

## Analysis of physical, mechanical and chemical properties of seeds and kernels of *Jatropha curcas*

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### Abstract

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The research was performed to examine the physical, mechanical and chemical properties of seeds and kernels of *Jatropha curcas*. The test parameters were the dimensions of the seeds and kernels, required energy for oil extraction, determination of fatty acids in the oil by gas chromatography method, determination of the iodine value, determination of the acid value, determination of total polyphenols by the Folin & Ciocault reagent and determination of tocopherols and tocotrienols (vitamin E) by High-performance liquid chromatography. It was ascertained that the size of the seed and kernel varies considerably. Pressing of whole seeds needs more energy (50%) than pressing of kernels. From a chemical point of view it seems to be very appropriate for a production of biofuels. *Jatropha curcas* contains more polyphenols and vitamin E, which act as antioxidants, than the rape. Due to the low content of unsaturated fatty acids it is chemically suitable to replace the rape-seed oil with *Jatropha curcas* oil.

**Keywords:** cake; chemical analysis; pressing

In recent years biofuels have obtained considerable interest, due to the implementation of ruling and a gradual replacement of fossil fuels (RUŽBARSKÝ et al. 2013). From the ecological point of view vegetable oil-based biofuels are in many aspects better than fossil fuels, such as in the agricultural machinery area (PEXA et al. 2013). A potential place to obtain these biofuels is primarily in tropical and subtropical areas where are facilities for the harvest a few times per year and yield maximization. The seeds of *Jatropha curcas* contain a large percentage of

oil, which has a wide use, for example in the production of biodiesel (SAMSURI et al. 2014).

Determining of the physical and mechanical properties of agricultural products is essential for producing suitable designs for the relevant processing technology (HERÁK et al. 2013). However, detailed understanding of the agricultural product properties before they are determined through testing is vital (DOBZANSKI, SZOT 1997; SIRISOMBOON et al. 2007).

One of production steps at gaining the oil is a pressing process (KABUTEY et al. 2012). The press-

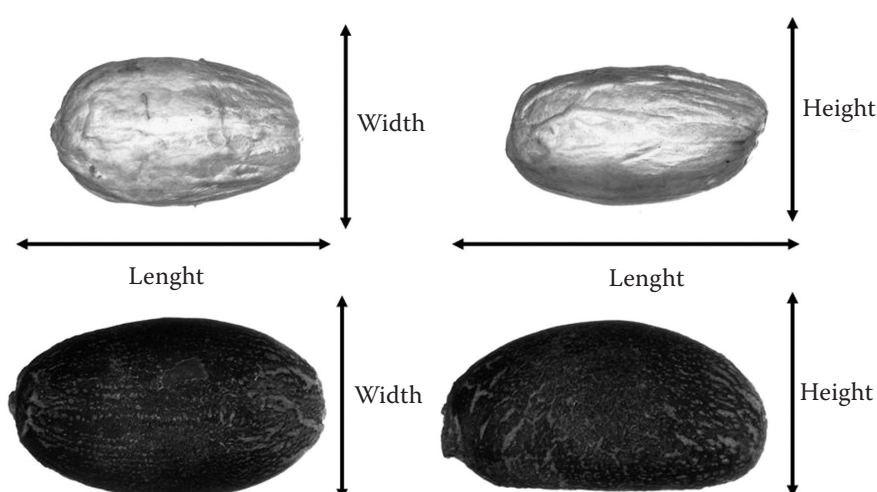


Fig. 1. Methods of measurement of dimensions of seeds and kernels of *Jatropha curcas*

ing is a complex process, to which a considerable amount of work currently devotes. Detailed understanding of the process of the oil extraction is one of the important factors that can help to find the system with the max. ratio of the energy output versus input (KABUTEY et al. 2012; PETRŮ et al. 2012; HERÁK et al. 2013). In manufacturing, there are two basic directions of the pressing; those are whole seeds and kernels.

The research was performed to examine the physical, mechanical and chemical properties of seeds and kernels of *Jatropha curcas*. The aim of this study was to determine the energy needed for the production of oil from the kernels and seeds of *Jatropha curcas*. Tested seeds were of a brown colour. The pressing went as cold-pressing. The chemical analysis of the pressed oil was performed. The aim of the chemical analysis was to determine the ratio of saturated and unsaturated fatty acids essential for possible application in the field of biofuels.

## MATERIAL AND METHODS

**Tested seeds.** Tests were performed at seeds of brown colour produced in Indonesia. At first the tested seeds and kernels (50 pieces) were analysed. Following dimensions of seeds were set: height, width and length (Fig. 1). Dimensions of seeds were determined by measurement using a Mitutoyo digital caliper (Mitutoyo Japan, Kawasaki, Japan). Due to the irregular geometric shape of seeds and kernels the measurement was repeated three times.

**Pressing.** The pressing was conducted at whole seeds and separate kernels (Fig. 2). Pressing was

performed on the LaborTest 5.50ST (50 kN) (Labortech s.r.o, Opava, Czech Republic). The deformation speed was 10 mm/min. The pressing device consists of a pressing piston and a pressing container. The pressing device has a cylindrical shape with a diameter of 60 mm. An infill portion was up to 80 mm. A deformation of the infill was constantly set at 50 mm.

**Chemical analysis.** A chemical analysis was conducted on compacted oil from seeds and kernels. The tested oil was not subjected to any modification. The resulting oil was subjected to the chemical analysis:

**Determination of fatty acids by GC.** Fatty acids were analysed as the methyl esters after trans-esterification by a gas chromatography. The method according to the Standard ISO 5509:1994 was used for the preparation of methyl esters. This method is



Fig. 2. Pressing of seeds and kernels of *Jatropha curcas*

based on the saponification of fats and esterification of fatty acids liberated from methanol in the presence of boron trifluoride. Varian 3300 gas chromatograph (Varian Instrument Group, Victoria, Australia) was used for the own analysis equipped with a quartz glass tube and the open DB 23 column (30 mm  $\times$  0.25 mm, film thickness of 0.25 nm) with a flame ionization detector 5  $\mu$  RP Aqueous (250  $\times$  4.6 mm) (Develosil, Phenomenex, USA).

**Determination of the iodine value by Hanus solution method.** The oil was dissolved in 10 ml of chloroform. Then exactly 25.0 ml of Hanus solution (in a fume hood burette) was added. Everything in the flask was mixed and let stand in the dark for 60 min at room temperature. During this time it was necessary to stir it several times by swirling. Then 20 ml of KI solution, 100 ml of distilled water and titrate with standard sodium thiosulfate were added to gain a pale yellow colour. Then 1 ml of starch paste was added and the titration was continuing until the complete discoloration of the upper phase solution.

**Determination of acid value.** 3–4 g of oil with an accuracy of 0.1 g was weighed into the titration flask, 50 ml of ethanol-benzene (1:2) was added and shaken. Then the content was carefully heated in a water bath until the oil dissolved. 5–8 drops of 1% phenolphthalein solution was added in ethanol and titrated with volumetric hot alcoholic solution of KOH 0.1 mol/l (6 g of solid KOH are dissolved in a small amount of water and diluted to 96% ethanol to 1 l) to gain faint red colour, which lasts about 30 seconds.

**Determination of total phenols (TP) by Folin-Ciocalteu reagent.** Finely powdered *Jatropha curcas* seeds were extracted in a HR 2185 Philips electric mill (Phillips, Amsterdam, Netherlands) (ca. 0.5 g) with 10 ml of 80% methanol and stirred for 10 min in an ultrasonic bath. The extract was centrifuged and transferred into 25 ml volumetric flask and the extraction was repeated. The extract was adjusted to 25 ml with 80% methanol and carefully stirred. For the TP determination 1 ml aliquots of sample solutions were pipetted. The sample extract (1 ml) was transferred into a 50 ml volumetric flask and diluted with approximately 5 ml of distilled water. Then, 2.5 ml of the Folin-Ciocalteu reagent (Penta, Chrudim, Czech Republic) and 7.5 ml of 20% (w/w) Na<sub>2</sub>CO<sub>3</sub> (Lach-Ner s.r.o., Neratovice, Czech Republic) were added, adjusted with distilled water to 50 ml, agitated and let stand for

2 hours. Absorbance of the sample was measured on the UV-visible spectrophotometer Spectronic Helios  $\gamma$  (Thermo Spectronic, Cambridge, Great Britain) at wavelength ( $\lambda$  = 765 nm) against a blank prepared with distilled water. Gallic acid (G.R. purity; Merck KGaA, Darmstadt, Germany) was used for calibration. The results were expressed as gallic acid equivalents (GAE) in mg/kg of the dry matter (d.m.) from three replicates (LACHMAN et al. 2013).

**Determination of tocols by HPLC-FLD.** Approximately 0.5 g of homogenised *Jatropha curcas* seed sample was placed into a 10 ml test tube, 10 ml methanol was added, everything sonicated for 10 min with occasional stirring, and consequently centrifuged at 5,000 RCF-g for 5 minutes. The supernatant was transferred into a 100 ml evaporating flask. The remaining pellet was reextracted with 10 ml of methanol and centrifuged. The supernatants were combined and evaporated until dry using Büchiroto vapor R-215 (Büchi Labortechnik GmbH, Essen, Germany) at 65°C. For HPLC analysis the samples were redissolved in 10 ml methanol and transferred through nylon microfilter into a dark vial. Analysis was performed using a High Performance Liquid Chromatograph Ultimate 3000 (Thermo Fisher Scientific, Sunnyvale, USA) with a quaternary pump, refrigerated autosampler, column heater and FLD Ultimate 3000 RS detector (Dionex Softron GmbH, Germering, Germany). All results were expressed as mean values of three replicates (LACHMAN et al. 2013).

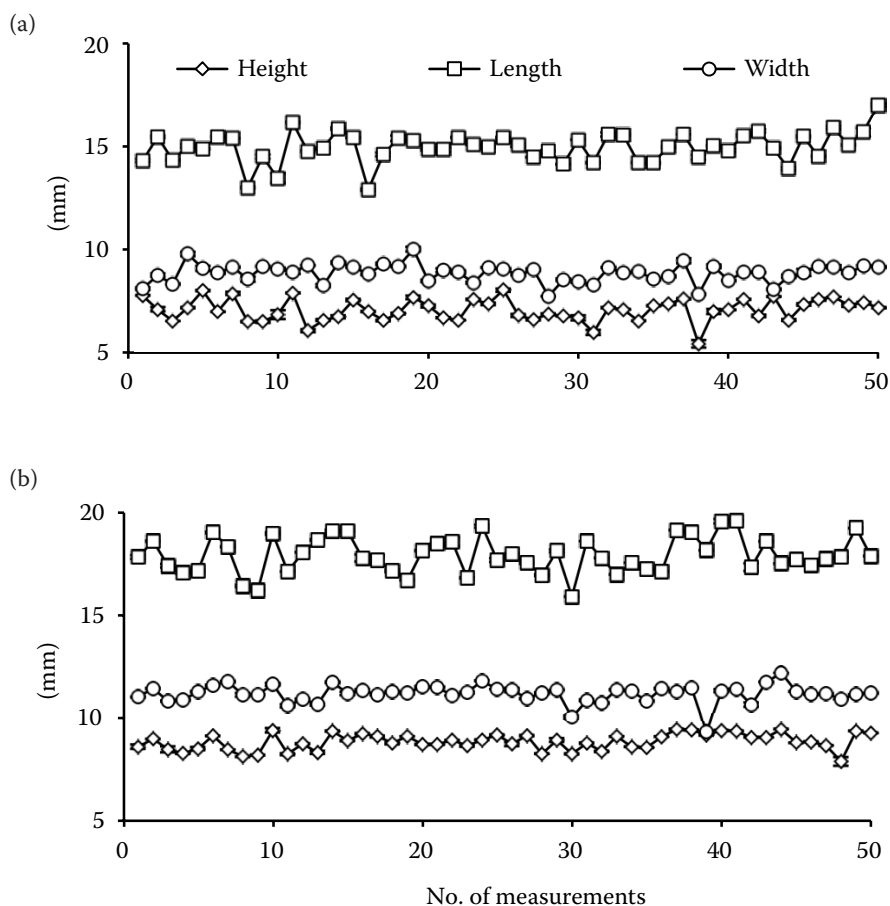
## RESULTS AND DISCUSSION

When testing the size of *Jatropha curcas* seeds and seed kernels, the following values were determined:

- seed: width  $11.17 \pm 0.45$  mm, length  $17.91 \pm 0.86$  mm and height  $8.82 \pm 0.40$  mm,
- seed kernel: width  $8.85 \pm 0.44$  mm, length  $14.97 \pm 0.75$  mm and height  $7.06 \pm 0.54$  mm.

A graphic presentation of the results of size of seeds and kernels of *Jatropha curcas* is evident from Fig. 3. When comparing the mean value of data a difference between the tested series is clear. From the results it is clear that research results cannot be generalized to one average seed. Differences are among the seeds and kernels.

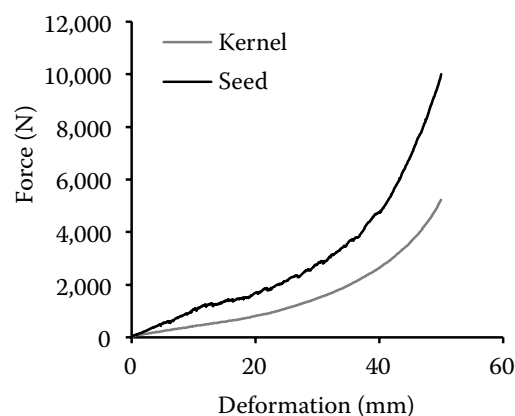
For obtaining the oil from the kernels and seeds of *Jatropha curcas* the cold-pressing was performed. An evaluation of data from the pressing

Fig. 3. Dimensions of *Jatropha curcas* (a) kernel and (b) seeds

process was performed in accordance with the methodology established by HERÁK et al. (2013). The pressing course is shown in Fig. 4. An energy for pressing of the seeds was 147,052 J. The energy for pressing of the kernels was 76,849 J. The difference of the energy required for pressing to obtain the oil is almost 50%. Cakes were formed from the pressing process (Fig. 5). This type of the waste can be effectively used by energy ways. The gross calorific value of cakes from *Jatropha curcas*, according to the research of RUŽBARSKÝ et al. (2013), ranges from 20 to 30 MJ/kg.

Results of the chemical analysis of fatty acids in the oil determined by GC are shown in Table 1. Selected saturated and unsaturated fatty acids were determined by the gas chromatography in the two samples. A ratio of saturated and unsaturated fatty acids is important for fuel utilization. A representation of unsaturated fatty acids which are prone to oxidation processes and thus degradation of the oil or double bonds, thus causing a rancidity of fats, is especially important. The rancidity of fats shows itself also after esterification in the form of sediment. The measured results show that the total contents

of unsaturated fatty acids is 70.25%. A rape contains 81.88% according to LI et al. (2013). The content of unsaturated fatty acids in *Jatropha curcas* is about 10% lower than at rape-seed oil. It can be said that this oil will be less susceptible to the degradation of double bonds than rape-seed oil. Methyl esters of fatty acids, especially rape-seed methyl ester (RME) under Czech conditions (PEXA et al. 2013),

Fig. 4. Pressing course of seeds and kernels of *Jatropha curcas*



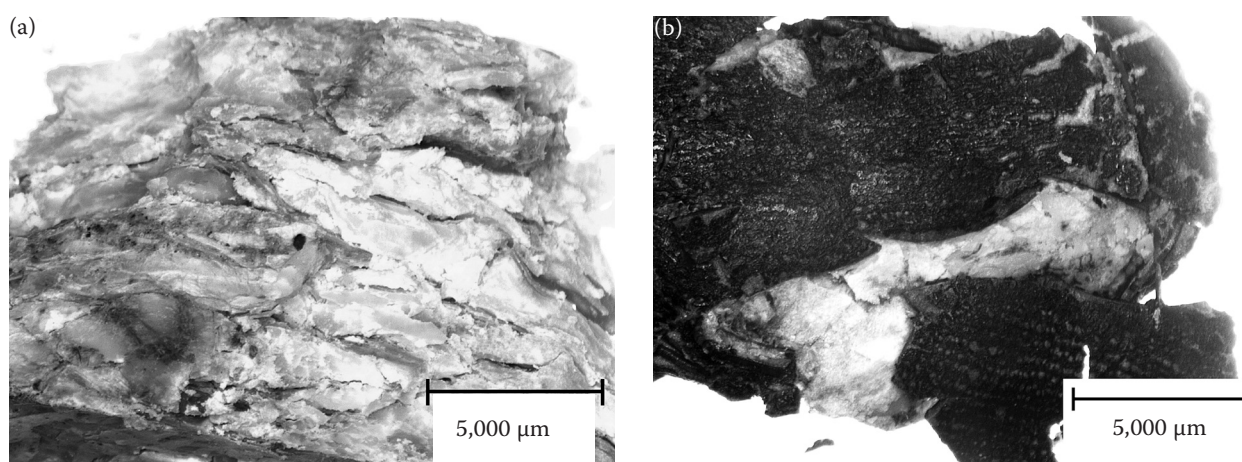


Fig. 5. Cake from pressing process of *Jatropha curcas* (a) kernel and (b) seed

push as the most appropriate substitute of the diesel fuel. The oil of *Jatropha curcas* is therefore more suitable for fuel using. Table 1 shows the contents of selected fatty acids in the oil of *Jatropha curcas* and for a comparison the contents of selected fatty acids of rape-seed oil by Li et al. (2013). *Jatropha curcas* seeds showed a much higher contents of oleic acid 37.21% than 1.48% of rapeseed oil. The content of linoleic acid in the *Jatropha curcas* was also increased by 32.10% compared with the rape. In contrast, linolenic acid (0.15%) was significantly lower than at rape-seed oil (60.56%).

The iodine value is the percentage of halogen which can, under certain conditions, add the double bonds of unsaturated fatty acids. The iodine value is thus a

measure of the contents of unsaturated fatty acids in the oil. The determination of the iodine value by the Hanus solution method showed minimal differences among the tested oils from *Jatropha curcas*. The seed oil has the iodine value of  $99.3 \pm 0.80\%$ . The oil from kernels reached values  $98.9 \pm 0.80\%$ . For example the rape reaches values 94 up to 100% (BATISTA et al. 2011).

The acid number indicates the representation of free fatty acids in the oil. Free fatty acids are formed as products of the lipid rancidity, sequential, either enzymatic or purely chemical (hydrolysis) decomposition or they may occur during bad treatment of fats by the hydrolysis. Their increased amount gives evidence of degradation in the quality of the

Table 1. Determination of fatty acids by gas chromatography

Fatty acid (FA)	<i>Jatropha curcas</i> (%)		Rape seed (Li et al. 2013)
	seed	kernel	
Arachidic acid	0.18	0.18	0.49
Behenic acid	less than 0.05	less than 0.05	0.18
Heptadecanoic acid	0.09	0.08	0.2
Capric acid	0.43	0.22	0.04
Lauric acid	less than 0.05	less than 0.05	0.3
Myristic acid	0.06	0.06	0.68
Palmitic acid	14.1	13.5	10.87
Stearic acid	5.9	5.85	2.17
Linolenic acid	0.15	0.14	60.56
Linolic acid	32.1	31.65	19.6
Oleic acid	37.21	37.15	1.48
Palmitoleic acid	0.79	0.75	0.24
Ratio of unsaturated and saturated FA	70.25:20.86	69.69:19.99	

Table 2. Determination of tocopherols and tocotrienols (vitamin E) by HPLC method

Sample	<i>Jatropha curcas</i> seed (mg/kg)	Rape seed (mg/kg) (Li et al. 2013)
$\alpha$ -tocopherol	not detected	124.3
$\gamma$ -tocopherol	$70.76 \pm 4.12$	9.2
$\gamma$ -tocotrienol	$337.726 \pm 36.39$	–
$\delta$ -tocotrienol	$45.906 \pm 0.53$	–

oil or old age after the long-term storage. From the results it is clear that higher values were achieved in the oil from whole seeds, where the acid number was  $4.79 \pm 0.06$ . The value for oil pressed from the kernel was  $4.56 \pm 0.04$ .

Polyphenolic compounds are natural substances which are present in each part of higher plant and in each of its body as secondary metabolites. The structure and the type of these substances are for individual plant species characteristic. They also have a significant correlation with antioxidant activity. In addition to positive effects on human health (and the health of all mammals) they have high antioxidant activity due to a protective effect on the quality of the oil. It protects the oil from oxidation and degradation of multiple bonds of unsaturated fatty acids. Polyphenolic substances have been set in the whole seed of *Jatropha curcas*. The value was measured as  $741 \pm 0.035$  mg/kg. Comparing with the rape-seed oil *Jatropha curcas* achieves more than 95% lower value (BATISTA et al. 2011).

Vitamin E, tocopherols and tocotrienols, is fat-soluble (lipophilic). It acts here as an antioxidant, also protects the fat before oxidation (degradation of multiple bonds of unsaturated fatty acids). The determination of tocopherols and tocotrienols (vitamin E) by HPLC method can be seen from Table 2. *Jatropha curcas* seeds showed overall higher contents of tocopherols and tocotrienols than rape seeds. The total content of vitamin E of *Jatropha curcas* seeds was 454.39 mg/kg. On the contrary, according to Li et al. (2013) a total content of vitamin E was 133.5 mg/kg at the rape. It can therefore be concluded that *Jatropha curcas* has a significant protective effect in the protection of oils than the rape.

## CONCLUSION

In recent years biofuels have obtained considerable interest, due to the implementation of ruling

and gradual replacement of fossil fuels. Potential places for acquiring new biofuels are areas providing crop several times a year. These areas include mainly crops from tropical and subtropical belt.

From the research results following conclusions can be stated:

- The pressing of whole seeds requires more energy than the pressing of kernels. The difference in the energy required for pressing to obtain oil is almost 50%.
- The dimensional analysis showed that there were significant differences in the length, width and height of seeds and kernels. The variability is following: the seed – width 4.95%, length 4.98% and height 7.6%, the seed kernel – width 4.06%, length 4.78% and height 4.52%.
- From a chemical point of view significant differences were not found between oils (the whole seed and the kernel alone). Due to the lower contents of unsaturated fatty acids, the oil is very suitable for use as fuel. The suitability of this oil is demonstrated by the higher contents of antioxidants, i.e. tocopherols, tocotrienols and polyphenols, compared to rape-seed oil.

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