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Full Length Article

Simultaneous solvent extraction and transesterification of jatropha oil for biodiesel production, and potential application of the obtained cakes for binderless particleboard

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HIGHLIGHTS

- We investigate the biodiesel production directly from jatropha seeds.
- We investigate the binderless particleboard production from cakes as by-product.
- Increasing *n*-hexane and methanol to seed ratios will increase yield and quality.
- Increasing cakes moisture content will increase binderless particleboard quality.

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ABSTRACT

This study investigated biodiesel production from jatropha seeds in a single step, i.e. by simultaneous solvent extraction and transesterification of jatropha oil, and possibility to transform the obtained cakes into binderless particleboards. *n*-Hexane was used as extracting solvent. The best operating conditions were identified to obtain optimal biodiesel yield and quality, and optimal physical and mechanical properties for binderless particleboards. Biodiesel yield was usually influenced by operating conditions, and the influences of both *n*-hexane to seed and methanol to oil ratios were most significant. An increase in *n*-hexane to seed ratio (from 1:1 to 3:1) combined with the decrease in methanol to oil ratio (from 13.3:1 to 8.0:1) led to an improvement in biodiesel yield. The best biodiesel yield (92% with a fatty acid methyl ester purity >98%) was obtained from 2:1 *n*-hexane to seed ratio, 10.6:1 methanol to oil ratio, 200–600 rpm stirring speed, 50 °C temperature and 6 h reaction time. Operating conditions had no significant effect on the biodiesel quality, except the *n*-hexane to seed ratio. Moreover, cohesive particleboards were produced from the obtained cakes, proteins and fibers acting respectively as binder and reinforcing fillers. An increase in the cake moisture content significantly improved the particleboard properties. The most promising binderless particleboard was manufactured from cake B under 20% cake moisture content and 160 °C pressing temperature. Its properties were 0.87 g/cm^3 density, 8.4% moisture content, 7.2 MPa modulus of rupture, 10.4 GPa modulus of elasticity, 0.14 MPa internal bonding strength, 52% water absorption and 20% thickness swelling after 24 h immersion in water.

1. Introduction

The cultivation of *Jatropha curcas* spread widely in Central and South America, South-East Asia, India and Africa [1]. It is a drought-resistant plant with many utilizations and great prospective [2–6]. This plant was widely developed to reclaim land, prevent and/or control erosion, as well as an energy source for biofuel production [7,8].

The seed is the highest potential of the jatropha plant due to its high oil (40–60%) and protein (20–30%) contents [1,8]. Furthermore, the biodiesel produced from jatropha oil is regarded as a potential substitute to diesel fuel with certain advantage, such as

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its high flash point [7]. It is thus not dangerous to stock up and handle.

The biodiesel synthesis from vegetable oils by conventional industrial technology consists of many separate steps including oil extraction from the seed by mechanical pressing and solvent extraction using *n*-hexane as extracting solvent [9–11], oil refining by degumming, neutralization, bleaching and deodorization [12,13], and then alkaline transesterification of oil with monohydric alcohol [14,15]. A large part of the industrial biodiesel production has applied this technology. However, as the cost for vegetable oil production is very high (approximately 70% of the biodiesel production costs) [7,8,16–19], a new biodiesel production process, simple, compact, efficient, low cost and less energy consumer, is necessary to be developed.

The in situ transesterification has been applied with success for biodiesel production from various oilseeds [6,8,16–30]. The conversion of jatropha seeds into fatty acid methyl esters (FAME) by acid-catalyzed in situ transesterification has been reported by Shuit et al. [26]. A 99.8% FAME yield was obtained under reaction conditions of 60 °C temperature, 24 h duration, 7.5 mL/g methanol to seed ratio, 15 wt.% H_2SO_4, seed particle size less than 0.355 mm and *n*-hexane as co-solvent [26]. In this study, the processing steps and cost for biodiesel production were successfully reduced by conducting the solvent extraction and the in situ acidic transesterification of jatropha seeds in a single step. On the other hand, the simultaneous solvent extraction with *n*-hexane as extracting solvent and in situ transesterification with KOH as catalyst have also been successfully carried out for the direct conversion of jatropha seeds to FAME [8]. A 87% biodiesel yield with a fatty acid methyl ester purity of 99.7% was obtained under reaction conditions of 50 °C temperature, 800 rpm stirring speed, 5 h duration, 6 mL/g methanol to seed ratio, 6.4 wt.% KOH and seed particle size of 35 mesh. The biodiesel production from jatropha seeds by simultaneous solvent extraction and in situ transesterification increased both yield and quality of biodiesel. The constant ratio of *n*-hexane added to the seed (i.e. 1:1 for all experiments, expressed in mL/g) was used in this study. In addition, a single step for solvent extraction and alkaline transesterification of oil from jatropha seeds at pilot scale with different ratios of *n*-hexane to seed has never been reported. Thus, a comprehensive study should be conducted to examine the effect of *n*-hexane to seed ratio. Furthermore, the latter should also investigate the possibility of reducing the large reacting methanol volume (molar ratio of reacting methanol to jatropha oil of 104:1) used in the previous study [8] while maintaining a high FAME yield.

The residual oil and FAME contained in the cake after biodiesel production by the in situ transesterification of jatropha seeds is high (more than 11%) [8]. Even though the direct utilization of the cake (i.e. for animal feed) is less favorable, the conversion of this cake into usable energy by combustion, gasification or pyrolysis is more advantageous [31,32]. In addition, due to its excellent action as reinforcing filler for a biodegradable polymer (i.e. polycaprolactone/PCL), the cake has considerable potential in biocomposite applications [33]. As a mixture of proteins and lignocellulosic fibers, the transformation of the cake into biodegradable and value-added agromaterials by thermo-pressing could also be considered [34–43]. Thus, the possibility to produce cohesive binderless particleboards from cakes obtained after in situ transesterification of jatropha seeds should be also examined.

This study was conducted to investigate biodiesel production from jatropha seeds in a single step, i.e. by simultaneous solvent extraction and transesterification of jatropha oil, at pilot scale. The influences of *n*-hexane to seed ratio, methanol to oil ratio, stirring speed, temperature and reaction time were examined to identify the optimal reaction conditions and to define best performance of biodiesel yield and quality. In addition, the influence of thermo-pressing conditions, i.e. pressing temperature and moisture content, on the physical and mechanical properties of binderless particleboards produced from the obtained cakes was also evaluated.

2. Materials and methods

2.1. Materials

For all trials conducted in this study, the whole jatropha seeds used were from IP2 Lampung variety, and they were supplied by the Indonesian Spices and Industrial Crops Research Institute (Sukabumi, Indonesia). At storage, the moisture content of jatropha seeds was 7.8 ± 0.5% (French standard NF V 03-903). *n*-Hexane (>98% purity) and methanol (>98% purity) were obtained from Brataco Chemical Ltd (Indonesia). All analysis solvents and chemicals were pure analytical grades, and they were obtained from Sigma–Aldrich, Fluka and J.T. Baker (Indonesia and France).

2.2. Experimental procedure for biodiesel production

For biodiesel production, the moisture content of jatropha seeds was less than 2% (French standard NF V 03-903). To obtain jatropha seeds with less than 2% moisture content, they were dried in a ventilated oven at 60–70 °C for 24–48 h. Then, the dried seeds were milled using an electric grinder fitted with a 20 mesh size.

Milled jatropha seeds (1000 g) and methanol in which KOH had been dissolved (concentration fixed at 0.075 mol/L) were mixed. The methanol to oil ratio (v/w, expressed in mL/g) was 8:1–13:1. To increase oil miscibility in the mixture, to accelerate the reaction and to complete it in a single phase, *n*-hexane was then added (1000–3000 mL), corresponding to a 1:1–1:3 seed to *n*-hexane ratio (w/v). The total solvent to seed ratio (v/w, expressed in mL/g) was thus 6:1 for all the experiments conducted. Both KOH amount and total solvent to seed ratio were based on optimal values from the previous study [8]. The biodiesel production was conducted using a reactor with 10 L capacity. The latter was equipped with a reflux system, an agitator and a heater. The other operating conditions tested were the stirring speed (200–600 rpm), the temperature (40–50 °C) and the reaction time (4–6 h).

When the reaction has been completed, the mixture was cooled to ambient temperature. Then, it was filtered vacuum, allowing the separation between the filtrate and the cake. Methanol and *n*-hexane were recovered from the filtrate by evaporation using a rotary evaporator, and this made possible to separate the filtrate into two layers. Dark brown in color, the lower layer contained glycerol. The upper one constituted the crude biodiesel, and it was yellow in color. It contained not only the fatty acid methyl esters produced but also the unreacted glycerides (triglycerides, diglycerides and monoglycerides) like other impurities. Methanol and *n*-hexane may extract other materials than glycerides, such as fatty acids and phospholipids. After washing with water until neutrality, the crude biodiesel was then dried (105 °C, 1 h). Its fatty acid methyl ester, triglyceride, diglyceride, monoglyceride and fatty acid contents were then determined using gas chromatography. The washed and dried crude biodiesel was weighed, and the biodiesel yield was calculated using the same equation as that used in the previous study [8]. Each experiment was conducted twice, thus leading to an average value and a standard deviation for the biodiesel yield. The isolated cake still contained part of the fatty acid methyl esters produced during the in situ transesterification because it was not washed with methanol to take out the esters. It was dried overnight at room temperature. The influence of all operating conditions (i.e. methanol to oil ratio, *n*-hexane to seed ratio, stirring speed, temperature and reaction time) on biodiesel yield and biodiesel quality was studied through a randomized