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Hydraulic pressing optimization and physicochemical characterization of *Jatropha curcas* seed oil

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ABSTRACT

Jatropha curcas seed oil can be used for numerous purposes including fuel, soap and cosmetics. Better quality oil is obtained by cold pressing, the effectiveness of which is unfortunately limited by a low extraction rate. The objective of this study was to optimize cold hydraulic extraction parameters of *Jatropha curcas* seed oil by increasing the oil recovery and characterising the extracted oil. The key extraction parameters (dwell time, pressure, compression speed, and press cage charge) were determined using a laboratory hydraulic press. The results show that the unhulled and dehulled seeds contained 5.1 and 2.9% moisture, as well as 33.6 and 51.7% fat, respectively. Under the optimal operating conditions and at ambient temperature (25 °C), the oil recovery from whole (crushed) material was 38.2 (42.5)% unhulled seeds and 71.3 (69.5)% dehulled seeds. The physicochemical characteristics of cold-extracted Jatropha oil were 10ppm phosphorus, 1.9 ppm iron, 0.0 ppm copper, 0.15% water-volatile matter, 0.918 (15 °C) density, and 37.72 cSt (37.8 °C) kinematic viscosity, respectively. In conclusion, the optimized cold hydraulic extraction of jatropha seed oil leads to high oil recovery and good oil quality suitable for industrial applications.

Keywords: *Jatropha curcas* seed oil; cold pressing; process optimization; oil recovery; physicochemical characteristics

1. Introduction

Jatropha curcas is a drought-resistant perennial plant belonging to the Euphorbiaceae family and widely represented in the tropical regions of Africa, America, and Asia. This plant is generally used to form hedges and restore degraded soil. However, all parts of Jatropha can be used as, for example, pesticides, traditional medicine, energy source, green manure, and fertilizer. The oil of Jatropha seeds is non-edible due to the presence of a toxic compound (the phorbol esters). It is particularly used in the manufacture of candles, soap, the cosmetics industry, and as a raw material in the production of biodiesel [1,2]. Extraction of the seed oil is usually carried out by employing organic solvents or mechanical pressing methods. Using organic solvents is a long-time and energy-consuming process which requires high usage of hazardous chemicals. Furthermore, mechanical extraction using a screw press is relatively inefficient, leaving about 8–14% of available oil in the de-oiled cake. Preheated seeds, heated press, high pressure, and increasing seed moisture content are often used to improve oil extraction from mechanical oil expellers [3]. When pressing seeds to obtain cold-pressed oils using a hydraulic press, less heat is used. Oils pressed at a lower temperature retain their non-deteriorated antioxidants which preserve them from oxidation [4,5]. Moreover, hydraulic pressing is not expensive in investment and is generally used on a small or medium scale. It does not require highly skilled technicians to operate the machines [6]. If for most conventional oleaginous crops (olive, sunflower, palm) the optimal pressing parameters are well known, for many unconventional species (jatropha, desert date, moringa) of industrial or food interest they remain to be established or completed to boost their production. This study aims to optimize the operating parameters of cold hydraulic extraction of Jatropha seed oil and to assess the quality of the extracted oil.

2. Materials and Methods

2.1. Plant materials and chemicals

Mature Jatropha seeds have been collected in the Toma region of Burkina Faso. The seeds were sun-dried for two weeks and the final mass fraction water content was determined using the standard oven method at 105 °C for 24 h. After drying, the seeds were cleaned by winnowing to remove impurities, then packed in plastic bags and stored at room temperature of about 25 °C until used. Fifty per cent of the oilseeds were dehulled before the tests to study the effect of shell removal (**Figure 1**). The total oil content of Jatropha unhulled and dehulled seeds was determined according to Soxhlet the extraction method ISO

659:1998(F) modified by micro-grinding and two steps extraction for eight hours full time. All chemicals used in this study were of analytical reagent (AR) grade.

(a) (b)

Fig. 1: *Jatropha curcas*unhulled (a) and dehulled seeds (b)

2.2. Hydraulic pressing device and procedure

Mechanical oil extraction was carried out using a laboratory hydraulic press (CARVER-3889CE, Wabash, Indiana, USA). **Figure 2** shows a schematic representation of the experimental setup.

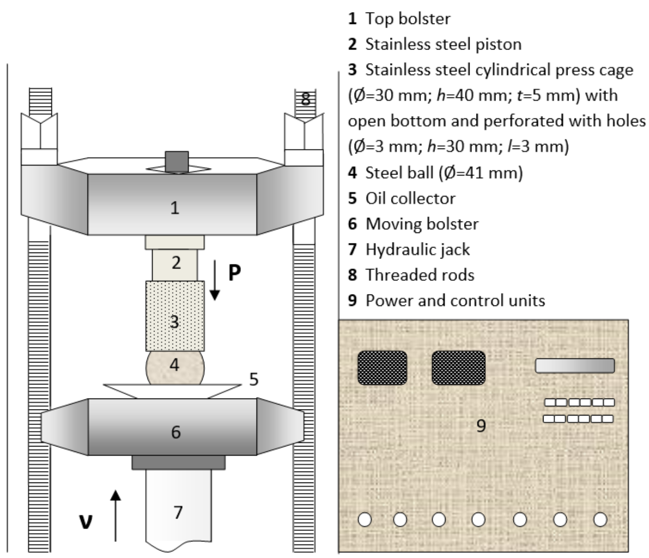

Fig. 2: Schematic representation of the hydraulic pressing setup

The pressing device includes a stainless-steel cylindrical press cage, a stainless-steel piston, a steel ball, an oil collector, and a take-out piece. The press cage had an inside diameter of 30 mm, a height of 40 mm, and a wall thickness of 5 mm. The press cage had an open bottom; their walls were perforated along 30 mm with holes of 3 mm diameter and separated from each other by 3 mm. The diameter of the piston was adjusted to ensure the sample compression by unidirectional pressing. This study used two particle sizes: whole and crushed (≤4 mm) seeds. A seed sample was weighed (±0.1 g) and placed into the press cage covered inside a cotton cloth filter used for direct filtration of the extracted oil and to prevent the dissipation of solid particles through the perforations [7,8]. During pressing, the press cage was positioned on a steel ball of 41 mm diameter to facilitate oil flow. All oil extraction experiments were carried out at an ambient temperature of about 25 °C without any heat pretreatment of oilseeds. Crude oil and press cake were collected and weighed (±0.1 g)after each experiment and stored in light-proof plastic containers at 4 °C until further analysis.

The effect of four processing parameters (dwell time, applied pressure, compression speed, and press cage charge) on the oil recovery was considered in this study. The optimal processing parameters were established based on the best oil recovery obtained for the different variables. The oil recovery was evaluated as follows (**Equation 1**) [9,10]:

$$Oil\ recovery\ (\%) = \frac{Mass\ of\ oil\ extracted\ \ from\ a\ given\ mass\ of\ seeds\ (g)\times 100}{Mass\ of\ oil\ contained\ \ in\ the\ seeds\ initially\ \ taken\ (g)} (1)$$

The mass of oil contained in the seeds initially taken was determined by the Soxhlet method which could be considered the most suitable extraction method since it can extract 95-98% mass fraction of the available oil in the seeds [2].

Table 1 presents the start operational parameters of hydraulic pressing of Jatropha oilseeds used in this study. Firstly, the optimal dwell time was determined based on a reference setpoint pressure of 49.9 MPa obtained from the literature [11] for the dehulled Jatropha seeds or that which has been pre-established in this study for the unhulled seeds. The dwell time is an important parameter that influences the efficiency of the oil extraction process. It was established by observing the oil flow throughout the holes of the press cage under a magnifying glass and measured with a stopwatch from the start of compression until the oil flow stopped. The optimal dwell time was verified by a time step of ±100 s and remained fixed for further extraction tests. The press cage has been fully charged in each case, and the compression speed has been maximal.

Tab. 1. Start the experimental design used in this study

Factors	Jatrophaunhulled seeds		Jatropha dehulled seeds	
	whole	crushed	whole	crushed
Applied pressure (MPa)	80.4*		49.9[11]	
Optimal dwell time* (s)	180	960	300	540
Maximal compression speed* (MPa/s)	5.1	3.1	1.3	1.2
Maximal press cage charge* (g)	7.0		8.0	

* Present study

The optimal applied pressure was determined by adjusting the reference setpoint pressure and keeping constant the optimal dwell time, the maximal compression speed, and the fully loaded press cage. It was defined as an interval of applied pressures with an average pitch of 10 MPa, given the best oil extraction rates. The optimal compression speed was determined by fixing the optimal dwell time, applied pressure, and the maximal press cage charge. Four speeds expressed in MPa/s and corresponding to 100, 75, 50, and 25% maximal compression speed were considered. The optimal press cage charge for different oleaginous materials was established based on the optimal dwell time, applied pressure, and compression speed. The load of 100, 75, 66.7, or 50% of the cage capacity has been tested. A flow diagram for the optimization of cold extraction parameters of Jatropha oil is shown in **Figure 3**.

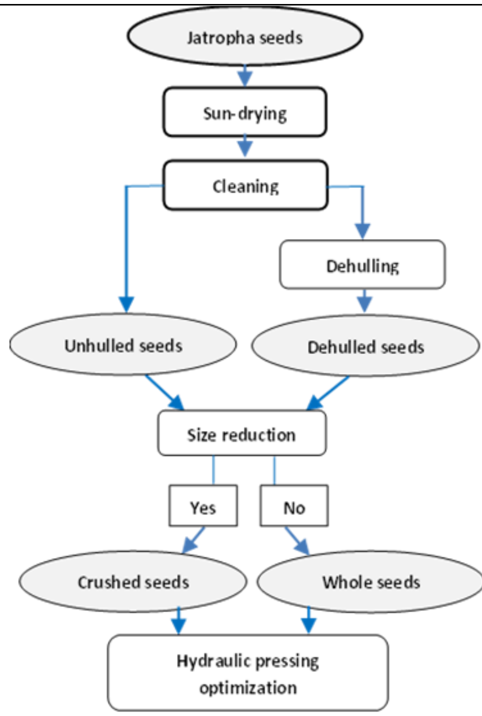

Fig. 3: Flow diagram

2.3. Physicochemical characterization of extracted oil

Physical and chemical parameters of *n*-hexane extracted and cold-pressed oils were determined according to standard methods: acid value (ISO 660:2009(F)), moisture and volatile matter (ISO 662), relative density (ASTM D-4052-96), kinematic viscosity (ASTM D-445), pour (ASTM D97-93) and cloud (ASTM D2500-91) points. Iron and copper contents in plant materials, extracted oils, and press cakes were determined by flame atomic absorption spectrometry (AA-7000 Atomic Absorption Spectrophotometer SHIMADZU, Japon) at 248.3 and 324.8 nm, respectively. Samples were calcined at 550 °C (CARBOLITE, BWF 11/13, England) for 10 h, after which ashes were wholly dissolved in 5% HNO_3. The phosphorus content in extracted oils was determined colorimetrically at 460 nm (T70 UV/VIS Spectrophotometer, PG Instruments Ltd, England) using the mixed solution of ammonium vanadate and ammonium molybdate. The moisture content of oilseeds was measured according to ISO 665 method. All analytical tests were performed in duplicate.

2.4. Statistical analysis

At least three replicate measurements were performed for all samples subjected to cold pressing. The determination of each parameter's mean and standard deviation was carried out using R software for Windows (Version 4.0.0). Data were analyzed using a two-way analysis of variance (ANOVA) with applied pressure, compression speed, and press cage charge as main factors with a confidence level of 95%. The statistical significance of the differences observed among mean values was assessed using Scheffe's test. A probability of $p \leq 0.05$ was considered significant.

3. Results and Discussion

3.1. Oil recovery

In this study, four types of oleaginous material were used: unhulled and dehulled Jatropha seeds with or without size reduction for each of them. The combined effects of hulling and grinding on the oil recovery obtained by cold pressing were evaluated. Furthermore, four pressing parameters such as dwell time, pressure, compression speed and press cage charge have been optimized for each type of Jatropha seeds. Jatropha unhulled and dehulled seeds used in this experiment have 33.6% and 51.7% w/w (d.b.) oil contents, respectively. Depending on the variety, the dehulled Jatropha seeds may contain 40–60% of oil [1]. Willems et *al*. [11] and Subroto et *al*. [12] found an oil content of about 58% w/w (d.b.)for Jatropha kernels, while for the hulls, it was negligible or equal to zero. According to these authors, the mass fraction of Jatropha hulls is around 36% w/w (d.b.). They consist mainly of hydrophilic compounds like lignocellulose, fibers, and mucilage. Otherwise, kernels are composed of more than 50% hydrophobic compounds (lipid fractions). Similar to this affirmation, the moisture contents of Jatropha unhulled et dehulled seeds used in this study were5.1 and 2.9% w/w (w.b.), respectively.

Table 2 gives the optimum oil recovery (mean ± standard deviation) obtained with optimal extraction parameters for Jatropha oilseeds.

Tab. 2. Oil recovery efficiency and optimized cold hydraulic pressing parameters for Jatropha oilseeds

	Parameters				Oil recovery, % w/w(d.b.)
	Dwell time, s	Applied pressure, MPa	Compression speed, MPa/s	Press cage charge, %	
Jatropha unhulled seeds					
whole	180	110.0 ± 10.4	5.1	100	38.2 ± 1.4
crushed	960	117.5 ± 6.6	3.1	100	42.5 ± 0.0
Jatropha dehulled seeds					
whole	300	87.7 ± 1.6	2.1	100	71.3±1.4
crushed	540	90.1 ± 9.7	1.8	100	69.5±1.4

The results presented in Table 2 show that the optimal dwell time is specific to each type of oleaginous material. The crushed seeds require more time to release the oil than the whole ones. That could be due to the significant compaction of the small-size material and, consequently, to the difficulties of draining the oil. The optimal dwell time measured at a reference pressure of 80.4 MPa for unhulled Jatropha oilseeds was 180 s (whole seeds) and 960 s (crushed seeds). For dehulled seeds, it was measured at 49.9 MPa [11] and equal to 300 s (whole seeds) and 540 s (crushed seeds). The rigidity of whole unshelled seeds does not allow a good bursting of the oil cells, which leads to a low rate of oil extraction and a reduced time for its flow. Conversely, hulls in the crushed material allow better oil drainage because the pressing cake obtained is less compact. However, the oil flow time, in this case, is longer.

The applied pressure is the most significant parameter in the hydraulic pressing of oilseeds [13]. This statement agreed with the results ($p < 0.0001$) of variance analysis obtained for applied pressure in this study. **Figure 4** shows the variation of oil recovery with applied pressure for Jatropha oilseeds. The fixed parameters were the previously determined optimum dwell time, the maximum compression speed, and the press cage charge (**Table 1**).

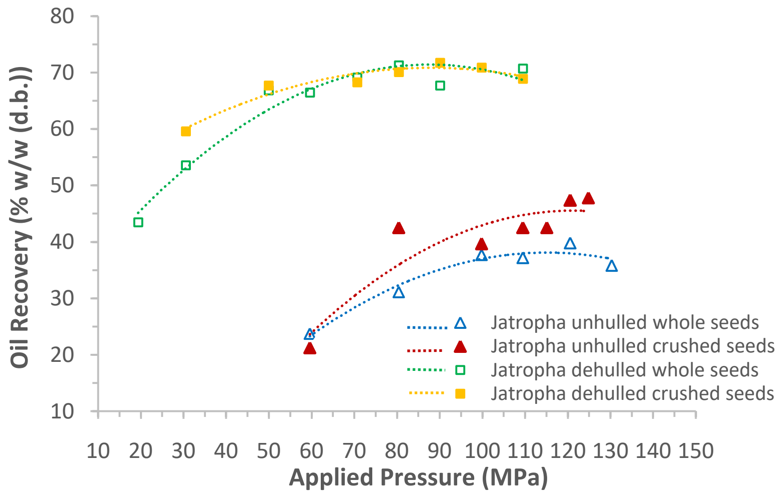

Fig. 4. Influence of applied pressure on the oil recovery for Jatropha oilseeds

As can be seen from the graph, the maximum oil recovery is within an optimum pressure range limited to about ±10 MPa. Increasing pressure beyond this range decreases oil recovery due to pressing cake compaction. Otherwise, the optimal pressures for whole and crushed seeds were quite close. Oil recoveries of 38.2 and 42.5% w/w (d.b.) were found for unhulled whole and crushed Jatropha seeds, respectively, at optimal pressures of 110.0 ± 10.4 MPa (whole seeds) and 117.5 ± 6.6 MPa (crushed seeds). The pressing of dehulled seeds requires a lower optimal pressure of87.7 ± 1.6 MPa (whole seeds) and 90.1 ± 9.7 MPa (crushed seeds) compared to the unhulled seeds with the highest oil recoveries of 71.3 and 69.5% w/w (d.b.), respectively. Similar results were obtained by Willems et *al*. [11], with oil recoveries of 33–52% for Jatropha seeds and 62–75% for Jatropha kernels pressed at20–70 MPa, 40 °Cfor 10 min. According to Subroto et *al*., the hulls consume part of the applied pressure and absorb part of the oil, which leads to a lower oil recovery [12].

The optimal compression speed was determined by fixing the optimal dwell time, optimal applied pressure, and the maximal press cage charge. The compression speed was studied at four levels: maximal speed (100%), minimal speed (25%), and two intermediate speeds (75 and 50%). The speed expressed in MPa/s represents the ratio of optimal applied pressure over time that elapses between the start of compression and the start of dwell time. The best oil recoveries from unhulled whole and crushed Jatropha oilseeds were recorded at a maximum compression speed corresponding to 5.1 and 3.1 MPa/s, respectively. In the case of dehulled Jatropha oilseeds, the same tendency was observed for the whole seeds with the maximal compression speed of 2.1 MPa/s. Otherwise, for the dehulled crushed seeds the oil recovery was practically invariable with the compression speed. The maximum speed of 1.8 MPa/s has been considered optimal.

In this study, four filling levels (100, 75, 66.7, and 50% cage capacity) of the pressing cage were tested. The fixed parameters were the optimal dwell time, applied pressure, and compression speed determined previously. Under the experiment conditions, a significant improvement in oil recovery ($p < 0.05$) with increasing press cage charge was observed for all oleaginous materials. The reduction in oil recovery with the low press cage charge could be explained by the more significant compaction of the pressing cake due to the reduced specific strength of the oleaginous material when its amount decreases. Furthermore, the results presented here were obtained for small quantities of oilseeds using small size pressing cage positioned on a steel ball and need to be confirmed with more replications and a larger press cage. Scaling up using more significant amounts of oleaginous material would require special consideration concerning the configuration of the pressing device (i.e., with or without a steel ball at the bottom of the press cage).

3.2. Physicochemical properties of extracted Jatropha oil

The values of some physicochemical properties of the cold-pressed Jatropha oil obtained from the dehulled seeds are shown in **Table 3**. They were compared with the properties of the *n*-hexane-expressed Jatropha oil and rapeseed oil according to the quality-related parameters of DIN 51605: 2010-10.

Tab. 3. Physiochemical properties of *n*-hexane extracted and cold-pressed Jatropha oils

Parameters	*n*-Hexane extracted Jatropha oil	Cold-pressed Jatropha oil	DIN 51605:2010-10 Rapeseed oil
Acid value, mgKOH/g	7.16	ND	2.0
Moisture and volatile matter, % w/w (d.b.)	5.88	0.15	0.075
Relative density (15 °C)	0.916	0.918	0.910-0.925
(25 °C)	0.909	0.911	
Kinematic viscosity, cSt (37.8 °C)	37.03	37.72	36 (40 °C)
(50.0 °C)	24.69	24.96	
Cloud point, °C	-1	+1	
Pour point, °C	-3	0	
Phosphorus, ppm	30	10	3.0
Iron, ppm	4.6	1.9	
Copper, ppm	0.0	0.0	

Moisture and volatile matter content of cold-pressed Jatropha oil was about 0.15% w/w (d.b.), indicating that it is of higher quality than the oil extracted with *n*-hexane containing 5.88% w/w (d.b.). However, the water content of cold-pressed Jatropha oil remains above the upper limit of 0.075% set by DIN 51605:2010-10 for rapeseed fuel. Therefore, a direct application as a fuel of Jatropha oil obtained by cold pressing is not recommended. Its transformation into biodiesel will require the use of appropriate methods.

The observed density and viscosity of cold-pressed Jatropha oil were 0.918 (15 °C) and 37.72cSt (37.8 °C), respectively, which is more or less similar to those of *n*-hexane extracted Jatropha oil and rapeseed oil according to the DIN 51605:2010-10 standard. Furthermore, the cold flow characteristics have been slightly different for the two types of Jatropha oil. Thus, the cloud and pour points of the cold-pressed Jatropha oil were +1 and 0°C, respectively, while the *n*-hexane extracted oil had-1 °C (cloud point) and -3 °C (pour point).

The phosphorus content was higher for *n*-hexane-extracted Jatropha oil (30ppm) compared to the cold-pressed oil (10ppm). This result agrees with the data reported by Brevedan et *al*., showing that the extraction using solvent produces high phospholipid (gums) content in the oil. In contrast, oils obtained by cold pressing contain small amounts of phosphatides [14]. Moreover, the phosphorus content in both types of extracted Jatropha oil was significantly higher than that indicated in the DIN 51605:2010-10 standard (3ppm). The phosphorus content could be corrected by degumming or refining the oil if necessary.

The cold-pressed Jatropha oil had a low level of iron (1.9 ppm) and no copper. The metals in vegetable oil can come from seeds or manufacturing or storage equipment. Their presence reduces the oxidative stability of the oil involved in rancidity processes[15]. The results show that Jatropha oil extracted with *n*-hexane contains 4.6 ppm of iron, indicating its natural origin. Therefore, the metal contents of the raw material (unshelled and shelled seeds), as well as cakes, were evaluated. As can be seen from **Table 4**, the iron and copper concentrations in the press cakes are higher than those in the seeds, suggesting that the oil drives only a negligible amount of these metals.

Tab. 4. Iron and copper concentrations in Jatropha oilseeds and their press cakes

	Iron, ppm	Copper, ppm
Jatropha		
unhulled seeds	45.3	13.0
press cake	56.3	15.0
dehulled seeds	55.3	11.1
press cake	76.1	15.3

4. Conclusion

Optimizing hydraulic pressing parameters was performed at 25 °C for the unhulled and dehulled Jatropha oilseeds using whole and crushed oleaginous material. The best oil recovery from cold-pressed Jatropha oilseeds was 71.3 ± 1.4% w/w (d.b.) obtained with dehulled and crushed seeds, a dwell time of 300 s, an applied pressure of 87.7 ± 1.6 MPa, a compression speed of 2.1 MPa/s, and 100% charged press cage. The quality of the cold-pressed Jatropha oil was better compared to *n*-hexane extracted oil, with lower contents of moisture, phosphorus, and iron. The density and viscosity of this oil met the requirements set by the DIN 51605:2010-10 standard for plant oil-based fuels. Future studies should focus on scaling up Jatropha oil hydraulic expression, operational parameters adjustment, and oil quality evaluation depending on the pressing temperature.

Conflict of interest

No conflict of interest to disclose.

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