</td>
<td>3</td>
<td>13.07</td>
<td>885</td>
</tr>
<tr>
<td>3</td>
<td>whole kernel, 4% w.b., 5 MPa, 60 °C, 10 min</td>
<td>1.05</td>
<td>1.6</td>
<td>7.43</td>
<td>702</td>
</tr>
<tr>
<td>4</td>
<td>whole kernel, 4% w.b., 15 MPa, 60 °C, 10 min</td>
<td>1.07</td>
<td>1.6</td>
<td>10.97</td>
<td>727</td>
</tr>
<tr>
<td>5</td>
<td>whole kernel, 4% w.b., 25 MPa, 60 °C, 10 min</td>
<td>1.07</td>
<td>1.4</td>
<td>12.04</td>
<td>771</td>
</tr>
<tr>
<td>6</td>
<td>whole kernel, 4% w.b., 15 MPa, 25 °C, 10 min</td>
<td>0.99</td>
<td>1</td>
<td>9.55</td>
<td>695</td>
</tr>
<tr>
<td>7</td>
<td>whole kernel, 4% w.b., 15 MPa, 90 °C, 10 min</td>
<td>1.03</td>
<td>3.3</td>
<td>7.25</td>
<td>755</td>
</tr>
<tr>
<td>8</td>
<td>whole kernel, 4% w.b., 15 MPa, 90 °C, 30 min</td>
<td>1.11</td>
<td>9</td>
<td>7.28</td>
<td>773</td>
</tr>
<tr>
<td>9</td>
<td>fine kernel, 4% w.b., 15 MPa, 90 °C, 10 min</td>
<td>1.16</td>
<td>6.8</td>
<td>13.44</td>
<td>838</td>
</tr>
</tbody>
</table>
Increasing moisture content of kernel from 2 to 6% w.b. increases the phosphorus content from 1.4 to 3 ppm and acid value from 1.03 to 1.19 (see Table 7, section B). According to Prior et al. (1991) [31], the phospholipid content in the oil is reduced by a decrease in seed moisture. Seeds with 5% and 9-11% moisture contained, respectively, two and three times as much phospholipid than seed with 2.5% moisture content. As a result of this rise in phosphorus content, the oxidative stability is also increasing; phospholipids and phytates are known as natural antioxidant [32,33]. Holser (2003) [34] studied the effect of moisture and temperature on the oil quality of conditioned meadowfoam (Limnanthes alba). It is reported that moisture has a stronger effect than temperature on the increase in FFA content (acid value). A higher moisture content of the seeds would enable hydrolysis of triglycerides.

Increasing pressing temperatures raises the acid values and phosphorus contents of the oil. Acid value increased with an increase in pressing temperature from 25 to 60 °C. However a slightly reduced acid value was observed when pressing temperature increased to 90 °C. Increasing temperature is also likely to promote hydrolysis as reaction rates increase at higher temperatures, however, at the higher temperatures this effect would be moderated by enzyme degradation. According to Abigor et al. (2002) [35], lipase isolated from jatropha seed shows the highest activity at a temperature of 37 °C. The disruption of cell structure and solubilization of phospholipids occurred at higher temperatures. Although phosphorus is present at a higher level when the oil is pressed at higher temperature, the oxidative stability of the oil is lower. High pressing temperature increases the rate of autoxidation and consequently it consumes the available natural antioxidants including phospholipids. This result is in accordance with the data reported by Tambunan et al. (2012) [15], showing that prolonged heat treatment at a high temperature reduces the oil’s oxidative stability and increases oil acidity.

Finely ground jatropha kernel has a higher acid value and phosphorus content than whole kernel. Adeeko and Ajibola (1990) [36] mentioned that particle size has a significant effect on acid value of oil with the finely ground nuts having a higher value. The formation of FFA in seeds starts with the destruction of cells. However, acid formation is limited to the destroyed cells during size reduction. A higher number of cells destroyed in the finely ground samples thus accounts for the higher acid value and phosphorus content. Size reduction increases exposed area to heat, moisture and oxygen. Size reduction also gave a higher oxidative stability, possibly due to the co-extraction of other natural antioxidants, such as tocopherol. High heating temperatures, long heating times and size reduction resulted in higher acid value and phosphorus content.

The presence of water can promote the hydrolysis reaction and the growth of microorganisms during storage of oil. Water content can increase during storage due to water...
absorption from air; thus proper packaging is needed to avoid water absorption [37]. A maximum water content of 750 ppm is required by German Standard DIN V 51605:2010-10.

3.4 Conclusions

From the parameters studied in this work the following can be concluded: For oil expression from kernels with high moisture content a slower compression speed is preferred. Pressure, temperature and pressing time all have positive effects on oil recovery, but negative effect on oil quality. Kernel size reduction has a negative effect on oil recovery and quality. Moisture content in the range of 4-5% w.b. gave the highest oil recovery. A minimum of 80% shell removal should be achieved to avoid the negative effect of shell presence on oil recovery. Five min of preheating time was found to be optimum in increasing the oil recovery. Jatropha kernel was pressed at 15 MPa, 90 °C pressing temperature, 4 % w.b. moisture content for 10 min of pressing gave an oil recovery of 86.1% and good oil quality. Further optimization within the pressure range of 10-20 MPa, 60-90 °C and moisture content 3-5% w.b. is expected to deliver the optimum oil recovery. This work will be presented in separated paper.
References


