

Physicochemical Characterization of *Jatropha podagrica* Seed Oil for Potential Biodiesel Production and other Industrial Applications in Thailand

(Pencirian Fisikokimia Minyak Biji *Jatropha podagrica* bagi Pengeluaran Biodisel Berpotensi dan Aplikasi Industri lain di Thailand)

DUANGPORN PREMJET, ABRAHAM KUSI OBENG, HAH YOUNG YOO, SEUNG WOOK KIM & SIRIPONG PREMJET*

ABSTRACT

Jatropha is considered as one of the most promising potential oil sources for biodiesel production and other industrial applications. However, research on the potential of *Jatropha* seed oil is mainly focused on *Jatropha curcas*, with other species receiving little attention. The physicochemical properties of *J. podagrica* seed oil was studied to determine its potential as feedstock for biodiesel production and other industrial applications in Thailand. The seed oil was extracted with *n*-hexane from milled kernels using the soxhlet extractor and subsequently characterised for free fatty acids, iodine value, viscosity, saponification value, density, and acid value. The fatty acid profile of the seed oil was also analysed using gas chromatography (GC). Analysis of the physical properties of the *J. podagrica* seed kernel showed lower average physical characteristics when compared to those of *J. curcas* seed kernel. *J. podagrica* seeds had high oil content comparable to *J. curcas* oil content. The main fatty acid components of the seed oil were oleic acid (15%) and linoleic acid (70%). Generally, the results of the physicochemical analysis indicated that *J. podagrica* seed oil would be very useful for the production of soap and shampoo in Thailand. To produce biodiesel from the seed oil, a two-step acid-catalysed transesterification process would be appropriate.

Keywords: Biodiesel; *Jatropha curcas*; *Jatropha podagrica*; methyl esters; non-edible oil

ABSTRAK

Jatropha dianggap sebagai salah satu sumber minyak yang sangat berpotensi untuk pengeluaran biodiesel dan aplikasi industri lain. Walau bagaimanapun, penyelidikan mengenai potensi minyak biji *Jatropha* hanya difokuskan ke atas *Jatropha curcas* manakala spesies lain kurang mendapat perhatian. Sifat fizikokimia minyak biji *J. podagrica* telah dikaji untuk menentukan potensinya sebagai bahan untuk pengeluaran biodiesel dan aplikasi industri lain di Thailand. Minyak biji telah diekstrak dengan *n*-heksana daripada isirong yang telah digiling menggunakan pengekstrak soxhlet dan kemudiannya dicirikan untuk asid lemak bebas, nilai iodin, kelikatan, nilai saponifikasi, ketumpatan dan nilai asid. Profil asid lemak minyak biji juga dianalisis menggunakan kromatografi gas (GC). Analisis sifat fizikal isirong biji *J. podagrica* menunjukkan purata ciri fizikal yang lebih rendah jika dibandingkan dengan isirong biji *J. curcas*. Biji *J. podagrica* mempunyai kandungan minyak yang tinggi standing dengan kandungan minyak *J. curcas*. Komponen utama asid lemak bagi minyak biji adalah asid oleik (15%) dan asid linoleik (70%). Secara umumnya, hasil analisis fizikokimia menunjukkan bahawa minyak biji *J. podagrica* akan sangat berguna untuk pengeluaran sabun dan syampu di Thailand. Untuk menghasilkan biodiesel daripada minyak biji, proses transesterifikasi pemangkin asid dua langkah adalah bersesuaian.

Kata kunci: Biodiesel; *Jatropha curcas*; *Jatropha podagrica*; metil ester; minyak yang tidak boleh dimakan

INTRODUCTION

The genus *Jatropha*, belonging to the *Euphorbiaceae* family, consists of over 70 species of shrubs including *J. curcas*, *J. pohliana*, *J. podagrica*, and *J. gossypifolia* (de Oliveira et al. 2009). These species are native to Central America, but are now growing in different tropical and

subtropical areas of the world (Adebayo & Ameen 2017). The plant grows rapidly and can withstand drought, pest, and diseases (Wu et al. 2012). It can also survive on marginal lands under a wide range of climatic conditions (Atabani et al. 2013). Cultivation of the plant requires simple technology and is not capital intensive (Pandey et al. 2012).

The *Jatropha* plant has many attributes, uses, and significant potentials. The seeds and other parts of the plant have traditionally been used for the production of oil, soap, and medicinal compounds. The stem has been used for the treatment of gum inflammation, toothache, gum bleeding, and sexually transmitted diseases. The leaves and latex have also been reported as very effective in the treatment of cuts, bruises, mouth, and throat sores (Pandey et al. 2012). *Jatropha* seed oil is a very important raw material for the production of broad industrial products. It can be used to produce cosmetics, soap, paraffin, and candles. Currently, *Jatropha* is considered as one of the most promising potential oil sources for the production of biodiesel worldwide (Borugadda & Goud 2012).

Non-edible oil from *J. curcas* seed has attracted great attention globally as a promising alternative oil source for biodiesel production and other industrial applications (Khan et al. 2014). Several researches have reported that biodiesel from *J. curcas* oil shares similar properties with petroleum-based diesel (Chouhan & Sarma 2013; Jaliliannosrati et al. 2013; Lim & Lee 2013; Raia et al. 2017). *J. curcas* biodiesel has distinct fuel properties that make it an alternative to petrol diesel. Quality fuel indicators such as the cetane index, viscosity, flash point, calorific value, sulfur content, and pour point of *J. curcas* biodiesel meet the ASTM standards (Thapa et al. 2018). However, research on the potential of seed oil

from other *Jatropha* species is very limited. Another common species of great importance in the genus *Jatropha* in Thailand is *J. podagrica* (Rumzhum et al. 2012). It can adapt to different environmental and soil conditions without the need for extra economic inputs. *J. podagrica* seed oil could be used as raw material for biodiesel production and other industrial applications in Thailand (Tsegay et al. 2017). However, the potential of seed oil from *J. podagrica* has not been exploited. *J. podagrica* seed oil was therefore assessed as potential feedstock for biodiesel production and other industrial applications in this study.

MATERIALS AND METHODS

PREPARATION OF *J. PODAGRICA* SEEDS

Fresh, healthy, and whole seeds of *J. podagrica* (Figure 1) were collected from Lablae, Uttaradit Province, Thailand (17° 39'44" N, 100° 1'57" E, 91 m altitude). Seed collection was carried out from July to September 2014. The coats of the seeds were removed and the physical characteristics of the kernel including length, width and weight were then recorded. The seed kernels were then dried in a hot air oven at 60 °C for 12 h. The dried seeds were then milled using the SM 100, Rtsch, Rheinisch-StraBe 36-D-42781 (Haan, North Rhine-Westphalia, Germany) miller and used for oil extraction.

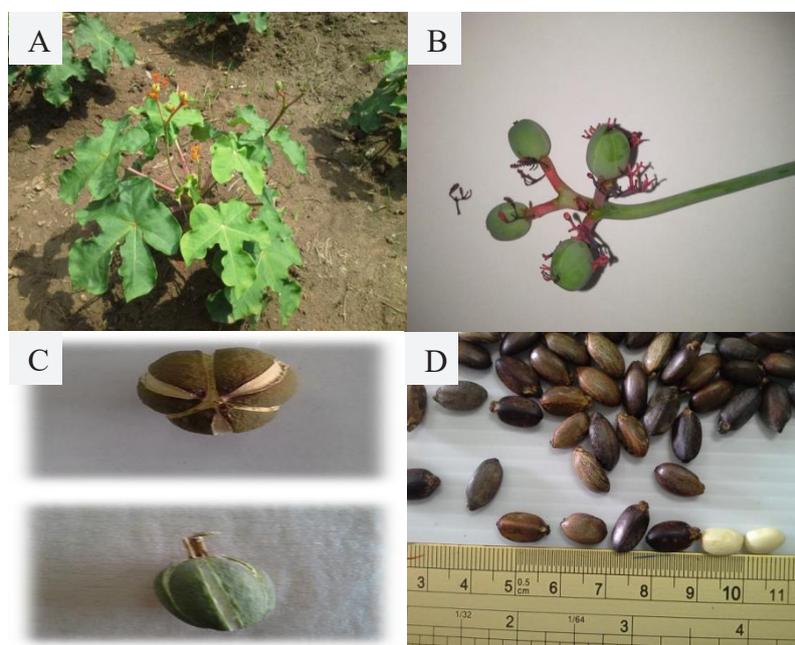


FIGURE 1. Photograph of (A) young *J. podagrica* plant on the field, (B) fresh fruits, (C) dry fruits and (D) seeds

OIL EXTRACTION

J. podagrica seed oil was extracted from the milled kernels (15 g dry weight) with n-hexane (200 mL) using the soxhlet extractor at 60 °C for 24 h by following the in-house method TE-CH-208 from the Association of Official Analytical Chemists (AOAC 2012). To remove the moisture, the micelle produced was dried over anhydrous MgSO₄ and filtered. The solvent was then removed using rotary evaporator at 45 °C. The oil was collected in a vial and kept in a desiccator till a constant weight was attained. The volume of oil obtained was then measured. The oil content was expressed as the percentage of oil in the dry matter of milled kernel.

FATTY ACID METHYL ESTERS ANALYSIS

The fatty acid composition of the seed oil was determined using the in-house method TE-CH-208 based on AOAC (2012). Pyrogallol acid was added to *J. podagrica* oil (0.2 g) to prevent oxidation of the oil. To analyse fatty acid composition, 0.1 mL oil was derivatized to fatty acid methyl esters (FAMES) using 1 mL of 10% (w/w) boron trifluoride-methanol (BF₃-methanol) before being injected into the gas chromatography (GC, Agilent 6890N, Agilent Technologies Inc., CA, USA). The GC was equipped with Agilent HP-FFAP capillary column, 25 m - 0.25 mm ID, 0.22 µm film thickness. Triundecanoin (C₁₁:O) was used as internal standard with helium as carrier gas at an inlet pressure of 120 psi. The temperature of the injection port and detector (FID) was programmed at 200 to 240 °C. The column was operated at the flow rate of 0.8 mL min⁻¹ and the temperature was initially set at 170 °C for 4 min, then adjusted to 180 °C and finally to 190 °C. The injection volume was 1 µL. Identification of FAME peak was achieved by comparing the retention times with those of authentic standards analysed under the same conditions.

CHARACTERISATION OF *J. podagrica* SEED OIL

The physicochemical properties of the extracted seed oil were analysed using standard methods from the AOAC (2012). The properties analysed included free fatty acid (FFA), density, viscosity, acid value, iodine, and saponification values.

PROXIMATE ANALYSIS

Proximate analyses of seed kernels, de-oiled seed cake and seed capsules including moisture content, ash, crude fibre, crude protein, oil content, and energy were carried out as described by AOAC (2006). The carbohydrate content was calculated from the other components.

STATISTICAL ANALYSIS

The data collected on proximate analysis were subjected to one-way analysis of variance (ANOVA) using SPSS version 16.0 (SPSS Inc., Chicago, IL, USA). Treatment means were compared by Tukey's test at 5% significance level. The analyses were performed in triplicate and the average values with their standard deviations were presented.

RESULTS AND DISCUSSION

PHYSICAL CHARACTERISTICS OF *J. podagrica* SEEDS

J. podagrica seed is the main organ for storing oil in the plant. Similar to *J. curcus*, the physical characteristics of the seed, among other factors, have significant influence on the oil yield (Wu et al. 2012). The average weight of 100 *J. podagrica* seed kernels were found to be 15.52±0.19 g, corresponding to an average seed kernel weight of 0.16 g. On the other hand, seed kernel in the current study had an average length and width of 12.11±0.71 and 6.03±0.41 mm, respectively. The average physical characteristics of *J. podagrica* seed kernel in this study were lower than those reported for *J. curcus* seed kernel (Abou-Arab & Abu-Salem 2010; Wu et al. 2012).

J. podagrica SEED OIL

The oil yield of *J. podagrica* seed kernel was affected by the season in which the seeds were harvested. Seeds harvested during the dry season contained higher (p<0.05) oil yield (31%) when compared those harvested in the rainy season (24%) (Table 1). The high oil yield makes seeds harvested in the dry season very ideal for biodiesel production and other industrial applications. The yield of oil extracted from *J. podagrica* seed kernel in the current study was comparable to those reported for *J. curcas* seeds by several researchers which were 27% (Abou-Arab & Abu-Salem 2010), 32% (de Oliveira et al. 2009), 32% (Kibazohi & Sangwan 2011), 34% (Evon et al. 2013), 43% (seed biomass size ≤ 0.5 mm), 32% (seed biomass size ≥ 0.5 but ≤ 1.1 mm) (Jaliliannosrati et al. 2013) and 47% (Akintayo 2004). However, the oil yield in this study was higher when compared to that of several promising non-edible oilseed crops for biodiesel production such as *Raphanus sativus* L. at 26% (Shah et al. 2013), *Sapium sebiferum* L. at 25% (Wang et al. 2011), rubber seed at 24% (Roschat et al. 2017), *Aleurites moluccana* at 20%, *Moringa oleifera* at 25%, and *Pachira glabra* at 23% (Kibazohi & Sangwan 2011).

TABLE 1. Proximate composition of *J. podagrica* seed kernel, cake and capsule

Composition (%)	Seed kernel		De-oiled seed kernel	Seed capsule
	Dry season	Rainy season		
Moisture	3.51 ± 0.41 ^c	5.61 ± 0.52 ^b	2.13 ± 0.36 ^d	13.00 ± 0.29 ^a
Ash	7.74 ± 0.63 ^a	7.58 ± 0.40 ^a	6.97 ± 0.37 ^b	4.20 ± 0.33 ^c
Oil	30.87 ± 0.88 ^a	24.09 ± 0.75 ^b	2.19 ± 0.47 ^d	5.38 ± 0.19 ^c
Protein	28.74 ± 0.69 ^a	22.42 ± 0.56 ^c	27.65 ± 0.51 ^b	2.00 ± 0.25 ^d
Fibre	7.45 ± 0.21 ^c	10.54 ± 40 ^b	7.13 ± 0.21 ^c	66.38 ± 0.39 ^a
Carbohydrates	21.69 ± 0.52 ^b	29.76 ± 0.49 ^a	21.69 ± 0.52 ^b	9.04 ± 0.23 ^c
Energy (cal g ⁻¹)	-	-	-	3632

Data are presented as mean ± SD. Means in the same row with similar superscript (^{a,b,c,d}) did not significantly differ (p>0.05)

FATTY ACID COMPOSITION

The main components of *J. podagrica* seed oil were the unsaturated fatty acids, linoleic acid (70%), and oleic acid (15%). Saturated fatty acids, primarily stearic acid (6%), and palmitic acid (9%), were also identified (Figure 2 & Table 2). Supamathanon et al. (2011) reported that the components of *J. curcas* seed oil cultivated in the North-Eastern part of Thailand were linoleic acid and oleic acid. In addition, stearic acid and palmitic acid were the major saturated fatty acids reported. Emil et al.

(2010) also reported the unsaturated fatty acids, linoleic acid, and oleic acid, as well as the saturated fatty acids, stearic acid, and palmitic acid, as the major fatty acid components of *J. curcas* seed oil from Thailand. The fatty acid components of *J. podagrica* seed oil in this study were comparable to those of oils from *J. curcas* seed, soybean and sunflower (Table 2). However, the linoleic acid content was very high while the oleic acid content was lower in *J. podagrica* when compared to those in *J. curcas* and soybean.

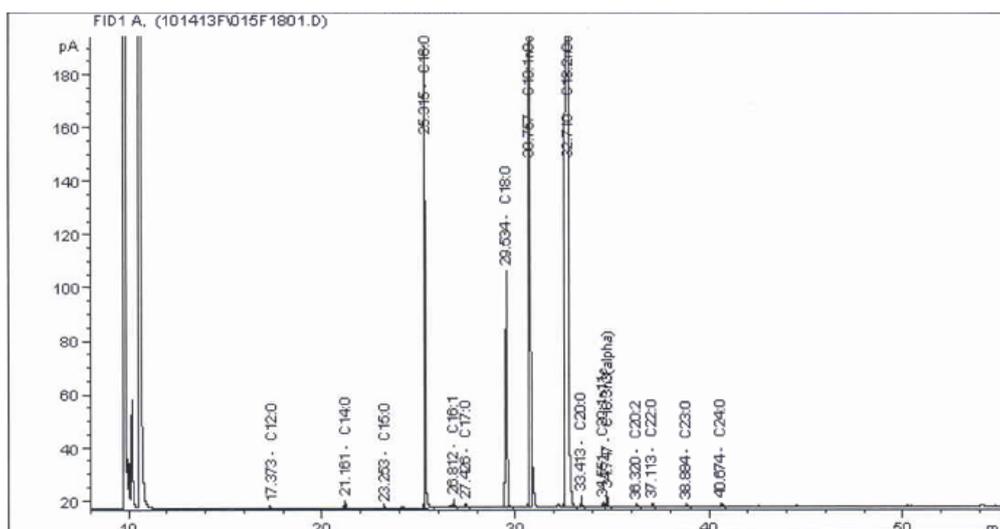


FIGURE 2. Fatty acid composition of *J. podagrica* FAMES detected by GC

TABLE 2. Fatty acid composition of *J. podagrica* seed oil

Fatty acid composition (%)	<i>J. podagrica</i> (Present study)	<i>J. curcas</i> (Emil et al. 2010)	Soybean (Karmakar et al. 2010)	Sunflower
Lauric acid (C12:0)	0.02	-	-	-
Myristic acid (C14:0)	0.13	0.10	<0.50	<1.00
Pentadecanoic acid (C15:0)	0.01	-	-	-
Palmitic acid (C16:0)	8.51	13.20	7.00-11.00	3.00-6.00
Heptadecanoic acid (C17:0)	0.09	0.10	-	-
Stearic acid (C18:0)	5.57	7.70	2.00-6.00	1.00-3.00
Arachidic acid (C20:0)	0.18	0.30	<1.00	0.60-4.00
Behenic acid (C22:0)	0.04	-	-	0.80
Tricosanoic acid (C23:0)	0.02	-	-	-
Lignoceric acid (C24:0)	0.05	-	-	-
Saturated fat	14.62	-	-	-
Palmitoleic acid (C16:1)	0.16	0.60	-	-
Oleic acid (18:1)	14.71	48.80	19.00-34.00	14.00-35.00
Eicosenoic acid (C20:1)	0.09	-	-	-
Monounsaturated fatty acid	14.97	-	-	-
Linoleic acid (C18:2)	70.15	28.80	43.00-56.00	44.00-75.00
α -Linolenic acid (C18:3)	0.24	0.10	5.00-11.00	<1.5
Eicosadienoic acid (C20:2)	0.02	-	-	-
Polyunsaturated fatty acid	70.41	-	-	-
Unsaturated fat	85.38	-	-	-

J. podagrica seed oil was found to be liquid at room temperature and showed a yellow to soft brown color. The chemical properties of the seed oil (Table 3) showed an iodine value of 134.11 g 100 g⁻¹, which was greater than the set iodine value standard of 120 g 100 g⁻¹ and 130 g 100 g⁻¹ recommended for biodiesel production by Europe's EN 14214 and EN 14213 specifications, respectively (Saluja et al. 2016), but less than that of the Spanish regional standards (140 g 100 g⁻¹) (Chuah et al. 2016). The iodine value in this study was also higher than that reported for *J. curcas* seed oil (Table 3), but lower when compared to those that have been reported for several non-edible seed oils studied for biodiesel production including *Cucurbita pepo* (150.37 g 100 g⁻¹) and *Cucumis melo* (135.36 g 100 g⁻¹) (Ibeto et al. 2012). However, the value was within the range reported for popular edible oils, soybean, and sunflower, which have been used in first

generation biodiesel production (Table 3). The high linoleic acid content (Table 2) in this study may have contributed to the high iodine value. Iodine value indicates the level of unsaturation in the oil (Emil et al. 2010). A high iodine value is associated with the production of several degradation products, due to low oxidation. This leads to the formation of deposits on the piston ring grooves, piston rings, and nozzles of diesel engines, affecting their smooth running (Pinzi et al. 2009). Different countries have established various iodine value limits around the EU EN 14214 specifications.

The saponification value (186.70 mg KOH g⁻¹), free fatty acids (0.91%), and viscosity (36.47 cP, 40°C) of *J. podagrica* seed oil in the current study were lower when compared to those reported for *J. curcas* seed oil. On the other hand, unlike the saponification value, the free fatty acids and viscosity of *J. podagrica* seed oil

was higher when compared to those of soybean and sunflower oils (Table 3). The saponification value is very significant in checking oil adulteration. Despite the lower saponification value in this study when compared to *J. curcas*, soybean, and sunflower, it was still very high. This makes *J. podagrica* seed oil more suitable for liquid soap and shampoo production. For the production of biodiesel from *J. podagrica* seed oil, alkaline-catalysed transesterification may not be suitable due to an increase in soap formation and possible increase in viscosity as a result of the high saponification value (Abbaszaadeh et al. 2012). However, acid-catalysed transesterification has been recommended for oils with high FFA or the conversion of soaps to esters, although the process is slow. Optimisation of the catalyst loading, alcohol to oil molar ratio, process temperature and time may help to enhance the efficiency of conversion during acid-catalysed transesterification (Borugadda & Goud 2012). To increase the yields of methyl esters, a two-step transesterification process may efficiently convert the oil to biodiesel (Ibeto et al. 2012). The viscosity of the oil is a very important factor to determine its suitability for biodiesel production. It affects the efficiency of injection as well as combustion. High viscosity results in high pressure and injection volume due to an increase in the pulling force in the injection

pump (Pinzi et al. 2009). The low viscosity of *J. podagrica* seed oil gave it better lubrication properties when compared to that of *J. curcas* (Table 3). However, it was too high to be used directly as fuel for engines. Transesterification of the oil may help to reduce its viscosity and enhance its use as biodiesel for engines.

The density of *J. podagrica* seed oil (0.90 g cm⁻³) was found to be similar to those of *J. curcas*, soybean and sunflower (Table 3). Nevertheless, the acid value of *J. podagrica* seed oil (127.45 mg KOH g⁻¹) was very high when compared to those of *J. curcas*, soybean, sunflower (Table 3) and other vegetable oil studied for biodiesel production including *Brachystegia eurycoma* (27.08 mg KOH g⁻¹), *Cucurbita pepo* (36.47 mg KOH g⁻¹), *Luffa cylindrica* (2.47 mg KOH g⁻¹), *Cucumis melo* (5.40 mg KOH g⁻¹), and *Arachis hypogea* (2.61 mg KOH g⁻¹) (Ibeto et al. 2012). The high acid value makes separation very difficult due to the formation of soap during transesterification. This results in low yield of biodiesel (Chuah et al. 2016). A sequence of acid catalysed processes is therefore ideal for biodiesel production from *J. podagrica* seed oil. Wang et al. (2011) reported a significant reduction in the acid value of *J. curcas* L., *Euphorbia lathyris* L., and *Sapium sebiferum* L. oils after application of a two-step catalytic process for biodiesel production.

TABLE 3. Chemical properties of *J. podagrica* seed oil

Properties	<i>J. podagrica</i> (Present study)	<i>J. curcas</i> (Emil et al. 2010)	Soybean (Karmakar et al. 2010; Verma & Sharma 2016)	Sunflower
Iodine number (g 100 g ⁻¹)	134.11	92.53	128-143	125-140
Saponification value (mg KOH g ⁻¹)	186.70	216.09	195.30	193.14
Free fatty acids (FFA, %)	0.91	1.69	0.35	-
Viscosity (cP, 40 °C)	36.47	47.50	28.87	35.84
Density (20 °C, g mL ⁻¹)	0.90	0.90	0.91	0.92
Acid value (mg KOH g ⁻¹)	127.45	42.78	0.71	0.50
Physical state at room temperature	Liquid	Liquid	-	-

PROXIMATE ANALYSIS

Analysis of the proximate composition of *J. podagrica* seed kernel, de-oiled seed kernel (seed cake) and seed capsule was shown in Table 1. The protein content (29%) of seeds harvested in the dry season was also higher

($p < 0.05$) than that of seeds harvested in the rainy season (22%). The rainy season seeds recorded 6% moisture, 11% fibre and 30% carbohydrate contents which were higher ($p < 0.05$) when compared to those of the dry season seeds (4, 7, and 22%, respectively). However, there was no difference ($p > 0.05$) between the two groups of seeds in

relation to their ash content (Table 1). The low moisture content of seeds harvested in the dry season further confirmed their suitability for biodiesel production and other industrial applications.

Oil extraction from *J. podagrica* seed kernel was accompanied by the production of large amount of seed cake. Analysis of the seed cake showed its good nutritional potential in the presence of high protein (28%) and carbohydrate (22%) contents (Table 1). This established a good basis for more research into the potential of *J. podagrica* seed cake as feed resource and other industrial applications.

The main component of *J. podagrica* seed capsule was found to be fibre (Table 1). Minor components including oil, carbohydrates, and protein were also identified, an indication of the poor nutritional value of the capsule. On the other hand, the capsule may be a good source of fuel due to its energy content.

CONCLUSION

J. podagrica seed oil in this study was unsaturated due to its significant linoleic acid and oleic acid contents. The study showed that the seed oil is more suitable for the production of soap and shampoo in Thailand. To convert *J. podagrica* seed oil to biodiesel, acid-catalysed transesterification process is appropriate. A two-step transesterification to increase the yields of methyl esters would be better in the conversion process. The seed cake produced after oil extraction would be very useful as animal feed due to its high nutritional content. Nevertheless, more research is needed to determine if the seed cake is non-toxic.

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Duangporn Premjet
Centre of Excellence in Research for Agricultural Biotechnology
Faculty of Agriculture Natural Resources and Environment
Naresuan University
Muang Phitsanulok, 65000
Thailand

Abraham Kusi Obeng
Department of Biotechnology
Faculty of Biosciences
University for Development Studies
P.O. Box TL 1882, Tamale
Ghana

Hah Young Yoo
Department of Biotechnology
Sangmyung University
Seoul 03016
Korea

Seung Wook Kim
Department of Chemical and Biological Engineering
Korea University
145, Anam-ro, Seongbuk-gu
Seoul 02841
Korea

Siripong Premjet*
Department of Biology, Faculty of Science
Naresuan University
Muang Phitsanulok, 65000
Thailand

*Corresponding author; email: siripongp@nu.ac.th

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